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Pacific Northwest Laboratories
Richland, Washington 99352

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PACIFIC NORTHWEST LABORATORY
MONTHLY REPORT ON ISOTOPES
DEVELOPMENT PROGRAMS
FOR AUGUST 1972

SEPTEMBER 15, 1972

MASTER

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PACIFIC NORTHWEST LABORATORY
MONTHLY REPORT
ON ISOTOPES DEVELOPMENT PROGRAMS
FOR AUGUST 1972

Compiled by J. H. Jarrett

September 15, 1972

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PACIFIC NORTHWEST LABORATORY
MONTHLY REPORT
ON ISOTOPES DEVELOPMENT PROGRAMS
FOR AUGUST 1972

08-01. RADIOISOTOPES PRODUCTION AND
MATERIALS DEVELOPMENT

08-01-03. FISSION PRODUCTS

II. UTILIZATION OF FISSION PRODUCTS Rh, Pd, Ru, and Tc -
F. P. Roberts, L. L. Clark and J. D. Kaser

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.*

Efforts were continued in developing of the economic analysis of the recovery of Rh, Pd, Ru and Tc from nuclear wastes.

An estimate was made of the capital equipment costs for a plant to recover Rh, Pd, Ru and Tc from the wastes generated by a 5 T/d fuels reprocessing plant; and using these capital costs, a cash flow model based on the assumptions stated in the July report was developed. Through the use of this model, the characteristics of the revenues and costs were determined, and the probable range of profitability was established. The relative contribution to the profitability of each of the products (Rh, Pd, Ru and Tc) was established.

The capital equipment costs for plant and equipment to recover Rh, Pd, Ru, and Tc using the conceptual process described in the June report were estimated at approximately \$2 million. This value includes the cost for a plant processing wastes from a 5 T/d fuels reprocessing plant and a storage facility for holding the recovered rhodium and ruthenium. It does not include facilities for decontamination and purification of the recovered products.

The evaluation of the relative value of each of the products showed palladium to be the most valuable of the individual products if it was assumed that it can be sold immediately on recovery, but all products contributed to the overall profitability. Technetium and ruthenium contributed least to the final present value and were, therefore, economically the least essential.

The effect on profitability of the time of earliest sale was explored. The results showed that palladium and rhodium sale time was the most critical, and that palladium sale must start in the first 10 years of the project to achieve a target return of 10%.

The range of profitability was explored by changing various combinations of cost and revenue. Since profitability in terms of rate of return depended on whether the financing was internal

or external, two cases were chosen. In the first case, debt financing from sources external to the company undertaking the venture was assumed. In this case the rate of return varied from 21% under the most positive conditions to 1% under the most negative. In the second case, financing was assumed to be completely internal. Under internal financing the rate of return would range from 23% to 1.5 to 2%, less the company's cost of capital which typically ranges from 5 to 10%.

Further work on the marketing analysis is being done to determine if rhodium demand is dependent upon platinum demand. This would critically affect our assumption of being able to sell rhodium during platinum shortage. Also the uses of Rh, Pd, Ru and Tc in industry are being studied to determine applications where fission product metals could possibly be licensed and used. Two important criteria to be used in determining feasible applications are the radiation hazard to humans and the possible contamination of natural source metals during recycling.

III. 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 use 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.

ORNL has conducted further beneficial purification of the stock of fission product xenon. Although this additional purification was impressive, it is not sufficient to provide for the purity required in the detector uses mentioned in last month's report. A possibility for further purification of the material to extremely low krypton-85 levels was confirmed with Airco personnel. The cost for this service would be very low, on the order of \$200 total. This purification step is being given strong consideration because of its reasonable cost and also because of the anticipated favorable effect on the demand for experimental quantities of xenon for application evaluation that could lead to commercialization.

V. STRONTIUM FLUORIDE COMPATIBILITY - H. T. Fullam

Lower cost strontium is possible by using the Hanford Waste Management Program (HWMP) produced fluoride as a fuel form instead of the currently used titanate. Many potential uses of strontium can be made more cost-effective if the HWMP material can be safely and efficiently utilized. The objective of this program is to determine the chemical compatibility and properties of strontium fluoride as produced by HWMP and develop suitable data for strontium fluoride licensing procedures.

The goals in FY-72 are to establish further contacts with regulatory agencies, detail the program work plan, and initiate the procurement of needed equipment and materials.

Work is underway to select the containment materials which will be evaluated with strontium fluoride. Prior work on strontium fluoride compatibility has been reviewed, as well as other projects which have involved metal fluoride containment. These have included the Molten Salt Reactor program at ORNL and the fluoride volatility programs at ANL, BMI and ORNL. Potential customers for SrF_2 heat sources have been contacted and their heat source specifications determined. While selection of the containment materials to be tested is still incomplete, it has been decided that the following alloys will be tested: Hastelloy C-276, Hastelloy N, Haynes 25, Inconel 625, and TZM. Also under consideration are Inconel 600, Hastelloy X and tungsten.

The initial testing program has been expanded to include radioactive tests during the first year. It is currently planned that the various test materials will be evaluated with three sources of SrF_2 :

1. high purity inert SrF_2 ,
2. high purity $^{90}\text{SrF}_2$ - the strontium will be purified by ion exchange in the 325 Building hot cells, and
3. $^{90}\text{SrF}_2$ prepared by the WESF flowsheet.

The tests will last for six months and will be carried out at three temperatures; 800°C, 1100°C and an intermediate temperature still to be selected. The tests with inert SrF_2 will start in October and those with $^{90}\text{SrF}_2$ in January.

Preparation of high purity inert fluoride is now underway. The fluoride is being prepared by the reaction of strontium nitrate solution with sodium fluoride. Analysis of the fluoride product presents a difficult problem, especially the determination of the water and oxide content. Normal methods for determining water are not applicable to SrF_2 , especially after it has been fired at 1100°C. The only methods which appear

applicable to hydrogen determination in SrF_2 are spark source mass spectrography (SSMS) and tritium analysis. In the tritium analysis the SrF_2 will be precipitated from tritiated water, fired at 1100°C , dissolved in 6M HCl and the residual tritium determined by a low-level counting technique. Spark source mass spectrography can be used for hydrogen, but the precision with SrF_2 will be about a factor of ± 3 . The only method currently available for oxygen is SSMS and again the precision is a factor of ± 3 . Use of the vacuum fusion method for oxygen is being investigated, but it presents severe problems when applied to SrF_2 . The KBrF_4 method can be used for oxygen in metal fluorides, but is not available at Hanford. Other laboratories are being contacted to see if they can analyze our inert fluoride by the KBrF_4 procedure.

The analysis by spark source mass spectroscopy of reagent grade SrF_2 after it has been fired at 1100°C shows the purity to be >99.9 wt%. This will be compared with fluoride prepared from strontium nitrate.

08-01-04. SOURCE DEVELOPMENT

I. MEDICAL ISOTOPE GENERATOR - J. H. Jarrett

The objective of this program is to investigate the related technical and economic factors to meet the need for a source of short half-life radioactive material that can be used for medical diagnostic applications. Emphasis will be placed on the study of a ^{13}N ($t_{1/2} = 10$ min) generation system that may be located in a hospital complex.

Effort on this program will be initiated in September.

II. BIOMEDICAL SOURCE DEVELOPMENT PROGRAM - J. H. Jarrett,
F. P. Hungate and F. T. Cross

The objective of this program is to design and fabricate portable, possibly implantable, sources for blood irradiation in support of an ongoing AEC-DBER program (Dr. F. P. Hungate, PNL, Principal Investigator) to evaluate in-vivo blood irradiation. Close liaison is being maintained with AEC-DBER staff and members of the medical community to assure optimum program responsiveness to source development needs.

Effort on this program will be initiated in September.

08-02. THERMAL APPLICATIONS

08-02-01. CIRCULATORY SUPPORT SYSTEMS -

F. T. Cross and B. A. Crea

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 50W plutonium heat sources.

I. Dosimetry Studies

Parametric computations of dose rates to organ systems and whole body continue, varying the source location and size.

II. Radiological Engineering Studies

Preliminary designs of spherical heat exchangers for dissipating heat directly to surrounding tissue were initiated.

Heat transfer studies with the Phase I heat exchangers and components were initiated.

08-03. RADIOISOTOPE SYSTEMS DEVELOPMENT

08-03-01. APPLIED INSTRUMENTATION DEVELOPMENT

I. Low-Flux Neutron Imaging Technology Development -

K. L. Swinth

The objective of this program is to develop and investigate low-flux neutron imaging techniques for use with radioisotopic neutron techniques to be used with radioisotopic neutron sources including ^{252}Cf . Previous efforts have resulted in development of improved imaging techniques using $\text{Gd}_2\text{O}_2\text{S:Tb}$ phosphors, europium foils, and a prototype imaging proportional counter. Optimum imaging techniques (high speed) are not necessary for reactor neutron radiography; however, development of such techniques will promote low-flux neutron radiography with radioisotope sources.

A prototype imaging proportional counter was fabricated and tested successfully and advanced versions are being designed for evaluation under the present effort. This counter will provide instantaneous digital readout of data representing the imaged object. Various other techniques such as image intensifiers, advanced scintillation phosphors and ^6LiF thermoluminescent foils are also under investigation.

An image intensifier system was evaluated for its use in decreasing exposure times in neutron radiography. A low dark current intensifier system was used as a light amplifier between a neutron-sensitive scintillator and a camera with high-speed film. Various large aperture lens configurations were evaluated to determine an optimum configuration. Images were made in as little as 20 sec in a flux of $\sim 10^3$ n/cm²-sec; however, image detail was poor. Using an exposure time of 120 sec with a less efficient lens coupling, holes 2 mm in diameter in a cadmium strip could be resolved.

An exposure curve was generated for direct exposures using a single 2 mil gadolinium backscreen. Using a 40:1 collimator ($\phi \approx 8 \times 10^2$ n/cm²-sec), it takes 150 hr to reach a radiographic density of 1.5. Images produced by this combination show excellent detail and will be used for comparison with techniques presently under development.

II. Advanced Source Development -

J. H. Jarrett and W. F. Riemath

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.

Cleaning and reassembly of the cold (non-radioactive) sputtering system have been completed. The system has been evacuated and placed on liquid nitrogen. The RGA has been

assembled but not evacuated, awaiting liquid nitrogen controllers which should be delivered and installed in September. Upon activation of the RGA, the cold system will be leak checked and a background level of system impurities will be determined.

The radioactive sputtering system has been cleaned and is being reassembled with completion expected in September. The radioactive vacuum system has been cleaned and new "O" rings installed which will enable baking the system out at 450°F. The new C.P.A. Liquid Nitrogen Cold Trap was installed at this time also, and the supply lines will be installed in September.

08-03-03. OCEANOGRAPHIC AND NATURAL RESOURCE APPLICATIONS

I. Nuclear Techniques for Seabed Mineral Exploration - R. W. Perkins, N. A. Wogman, H. G. Rieck, Jr., and J. R. Kosorok

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.

A test site for the initial sea trials of the seabed mineral analyzer has been selected and arrangements have been made to use the site. The privately-owned site at Port Townsend, Washington, has a crane for positioning the seabed probe on the ocean floor at the end of the dock. There is approximately 50 ft of water over a sandy, relatively level bottom at the location. Currents are negligible, so it will be possible to accurately position the support structure. The lack of currents will also allow the use of a diver if this should prove necessary.

Normal transportation facilities will be used for the 260 μg ^{252}Cf source in its shipping cask, since dose levels at the surface of the cask are well below the limits specified by radiation protection guidelines.

Samples of several synthetic minerals have been cast in concrete blocks which have a composition and density that simulates actual mineral deposits. These synthetic minerals will be placed on the sea floor during the trials to determine detection efficiency and sensitivity.

A delay in the ocean testing resulted from the failure of the Richards vacuum valve in the X-ray detector's pressure housing. A temporary system of seals and valves was designed to maintain the required low pressure in the housing for an adequate time to perform undersea measurements. Replacement of the faulty valve must eventually be performed at the vendor's laboratory.

It was observed that operation of the TV camera causes some degradation of the resolution of the X-ray detector signal. Until this can be overcome, it is planned to turn the camera off during the counting period.

PROMETHIUM INVENTORY - AUGUST 31, 1972

Shipments - Through ORNL Isotope Sales	1,718,530 Ci
PNL Available Inventory	13,873,906
Crude Feed	10,734,604
Pre-Purified	2,983,221
Product - Run 59	51,202
Run 60	39,200
Run 61	44,451
Run 62	21,228
(SUBTOTAL)	13,873,906
Decay	13,762,270
Unaccounted For	1,431,941
Waste	423,721
Disposition of Promethium, <u>TOTAL</u>	<u>31,210,368</u>
Received from DWDL	45,000
Received from ARHCO	31,165,368
TOTAL RECEIPTS	<u>31,210,368</u>

Pm Inventory Account Activities - D. H. Siemens

No purification runs or shipments were made during this report period. Current status of the crude ^{147}Pm is as follows:

<u>TK</u>	<u>Volume (gal)</u>	<u>H⁺ (M)</u>	<u>SP.G</u>	<u>Temp (°C)</u>	<u>Evaporation Rate (GPD)</u>
PT-1	1600	0.60	1.160	59	6
PT-2	1765	0.55	1.150	61	5

A replacement has been ordered for the failed T-5 pump motor. Tank-5 has been flushed both internally and externally. A radiation survey is scheduled to determine if additional flushing will be required.

ATLANTIC RICHFIELD HANFORD COMPANY (ARHCO) FISSION PRODUCT
ACTIVITIES - G. E. Benedict

Mr. Harold "Perk" Bibb of the Naval Facilities Command visited Atlantic Richfield Hanford Company (ARHCO) on August 1, 1972, to discuss Navy uses of ^{90}Sr and problems of substituting ARHCO $^{90}\text{SrF}_2$ for the titanate presently used. Mr. Bibb gave a brief lecture on the proposed naval thermoelectric generators and toured the Waste Encapsulation and Storage Facility (WESF) with personnel from ARHCO, Pacific Northwest Laboratory (PNL) and Atomic Energy Commission - Richland (AEC-RL).

A conference call was arranged on August 21, 1972, between personnel from ARHCO, PNL, Oak Ridge and Teledyne Corporation to discuss questions which Teledyne had concerning the Federal Register Document 72-12333, "Atomic Energy Commission Fission Product Radioisotopes - Distribution and Pricing Policy".

The main points covered in this call are summarized below:

- Teledyne has an interest in capsules containing 50,000 to 75,000 curies of ^{90}Sr . What is the cost penalty over the standard 150,000 curie capsule? ARHCO supplied the information that as long as the standard diameter capsule was used, the cost penalty would only include the extra time and material for filling, welding and testing two to three times as many capsules per unit of production. Full cost recovery estimates can be obtained for specific capsules and schedules through the AEC.
- Radiochemical purity of ^{90}Sr for encapsulation will be supplied as analyses are obtained.

- Teledyne's requirements on the actual content of ^{90}Sr in a capsule is $\pm 5\%$ of design specification, and they are concerned about the WESF capsules being able to meet this requirement. ARHCO has calorimetry equipment being tested which has the capability of measuring the ^{90}Sr content of a capsule to $\pm 5\%$. However, the $^{90}\text{SrF}_2$ powder is compacted into the capsule and batch size is controlled by the volume and ^{90}Sr content of the feed to the precipitation step and the completeness of precipitation. Variations may lead to many capsules being outside the $\pm 5\%$ requirement. It is believed, however, that selected capsules could be supplied within the specification limit without additional control steps.
- Teledyne personnel had many questions about reasons for selection of capsule diameter, capsule thickness, etc. These questions were answered with the repeated explanation that capsule design was based on AEC - Division of Production and Materials Management requirements for water basin storage.
- Teledyne personnel are very concerned about continuity of supply of licensable ^{90}Sr sources over the next two or three years. They were referred to the AEC for discussion of this problem.

DAT BUDGET STATUS - FY-1973 THROUGH AUGUST 31, 1972

		<u>Current Month</u>	<u>FY-1973 CTD</u>	<u>FY-1973 Budget</u>
	<u>08-01 Radioisotope Production and Materials Development</u>			
	<u>08-01-03 Fission Products</u>			
7A50-B46560	II. Rh,Pd,Ru, and Tc Utilization	10.1	12.2	40
7A50-B46341	III. Xenon Utilization	0.7	1.4	10
7A53-B46239	V. SrF ₂ Compatibility	10.5	18.3	150
	Subtotal 08-01-03	21.3	31.9	200
	<u>08-01-04 Source Development</u>			
7A50-B46246	I. Medical Isotope Generator	---	---	20
7A50-B46247	II. Biomedical Source Development (anticipatory account)	---	---	25
	Subtotal 08-01-04	---	---	45
	Subtotal 08-01	21.3	31.9	245
	<u>08-02 Thermal Applications Development</u>			
	<u>08-02-01 Circulatory Support Systems</u>			
7K21-B46850	Recipient Radiation Exposure	2.7	9.9	75
7K81-B46708	Population Radiation Exposure	5.6	12.3	55
	Subtotal 08-02	8.3	22.2	130
	<u>08-03 Radioisotope Systems Development</u>			
	<u>08-03-01 Applied Instrumentation Development</u>			
7A20-B45910	I. Low-Flux Neutron Imaging	3.6	4.7	35
7A50-B46234	II. Advanced Source Development	4.2	7.5	35
7K81-B46925	Cost Benefit Study	6.4	16.5	50
	Subtotal 08-03-01	14.2	28.7	120
	<u>08-03-03 Oceanographic and Natural Resource Applications</u>			
7K27-B46860	Seabed Mineral Exploration	3.6	6.2	60
	Subtotal 08-03-03	3.6	6.2	60
	Subtotal 08-03	17.8	34.9	180
	<u>08-04 Radiation Processing Applications Development</u>			
	<u>08-04-01 Basic Technology Development</u>			
7K81-B47485	Hospital Irradiator	5.3	5.5	50
	Subtotal 08-04	5.3	5.5	50
	Total 08 Operating Budget	52.7	94.5	605
	08 Equipment Budget	---	---	15*
	Subtotal 08	52.7	94.5	620
7A50-B46237	Pm Inventory Account	14.7	29.7	225
	PROGRAM GRAND TOTAL	67.4	124.2	845

* Spent and outstanding commitments on interim allocation.

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