BNWL-1308-3
UC-23

PACIFIC NORTHWEST LABORATORY
DIVISION OF ISOTOPE
DEVELOPMENT PROGRAMS
QUARTERLY REPORT: MAY-JULY 1970
August 1970

AEC RESEARCH &
DEVELOPMENT REPORT

BNWL-1308-3

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Radiation Applications

PACIFIC NORTHWEST LABORATORY
DIVISION OF ISOTOPE DEVELOPMENT PROGRAMS
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Edited by J. E. Hansen

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PACIFIC NORTHWEST LABORATORY
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INTRODUCTION

This report, BNWL-1308-3, is the 15th report of a series initiated in 1966 to provide detailed and timely information on the results of the isotopic fuels program and related research being conducted at the Pacific Northwest Laboratory by Battelle-Northwest for the U.S. Atomic Energy Commission. The previous reports of the series are BNWL-385, 466, 570, 680, 681, 781, 877, 924, 1010, 1085, 1177, 1256, 1308-1 and 1308-2.

This report covers the three-month period, May through July 1970.

SUMMARY

PROMETHIUM COMPATIBILITY

The $^{147}\text{Pm}_2\text{O}_3$ compatibility program has been completed with the issuance of two reports detailing the compatibility of $^{147}\text{Pm}_2\text{O}_3$ with containment materials and reactions of the sesquioxide with water. Results are presented in this report for the compatibility of Sm metal (stand-in for Pm metal) with containment materials. The materials showing the least interaction with Sm include Ta, Ta-10W, Ti, Zr, and TZM.

ENCAPSULATED WASTE MANAGEMENT CESIUM AS A PROCESS RADIATION SOURCE

Definition of a complete flowsheet for HWMP strontium purification is continuing. The effect of impurities in the HWMP strontium fluoride is being studied in relation to properties data for the pure compound. The economics of producing alternate strontium forms at HWMP is being investigated.

RADIATION CHARACTERISTICS OF FISSION PRODUCT RHODIUM AND PALLADIUM

Measurement of the decay of $^{102\text{m}}\text{Rh}$ of the last 12 months indicates a half-life of 3.4 ± 0.8 years. The precision of this measurement is expected to increase as the data is acquired for one more year.

EVALUATION OF LARGE SCALE USES FOR PURIFIED FISSION PRODUCED XENON

Purification of the subject xenon has been delayed by the strike at ORNL. Purification is expected in September. The Food and Drug Administration will have to provide special clearance before the fission product xenon can be used experimentally for human anesthesia.

NONDESTRUCTIVE TESTING FOR IRRADIATION SOURCES

Accomplishments on the NDT methods and device development program included the final construction and testing of the multielement transducer and cathode ray tube (CRT) readout systems, including a new contrast reversal readout technique. NDT technology initially developed on other BNW programs continued to be utilized when a pulsed laser schlieren system was used to image the ultrasonic beam pattern from the multielement system's transmitting transducer. DID headquarters and several contractors were visited for programmatic discussions to ensure that the program remains relevant to the needs of the contractors and industry. Steps are being taken to acquaint design and compatibility personnel with the material-evaluation potential of the ultrasonic critical angle technique. The topical report on multielement test techniques is in its final preparation and abstracts and invention reports based on this work are being submitted to appropriate parties.

ADVANCED SOURCE DEVELOPMENT

The evaluation of using rf-sputtering to prepare thin-film radiation sources was continued. The five ^{241}Pm photon sources sent to Oak Ridge National Laboratory in the previous quarter have been tested in their AEC-Division of Isotopes Development Source Safety Testing Program. Two alpha-photon source preparation runs were completed. Six of the 25 sources prepared in the first run were also sent to ORNL for testing. Testing on both the photon and alpha-photon sources is over 90% complete. Test results to date are quite encouraging, and indicate the utility of rf-sputtering in preparing thin-film radiation sources. PNL-prepared radiation sources were counted on-site for alpha and photon activity. Reference sources of known activity were prepared from a ^{241}Am solution of known concentration. Source activity in millicuries per square centimeter was determined. A review and evaluation of

the literature on the effects of alpha-radiation on different materials was completed. Materials that should best withstand the effects of alpha-radiation were identified. Stainless steel, among other materials, appears to be good candidate material for alpha-radiation sources. A rf-sputtering system for cold (non-radioisotopic) experimental work has been procured and placed in operation.

ISOTOPE PRODUCTION EVALUATIONS

Preparation of Nuclide Tables containing the properties of 2000 nuclides and isomers was completed. Firms actively involved with fuel reprocessing were visited as part of a study to determine the time and circumstances when fission-produced cesium and strontium will be attractive.

RADIATION CHARACTERISTICS OF CIRCULATORY SUPPORT ISOTOPIC HEAT SOURCES

A topical report on the "In-Phantom Dosimetry of Plutonium-238 Heat Sources in Relation to Circulatory Support Systems" is undergoing publication. Future effort is directed to establish the perturbation of prototypic heat source subsystems and source impurities on the tissue dose rates and detailing of the doses absorbed by various organ systems. A comparison of ^{147}Pm and ^{238}Pm heat sources indicates that ^{238}Pm is the more advantageous source, primarily due to the ^{146}Pm impurity and the longer half-life of ^{238}Pu .

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

A feasibility demonstration of the seabed mineral exploration technology was conducted at Battelle-Northwest's Marine Research Laboratory at Sequim, Washington. A 210 μg ^{252}Cf source was used. This demonstration verified expectations that many of the precious elements can be measured at concentrations of a few ounces per ton. Larger

^{252}Cf sources are expected to be more satisfactory for seabed mineral exploration. Arrangements have been made to continue experimentation with 2 and 10 mg ^{252}Cf sources, but their delivery is being delayed by the ORNL strike.

PROMETHIUM COMPATIBILITY

H. T. Fullam and J. H. Jarrett

The following two reports were printed and distributed during the past quarter:

H. T. Fullam. High Temperature Compatibility of Promethium Sesquioxide, BNWL-1389.

H. T. Fullam and F. P. Roberts. Reactions of the Sesquioxides of Pm, Nd, and Sm with Water, BNWL-1421.

The compatibility testing of various containment materials with samarium metal (stand-in for ^{147}Pm metal) has been completed. Ten different materials have now been tested at 400 °C, 700 °C, and 1000 °C for times of 1000, 4400, and 8800 hr. These times are equivalent to 1.4, 6, and 12 months, respectively. Summary compatibility information is presented in Table 1. The data shown in the last column are from the specimens taken out of the compatibility furnace in June. The data in the remaining columns were presented in the February-April 1970 DID Quarterly Report, BNWL-1308-2. The information is again presented for purposes of comparison.

Containment materials have been identified which show no more than a 2 mil penetration of the samarium-containment material interaction zone into the containment material wall. This is indicated quite clearly in Table 1. In each case the penetration was determined visually by metallographic examination of a cross-section showing the containment material-interaction zone interface. Materials tested that show the least interaction with samarium are Ta, Ta-10W, Ti, Zr, and TZM.

**TABLE 1. Metal-Containment Material Compatibility Using
Samarium Metal as a Stand-in for ^{147}Pm Metal**

Containment Material	Compatibility ^(a)								
	1000 hr 400 °C	4400 hr 400 °C	8800 hr 400 °C	1000 hr 700 °C	4400 hr 700 °C	8800 hr 700 °C	1000 hr 1000 °C	4400 hr 1000 °C	8800 hr 1000 °C
Tantalum	<1	<1	<1	<1	<1	<1	<1	<1	2
Ta-10W	<1	<1	<1	<1	<1	<1	<1	<1 ^(c)	2
TZM	<1	<1	<1	<1	<1	10	<1	<1 ^(b,c)	(d)
Titanium	<1	<1	<1	<1	<1	<1	<1	<1 ^(c)	(d)
Zirconium	<1	<1	<1	<1	<1	<1	<1	(d)	(c)
Inconel	<1	<1	80	125	125	125	75	(e)	(e)
Haynes-25	<1	<1	40	60	50	50 ^(c)	75	(e)	(e)
Hastelloy-X	<1	<1	75	65	63	65 ^(c)	100	(e)	(e)
Type 304L SS	<1	<1	20	40	53	50	125	(e)	(e)
Type 316L SS	<1	<1	30	60	61	55	125	(e)	(e)

- a. *Compatibility - The maximum penetration of the interaction zone into the 125 mil containment material wall, mil. The penetration was determined by the metallographic examination of a cross section showing the containment material-interaction zone interface*
- b. *3400 hr test.*
- c. *There was evidence that samarium metal may have been released from the capsule--probably in the weld area. The samarium release appeared to result from lack of weld integrity since the interaction between the capsule wall material and samarium metal was in most cases minimal*
- d. *The quartz ampoule containing the capsule broke, allowing the capsule and the samarium metal to oxidize.*
- e. *Because of the severe interaction between samarium and Inconel, Haynes-25, Hastelloy-X, Type 304L SS, and Type 316L SS in the 1000 hr, 1000 °C tests, these materials were not tested further at 1000 °C.*

ENCAPSULATED WASTE MANAGEMENT CESIUM
AS A PROCESS RADIATION SOURCE

H. H. Van Tuyl

Discussions were held on May 19 at Brookhaven with AEC-DID, BNL, ORNL, and ARHCO regarding future uses and production of cesium radiation sources. The discussions did not result in any change in scope, emphasis, or direction for the current PNL program.

Primary emphasis is currently being placed on documenting the study thus far relative to cesium. Most of the results have previously been reported in monthly or quarterly reports, but they are being organized and expanded to provide a more complete and widely distributed source of information regarding Hanford Waste Management cesium sources. Completion of the cesium report is scheduled for August.

A complete flowsheet for strontium purification has not yet been defined for the Hanford Waste Management Program (HWMP). Recent experiments with the proposed flowsheet using inorganic ion exchangers showed unsatisfactory performance with actual plant solutions. At present, the most favorable flowsheet appears to be a combination of precipitation and solvent extraction processes. Purity is still expected to be essentially the same as that used in compatibility studies.

The compilation of measured properties of strontium fluoride has resulted in a reasonably complete set of data for the pure compound. The material which will be produced in HWMP will not be completely pure, but will contain about 2 to 5% impurities. Some properties of the as-produced mixture must be known to permit licensing. A study has been initiated on the properties of the impurities to determine whether laws of solid solution can be applied to the mixture. If the mixture forms a solid solution, most of the properties of the mixture can be determined from the properties of the

components, but otherwise the properties of the mixture will have to be measured for accurate results.

The economics of producing alternate strontium forms at HWMP is being investigated. This study includes alternate source geometry for either more or less strontium per container, cost for higher density strontium fluoride, and capability and cost for producing other fuel forms such as strontium oxide. Close coordination with HWMP is being maintained in this study to determine what is possible in their plant, and what must be performed in a different facility.

A potential government use for large quantities of strontium has been identified. Contracts for a preliminary study are to be awarded by the sponsor within the next month or two. Information on strontium will be supplied to the contractor to assure that his study includes the latest data on future supplies, available fuel forms, and fabrication limitations.

A paper on "Future Availability of Fission Product Cesium and Strontium" was presented at the American Nuclear Society meeting in Los Angeles, and has been published in the Transactions of the American Nuclear Society.

RADIATION CHARACTERISTICS OF FISSION
PRODUCT RHODIUM AND PALLADIUM

F. P. Roberts

^{102m}Rh HALF-LIFE

The exploitation of fission product rhodium for some industrial applications may be limited by the amount of radioactive rhodium present. From earlier work it is known that ^{102}Rh and ^{102m}Rh are the principle radioactive isotopes present in power-reactor produced rhodium. The ^{102m}Rh has a half-life in excess of 3 years and will be the limiting factor for determining time needed for decay of the radioactivity to acceptable levels. At present the value of the half-life of ^{102m}Rh is not known with sufficient accuracy for this purpose. A study is in progress to establish an accurate value for the half-life.

Samples of rhodium have been recovered from 13-year-old Hanford waste. Because of the great age of this material it contains an insignificant amount of the short-lived ^{102}Rh and is ideal for following the decay of ^{102m}Rh .

The decay of the rhodium samples has been followed for 12 months. Calculations made at the end of this report period show a half-life ^{102m}Rh of 3.4 ± 0.8 years. This is based on a decrease in activity of 18.4%. The precision of the measurement will increase with time. It is intended to continue the acquisition of data for one more year.

EVALUATION OF LARGE SCALE USES FOR PURIFIED
FISSION PRODUCT XENON

C. A. Rohrmann

A prolonged labor dispute at ORNL, which was settled in late June, prevented the earlier initiation and completion of the proposed xenon purification campaign. A previously scheduled ^{85}Kr campaign which was also similarly delayed, caused further interruption of progress on the xenon efforts since the shipping containers used for ^{85}Kr were also to be used for receiving the xenon from NRTS. However, xenon residues recovered and retained from the present and earlier ^{85}Kr campaigns at ORNL will provide a substantial portion of the hoped for amounts of xenon. These residues are, on the basis of reported analyses, essentially identical with the NRTS material.

Although it is not planned to make any efforts at investigating large scale uses until after satisfactory purity of a substantial quantity of the gas has been obtained, it is expected that material will be on hand in early September.

On the basis of the communication received in respect to an earlier request made to the Division of Radiation Protection Standards, the Food and Drug Administration will have to provide additional clearance in order to allow use of fission product xenon experimentally for human anesthesia; exemption will also have to be made to existing regulations before this xenon can be used in the other applications which are being considered.

NONDESTRUCTIVE TESTING FOR IRRADIATION SOURCES

D. R. Newman

MULTIELEMENT TECHNIQUES

The improvements in the signal synthesis electronics mentioned in an earlier quarterly report⁽¹⁾ have been made and allow better contrast on the matrix-type multielement transducer CRT readout. Figure 1a illustrates the mapping of three 0.125 in. diameter partially drilled holes in a "cold" ^{60}Co irradiation source. Figure 1b illustrates the new feature of the electronics which allows the contrast to be reversed by merely flipping a switch. The NDT designer can thus choose the most appropriate display.

PULSED SCHLIEREN

The effectiveness of this program here has often been enhanced by taking advantages of other recent developments at BNW. Continuing this, we have used a new pulsed laser schlieren system to study the beam transmitted from the large transmitter in the multielement transducer system. This transmitter is a cylindrically focused 5 MHz transducer approximately 0.5 by 1.5 in. with a designed focal length of 1.5 in. To evaluate the epoxy lens, measure the focal length, and check for side lobes/diffraction effects, the new schlieren system was used.

The image of a side view of the sound radiated from the transducer is shown in Figure 2. The focusing effect, side lobes, and width of focal point can all be seen. The schlieren system itself is shown in Figure 3. The ultrasound travels down through the water, and the laser, which has an extremely short light pulse duration, is synchronously strobed so that the ultrasonic pulse appears to remain stationary. The ultrasonic waves deflect some of the light which is then imaged in the camera.

FIGURE 1a. 0.125 in. Diameter
Partially Drilled Holes
in Dummy ^{60}Co Strip

FIGURE 1b. Contrast Reversal of 1a

FIGURE 2. Pulsed Laser Schlieren Photo
of Focused Transducer

In addition to the transducer evaluations, the system can be used to model such things as weld configurations to show the NDT designer exactly where the various reflections are coming from.

MATERIAL EVALUATION STUDIES

Material evaluation studies include two areas: the study of (1) cladding properties such as ultrasonic velocity and attenuation or conductivity for eddy currents and (2) properties such as cladding thickness, oxide coating thickness, cold work, reaction zone thickness, etc. that are important to capsule design or compatibility study personnel.

FIGURE 3. Pulsed Schlieren System

The results of some other AEC programs, discussed below, indicate that the ultrasonic critical angle technique can be a valuable tool for measuring surface or near surface material properties.

The critical angle effect for ultrasound is basically analogous to the familiar optical effect where a wave incident on an interface at an angle θ_1 is refracted at a different angle, θ_2 , according to Snell's law:

$$\frac{\sin \theta_1}{\sin \theta_2} = \frac{V_1}{V_2}$$

where V_1 and V_2 are the velocities of the wave in medium 1 and 2, respectively.

This result is complicated in the ultrasonic case because sound can be propagated in various longitudinal and transverse modes which have different velocities. Also, the complex amplitude of the wave must be taken into account which considerably complicates the expression for Snell's law.

For the purposes of surface measurements, the critical angle of interest occurs when the incident longitudinal wave is converted to a surface, or Rayleigh wave. The problem then becomes one of accurately measuring this angle. This is most easily done by measuring the amplitude of the reflected wave; a sharp dip occurs in the amplitude at the Rayleigh critical angle. This dip was not predicted by classical wave theory (see Figure 4), but a new theory developed at Battelle-Northwest by Becker and Richardson⁽²⁾ fits the experimental evidence very well, allowing many applications to be put on a sound basis and suggesting several new ones.

Figure 5 indicates how the critical angle can be used to measure coating thickness. The amplitude of the reflected wave is shown plotted versus gold plate thickness on a stainless substrate, and the change in critical angle is quite large.

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FIGURE 4. Reflected Beam Amplitude Versus Incident Angle, Classical, Experimental, and New Wavemodel Results

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FIGURE 5. Rayleigh Critical Angle Versus Gold Film Thickness
(Stainless Steel Substrate Frequency = 16 MH_z)

The critical angle changes with coating thickness because the acoustic surface waves are not traveling entirely on the surface but must extend some finite distance below. The amplitude of the wave decays exponentially with depth; its $1/e$ point is approximately one wavelength (i.e., at 16 MHz, about 0.008 in. in gold and 0.015 in. in stainless steel). Since part of the wave is in the gold and part is in the stainless steel, which have different velocities, the critical angle of the combination will be intermediate between that of one or the other.

By varying the frequency of the ultrasound, and thus the depth of penetration, the optimum range and resolution of the technique can be selected. Figure 6 shows critical angle variation with copper platings only a few microns thick on an alumina substrate.

Because of this surface penetration effect, this technique can be used to measure many parameters that vary with depth or acoustic velocity, such as cold work, alloying, etc. Figure 7 shows a well-defined plot of critical angle versus hardness, as determined by a Knoop tester.⁽³⁾

Results such as these indicate that it may be possible to measure nondestructively such things as oxide coating or cladding thickness and perhaps determine something about the reaction zone. These parameters are of considerable interest to ongoing DID programs.

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FIGURE 6. Rayleigh Critical Angle Versus Copper Film Thickness (Alumina Substrate Frequency = 15 MH_z)

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FIGURE 7. Critical Angle Versus Knopp Hardness

ISOTOPE PRODUCTION EVALUATIONS

R. W. McKee

ISOTOPE DATA COMPILATIONS

Preparation of the isotope property tables was completed using the ISOTAB-II computer program. The properties of approximately 2000 nuclides and isomers are included in 14 tables consisting of ordered listings of the nuclides by half-lives and radiation energies for each of the primary decay modes.

A limited number of copies were prepared as a report entitled "Nuclide Tables" and this has been distributed for comment.

FISSION-PRODUCT PROFITABILITY STUDY

A study was commenced that has the objective of determining the time and circumstances when fission-product cesium and strontium will be attractive. As a first step in this study, visits and discussions were carried out with the firms actively involved with fuel reprocessing ventures. The information obtained is being used to develop the basis and assumptions for the study.

RADIATION CHARACTERISTICS OF CIRCULATORY
SUPPORT ISOTOPIC HEAT SOURCES

F. T. Cross and J. C. Sheppard

IN-PHANTOM DOSIMETRY MEASUREMENTS

Work was initiated on the determination of organ doses from implanted ^{238}Pu heat sources.

The topical report (BNWL-1489) on the "In-Phantom Dosimetry of Plutonium-238 Heat Sources in Relation to Circulatory Support Systems" is undergoing publication. The report conclusions are summarized below.

The dose rates from LASL-1 are satisfactorily calculated using the modified code QAD-P5A. It is expected that the computed photon dose rates will agree even more closely with the measured dose rates once the discrepancies in emission rate are removed and the dose buildup factors are determined more precisely. For practical purposes, no further improvement is needed in the neutron dose rate computations. The agreement with measured values is good and the neutron dose contribution is a small fraction of the total dose except for positions quite close to the source where the contribution to the dose equivalent would be about 50% if a neutron quality factor of 10 is chosen.

Future experimentation will establish the perturbation of prototypic heat source subsystems and source impurities on the tissue dose rates and will detail the doses absorbed by various organ systems.

The previous measurements on promethium heat sources indicated that a nominal 15 W source with 0.25 ppm ^{146}Pm impurity and weighing 1.2 lb would produce a dose rate at the level of the spleen (about 20 cm away) of 20 to 35 mrem/hr depending on the source orientation. At the same location, the nominal 30 W plutonium source with shields weighing

An essential step in developing this seabed mineral exploration technology is an in situ feasibility demonstration of the technique. Using this small ^{252}Cf source, a feasibility demonstration was conducted at the Battelle-Northwest Marine Research Laboratory at Sequim, Washington. For this study, 200 lb samples of ore or minerals to which known amounts of silver, gold, manganese, and copper had been added were placed in the tideland ocean floor during low tide. These mineral samples were contained in cutoff 55-gallon drums approximately 25 cm deep by 56 cm in diameter (see Figure 10). To allow precise positioning of a ^{252}Cf source and the Ge(Li) diode detector, the tops of the barrels were covered with a plywood disk which contained a 15 cm diameter center position hole. Both the source and the detector were contained in 15 cm diameter stainless steel cylinders which were precisely positioned over the center of the mineral samples during their respective exposure and counting periods. Figure 11 shows the minerals in their containers with the plywood covers in place. Also shown in this figure is the 1.5 m long by 46 cm diameter tank which contained the diode, the liquid nitrogen reservoir, and associated electronics. The diode is actually located in a short 15 cm diameter extension on the bottom of this tank. Figure 12 shows the measurements in progress during high tide when the samples were covered with 1.2 to 1.5 m of water. The objective of these studies was to determine if any unforeseen problems existed with in situ measurements which would affect the practicality or sensitivity of elemental measurements in seabed minerals.

Feasibility studies were very successful and did not indicate any sensitivity limitations other than those observed in the laboratory. For example, there was no significant interference from the production of radiosodium and radiochlorine either in the surrounding water or in the mineral sample itself.

Figure 13 shows the gamma-ray spectrum obtained from one of these samples, a dunite sample containing about 0.05% silver. This sample had received a 2 minute irradiation with the 210 μg ^{252}Cf source and was allowed to decay 36 sec prior to a 200 sec counting period. It is obvious that silver, as well as the manganese which was present at about 0.1%, is easily measurable. With a 10-fold larger source, a 2-fold more efficient diode, and a closer mounting of the diode to the container face, it is estimated that these elements could be measured with good precision at 10- to 100-fold lower concentrations. Measurements of gold, copper, and other elements in these samples were also successful. These studies indicated that the sensitivities for in situ measurements of elements in minerals will be similar to those given in Table 14 of the last quarterly report; thus, many of the precious elements can be measured at concentrations of a few ounces per ton.

FIGURE 10. Mineral Samples Contained in Cutoff 55-Gallon Drums

FIGURE 11. Containers with Covers in Place. (Also shown are tank containing diode, the liquid nitrogen reservoir, and associated electronics.)

FIGURE 12. High Tide Measurements in Progress

FIGURE 13. Gamma-Ray Spectrum of Silver-Bearing Dunite on the Ocean Floor

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