PACIFIC NORTHWEST LABORATORY
ISOTOPE DEVELOPMENT PROGRAM
QUARTERLY REPORT:
FEBRUARY - APRIL 1973

Battelle

Pacific Northwest Laboratories
Richland, Washington 99352

MAY 1973

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PACIFIC NORTHWEST LABORATORY
ISOTOPE DEVELOPMENT PROGRAMS
QUARTERLY REPORT: FEBRUARY - APRIL 1973

Compiled by J. H. Jarrett

May 1973

BATTELLE
PACIFIC NORTHWEST LABORATORIES
RICHLAND, WASHINGTON 99352

CONTENTS

INTRODUCTION	1
SUMMARY	2
STRONTIUM FLUORIDE COMPATIBILITY.	5
MEDICAL ISOTOPE GENERATOR	8
BIOMEDICAL SOURCE DEVELOPMENT PROGRAM	10
CIRCULATORY SUPPORT SYSTEMS	14
Dosimetry Studies	14
Radiological Engineering Studies	15
ADVANCED SOURCE DEVELOPMENT PROGRAM	16
NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION	18
ATLANTIC RICHFIELD HANFORD COMPANY (ARHCO) FISSION PRODUCT ACTIVITIES	21

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INTRODUCTION

This report, BNWL-1308-14, is the 26th 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 1308-13.

This report covers the three-month period, February through April 1973.

SUMMARYSTRONTIUM FLUORIDE COMPATIBILITY

The compatibility tests with high-purity inert SrF_2 were completed. Evaluation of the 4400-hr test capsules is now under way, while evaluation of the 1500-hr capsules has been completed. The 1500-hr tests with WESF grade $^{90}\text{SrF}_2$ have been completed and the couples are being sectioned. The 4400-hr tests are continuing. The high-purity $^{90}\text{SrF}_2$ couples have been fabricated and testing will begin in May. Testing will also begin in May on couples fabricated from high-purity inert SrF_2 with controlled levels of impurities.

MEDICAL ISOTOPE GENERATOR

A system for proof-of-principle testing of americium-boron mixtures as a source of ^{13}N has been designed and assembled. Studies on optimization of emitter-target configuration, nuclear reactions which compete with ^{13}N formation, and design requirements for a practical ^{13}N generator have been initiated.

BIOMEDICAL SOURCE DEVELOPMENT PROGRAM

The objective of this program is to design and fabricate portable sources for blood irradiation. A ^{90}Sr - ^{90}Y source has been designed, built and partially characterized in support of an AEC-DBER program (Dr. F. P. Hungate, PNL, Principal Investigator). The irradiator achieved the main design goal of a transit dose in the range of 10 to 100 rads with a flow rate of 100 ml/min. It will be evaluated further in the next period.

CIRCULATORY SUPPORT SYSTEMS

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

Improved input data and an ancillary code developed for specification of source strength and spectra is being used in the dose rate calculations.

The conceptual designs of noncirculatory heat exchangers for abdominal implantation of heat sources has been completed.

ADVANCED SOURCE DEVELOPMENT

A special sputter-etch run has been completed which demonstrates that the substrates can be heated enough in a 10-min etch period, prior to platinum containment layer deposition, to promote excellent adherence between the AmO_2 and containment layer.

Information was gathered on costs to set up a complete sputtering laboratory with required equipment.

We have been investigating equipment which would best suit our needs to further automate the present sputtering systems.

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

Open ocean tests of the in situ seabed probe suggested necessary modifications to the probe assembly. These consist of moving the power supplies for the light ballast, the TV preamplifier, the detector bias, and the detector preamplifier from topside to a position on the probe. In addition, the ground loop noise interference which hampered the early test has been eliminated using an armored cable containing two micro-coaxial cables, three No. 12 wires for power and ground, and five No. 18 wires for control.

ATLANTIC RICHFIELD HANFORD COMPANY (ARHCO) FISSION PRODUCT ACTIVITIES

The Hanford strontium waste prototype capsule cracked when subjected to the Navy hydrostatic qualification test. The failure is attributed to insufficient strength to withstand this pressure as well as the low ductility of the heat-treated container.

ARHCO can supply thicker outer containers on a full cost recovery basis.

STRONTIUM FLUORIDE COMPATIBILITY

H. T. Fullam

At Hanford, strontium will be separated from the high level waste, converted to the fluoride, and doubly encapsulated in small high-integrity containers from subsequent long-term storage. The fluoride conversion, encapsulation and storage will take place in the Waste Encapsulation and Storage Facilities (WESF). The encapsulated strontium fluoride represents an economical source of ^{90}Sr if the WESF capsule can be licensed for heat source applications under anticipated use conditions. The objectives of this program are to obtain the data needed to license the $^{90}\text{SrF}_2$ WESF capsules. The information needed for licensing can be divided into three general areas:

- 1. Long-term SrF_2 compatibility data*
- 2. Chemical and physical property data on $^{90}\text{SrF}_2$*
- 3. Capsule property data such as external corrosion resistance, crush strength, etc.*

The current program is designed to provide the required information.

All of the tests with high-purity inert SrF_2 have been completed. Couples tested for 4400 hr have been section for metallographic examination and analysis. The couples tested for 1500 hr have been examined, and the metal attack, as determined by electron microprobe, scanning electron microscope, and photomicroscopy, is given in Table 1. Tungsten and TZM samples showed no evidence of attack. The test samples were identical in appearance to reference samples tested at the same temperatures for 1500 hr without SrF_2 present. The Haynes 25 and Inconel 600 samples showed very little attack. The photomicrographs indicated only a slight roughening of the metal surface. The other metals all showed significantly more attack. Hastelloy C-276 was badly corroded by the SrF_2 , especially at the higher temperatures. The samples showed extensive subsurface void formation and intergranular corrosion.

The 1500-hr tests with WESF grade $^{90}\text{SrF}_2$ were completed. The capsules are now being sectioned for evaluation.

TABLE 1. Metal Attack Found in High-Purity SrF_2 Couples
Tested for 1500 hr

Metal	Depth of Metal Affected, mils (a)		
	800°C	1000°C	1100°C
Hastelloy C-276	1	4	6
Hastelloy X	1	1	2
Hastelloy N	2	3	<1
Haynes 25	<<1	<<1	<<1
Haynes 188	2	2	2
Inconel 600	<1	<1	<1
Inconel 625	1	3	2
TZM	0	0	0
Tungsten	0	0	0

a. As determined from photomicrographs, electron microprobe analysis, and scanning electron microscopy.

A total of 1600 g of high-purity $^{90}\text{SrF}_2$ was prepared by a combination of ion exchange and the WESF flowsheet. Samples of the high-purity $^{90}\text{SrF}_2$ and WESF $^{90}\text{SrF}_2$ were sent to Argonne National Laboratory-West for impurity analysis by atomic adsorption. These analyses will supplement the onsite atomic adsorption analyses and other onsite analyses on the two materials.

The test capsules have been fabricated using high-purity $^{90}\text{SrF}_2$. Testing will start as soon as the couples have been sealed in their protective jackets and transferred to D-cell in the 324 Building.

Work has started on fabrication of the test couples containing high-purity inert SrF_2 with controlled levels of impurities. A total of 12 impurities will be evaluated; H_2O , O, Si, Al, Ba, Ca, Cu, Fe, Mg, Mn, Na and Pb. The selection of impurities for testing was based on analysis of the $^{90}\text{SrF}_2$ prepared by the WESF flowsheet. The oxygen will be added as SrO and Si as SiO_2 . The cations are being added as the fluorides. The tests will be

carried out at three temperatures (800, 1000 and 1100°C) with the nine different metals for 1500 and 4400 hr. Testing individual couples for each set of conditions would require a total of 1296 couples (3 temperatures x 9 metals x 12 impurities x 2 concentrations x 2 time periods = 1296). Since it is impractical to test this many couples, a statistically designed experiment is being used to reduce the number of test couples to 216. Testing of the couples will start in May.

MEDICAL ISOTOPE GENERATOR

J. W. Bartlett and J. H. Jarrett

Medical diagnostics and research frequently use isotopes. Availability of isotopes is limited, however, by the supply system, i.e., isotope half-lives must be long enough to survive relatively long delays between generation and use.

The objective of this program is to investigate the technical and economic feasibility of an isotope generator that could be installed in a facility such as a hospital and thereby supply short-lived isotopes for immediate use. Availability of such a device could open new areas of isotopes-using diagnostics, eliminate unwanted aftereffects from long-lived isotopes, and reduce radioactive waste disposal problems.

Major emphasis has been placed on development of a generator to produce ^{13}N . Such a device could use previously developed technology for α -generated isotopes.

Work during the report period extended prior studies of design variables to determinations of specific development requirements, design and construction of an initial ^{13}N -release test apparatus, and design concepts for the ^{13}N source in a practical generator.

Major areas in which work will be required were defined to be

- Proof-of-principle ^{13}N generation and recovery
- Optimization of emitter-target configurations
- Evaluation of competing reactions
- ^{13}N generator design

Proof-of-principle ^{13}N generation and recovery will be done in an experimental system in which an Am/B source is tested for ^{13}N release. This system, which was designed and constructed during the report period, is basically a chamber, which can be evacuated and back-filled as necessary, and connected to radiation detection equipment. The test source will be placed in the chamber and emitted ^{13}N will be transported to the radiation detection equipment. Annihilation gammas will be the measure of ^{13}N . The design of this system attempts to reflect some of the variables (e.g., ^{13}N transport time) expected to be significant in a commercial unit.

Fabrication of the test source has been delayed because of other commitments for the sputtering equipment. However, the experimental sequence for this apparatus is expected to be completed in May.

Studies on emitter-target configuration optimization have resulted in two basic design concepts: hollow microballoons containing fissures which will allow ^{13}N to escape from the interior, and a void-metal composite (cermet) which would be made from the emitter-target materials. Both concepts should aid ^{13}N release from the source. The microballoons would be used in a fluidized bed system; preliminary calculations indicate that the fluidized bed container would have to be about the size of a volleyball. Comparative evaluations of these two concepts are under way.

Potential competing reactions (those that consume α particles but do not produce ^{13}N) have been identified. They include production of ^{19}F or ^{19}Ne (from oxygen incorporated in the cermet), poisoning by other isotopes of boron, and α reactions with Li produced by neutron absorption in ^{10}B . Neutron capture reaction products may also compromise ^{13}N detection. Evaluation of the effect of these reactions is also under way.

^{13}N generator design is expected to be affected by factors such as

- neutron thermalization, utilization, and shielding
- heat removal
- ^{13}N removal from source and transport to use site
- safety
- size requirements.

Preliminary evaluation of these design factors was begun during the report period but no firm conclusions have been obtained. Their effect on a practical design will be determined primarily by the results of the source tests, the optimum source configuration, and the magnitude and effects of competing reactions.

BIOMEDICAL SOURCE DEVELOPMENT PROGRAM

F. P. Hungate, W. F. Riemath and J. H. Jarrett

The objective of this program is to design and fabricate portable sources for blood irradiation in support of an ongoing AEC-DBER program (Dr. F. P. Hungate, PNL, Principal Investigator) to evaluate extracorporeal 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.

Efforts in the report period were concentrated on the design, manufacture and testing of a ^{90}Sr - ^{90}Y irradiator. The device was designed using the "BETBLUD" computer code developed by Slatkin and Robertson. The basic device parameters are listed below:

Total Activity:	2 Ci, ^{90}Sr - ^{90}Y
Length of Active Region:	1 cm
Transit Dose:	~40 rads at 100 ml/min flow rate
Surface Dose at Contact:	50 to 300 mrem/hr
Silastic Tubing Characteristics	
ID:	0.105 in.
OD:	0.145 in.
Wall Thickness:	0.020 in.
Source Containment Layer:	Aluminum foil, 0.001 in.
Shielding Material:	High density tungsten alloy
Total Weight:	~4 lb

The transit dose indicated above was measured by pumping a NaCl stabilized Fricke dosimeter fluid through the silastic irradiator tubing at 100 ml/min. The absorbed dose was then determined colorimetrically with a spectrophotometer.

Several observations can be made on blood irradiators active with beta emitters. The code "BETBLUD" provides a good tool for the design of this type irradiator. The design of a ^{90}Sr - ^{90}Y irradiator to achieve a 40 rad transit dose while maintaining low mass portability and acceptably low external surface dose rate has been demonstrated. Radiation damage to the silastic tubing in this irradiator has been observed and must be given serious consideration in design of future irradiators.

The total useful life of the tubing in the present irradiator is limited to a period of days in any one position within the irradiator.

It may be noted that a much smaller percentage of the energy impinging on the tubing would be deposited in the tubing wall in a blood irradiator active with a photon emitter and that the material problems associated with the capability of the tubing to withstand the radiation would be of a smaller magnitude. On the other hand, the external shielding problems would be greater.

Figure 1 shows the irradiator half-shells opened while Figure 2 shows the device closed. The silastic tube through which blood will flow is not shown. An outer housing will be added to facilitate handling of the device. The outer housing design and the testing protocol is being worked out in close cooperation with Dr. Frank Hungate.

Future work on thulium and promethium irradiators is being planned. This effort will benefit markedly from the experience gained on the ^{90}Sr - ^{90}Y irradiator. As mentioned in previous reports, we anticipate that the thulium irradiator will be assembled cold and then activated by neutron irradiation. The promethium irradiator will present challenges since the isotope containment layer and the blood compatible layer must be thick enough to be effective yet thin enough to allow passage of the promethium beta radiation.

The following two abstracts were submitted and accepted for presentation during the quarter:

B. I. Griffin, F. T. Cross, "An Extracorporeal Blood Irradiator Source Study," 18th Annual Health Physics Society, June 17-21, 1973, Miami, Florida.

F. P. Hungate, B. I. Griffin, F. T. Cross and W. F. Riemath, "Improvements in Design of a ^{90}Sr - ^{90}Y Portable Blood Irradiator," 26th Annual Conference on Engineering in Medicine and Biology, Minneapolis, Minnesota, September 30 - October 4, 1973.

FIGURE 1.

FIGURE 2.

CIRCULATORY SUPPORT SYSTEMS

F. T. Cross and M. G. Zimmerman

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 source holders 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

Because of a change in personnel previously involved in the dose-rate calculations, some effort has been spent to clarify the parameters and data used as input to the modified QADP-5A code. We have successfully duplicated some of the previous calculations but are uncertain as to why certain normalizations in the data have been made. Therefore, we are rerunning a few of the earlier experimental-calculational correlations. In the process, we will be using improved data and a code developed in another project for specification of source strength and spectra, including the daughter products, as a function of source aging.

When satisfied that the code is being used properly, we will continue the computations of dose rates to organ systems and whole body from prototypic heat sources.

RADIOLOGICAL ENGINEERING STUDIES

The conceptual designs of noncirculatory heat exchangers for abdominal implantation of heat sources have been completed. Shams of these devices will probably be implanted in miniature swine prior to actual fabrication of the exchangers to determine whether the animal can accommodate their shape and size.

ADVANCED SOURCE DEVELOPMENT PROGRAM

W. F. Riemath and 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.

As discussed in the report for the previous period (BNWL-1308-13) poor adherence was indicated between the sputtered americium oxide active layer and the sputtered platinum containment layer when americium oxide deposition times were extended from 50 min to 15 hr (Runs AP-16, 17) and from 50 min to 4 hr (Run AP-18). In a previous source preparation run (AP-15B) where americium oxide was deposited for only 50 min, excellent adherence between the two layers was demonstrated when the substrates were heated by sputter etching, both before and after deposition of the americium oxide layer. It appears possible that during extended americium oxide deposition runs the sources cooled down enough so that when they were sputter-etched prior to deposition of the platinum containment layer, they still were not raised to a temperature high enough to promote good adherence. The needed higher temperature may be obtained by increasing the etch time immediately before deposition of the platinum layer.

This reasoning was investigated during February, starting with sources from Runs AP-14 and AP-18 that had demonstrated poor adherence between the sputtered americium oxide and sputtered platinum layers. The sources were placed in the sputtering chamber, sputter-etched for 10 min (about 2 times as long as in previous runs) and a platinum containment layer was then deposited. Excellent adherence between the americium oxide and the platinum layer resulted, indicating sufficient substrate heating.

Gamma radiation readings taken before and after the sputter-etching/platinum deposition steps indicated that the ratio of

$$\frac{\text{Rate of americium oxide removal by etching at 400 W (6-in. diam surface)}}{\text{Rate of americium oxide deposition at 200 W (4-in. diam target surface and 2-in. target substrate spacing)}}$$

is approximately 8 or less.

The glove box sputtering system has been cleaned and a new insulator replaced in the "J" assembly. Due to the many hours of sputtering and etching, the insulator became coated, which allowed undesirable arcing between the cathode and dark space shield.

The time between replacement of this insulator (approximately one year) is significant, as prior to purchasing the 6-in. "J" cathode assembly we were replacing insulators at very short intervals (one day to one month), thus the new cathode assembly has resulted in a substantial savings of program operating funds.

We have been investigating which equipment would be most desirable to further automate the present sputtering systems and provide for recording of system operating data such as forward and reflected power, argon pressure and Watt-hr of operation.

Information was gathered on costs to set up a complete sputtering laboratory with required equipment. This material will be used in a summary report on this program that will be prepared by the end of FY-1973.

NUCLEAR TECHNIQUES FOR SEABED MINERAL EXPLORATION

N. A. Wogman, H. G. Rieck, Jr., H. L. Nielson, J. R. Kosorok
and R. W. Perkins

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.

During the field evaluations of November/December 1972, in situ measurements were made of the depth to which a silver/copper/gold agglomerate could be detected with a 4-in. thick by 6-in. diam NaI(Tl) crystal or a 15% Ge(Li) diode as the detector of choice with 200 μg of ^{252}Cf as the neutron source. The higher resolution of the Ge(Li) diode identified the gamma ray energies of the elements at depths to 6-in., whereas the NaI(Tl) crystal could not resolve the separate gamma-ray lines. The NaI(Tl), however, could detect the activity as deep as 8 in. Laboratory studies of the attenuation of gamma-rays in typical and simulated sea floor sediments are being continued which, with the neutron depth gradients previously determined, will allow actual depths of detection to be calculated for a variety of sediment compositions and densities.

During the field evaluation of the seabed probe in 1972, design problems were noted which suggested changes that would enhance its operation. The modifications which have presently been completed or are underway consist of moving the power supplies for the light ballast, the TV preamplifier, the detector bias, and the detector preamplifier from topside to a position on the in situ probe.

There are two structural changes being made. The rotary actuator which was used to position the ^{252}Cf source and the Ge(Li) diode are being replaced by two hydraulic cylinders. The pivot point for the arm which provides a source to diode distance of 10 ft is also being moved to the central rotation point of the ^{252}Cf detector and Ge(Li) diode. Leakage of hydraulic fluid in the original rotary actuator resulted in movement of the detector assembly during its counting period, which required an electrically operated latch mechanism. The new system will employ the locking action of the hydraulic pump to hold the assembly in its fixed position.

These two structural changes will eliminate the 50-lb actuator, two electrical leads, the solenoid-operated latch mechanism, and the counter-balance weight used in the diode arm.

The Ge(Li) diode has also been updated. Its freon/propane solid cryogen has been replaced with freon-22 to eliminate any chance of fire if cryogen leakage occurs. A new, improved Richards valve was also installed to prevent the occasional diode vacuum leak which previously had plagued the system. The updated diode now has a resolution better than 3 keV for ^{60}Co at its operating temperature of -145°C .

In the Florida tests, a ground loop problem created degraded TV and diode signals approximately 30% of the time. An electrical modification has been undertaken to eliminate the cause of the degraded data. Two test cables were fabricated at Battelle-Northwest by taping signal and power leads together to be used in determining the amount of electrical shielding and the type of ground lines that are necessary to obtain reasonable signal-to-noise ratios for the TV camera and diode.

The first test cable has a braided shield around the ac power and the control signal pairs. This cable was tested in two configurations. The first configuration had two separate grounds, one for the signals and one to maintain earth ground; the other configuration had the signal and earth grounds common. The second test cable did not have the shielding for the ac power and signal lines and was tested in the same two modes as the first. This evaluation of the noise of the diode signal, the voltage drop through the power leads, and the hydraulic motor interference on both the diode and TV signal, indicated that both newly developed cable designs would eliminate the grounding problems. However, the voltage power loss in the cables dictated a system using fairly heavy power leads of 12 gauge wire. The new cable will thus consist of two microcoaxial cables with the characteristics of RG-59U for the TV and diode signals, one set of three No. 12 insulated wires for power and ac ground, and five No. 18 insulated wires for control purposes. This new cable will be exterior armored with two reversed layers of galvanized round wire capable of supporting 5000 lb of dead weight and applied to provide torsional balance of the finished cable. The cable will be wrapped on an electrically driven winch capable of a retrieval rate between zero and 30 ft/min on a 2000-lb load with a positive brake of 5000-lb strength.

These modifications will minimize the number of separate power leads required in the armored undersea cable. The required multiconductor cable for deep sea work is thus smaller and less expensive. These modifications should eliminate the ground loop electronic problems and allow a more simple delivery of the probe from the ship deck to its analysis position on the sea floor.

ATLANTIC RICHFIELD HANFORD COMPANY (ARHCO) FISSION PRODUCT ACTIVITIES

G. E. Benedict

The Atlantic Richfield Hanford Company (ARHCO) is presently recovering fission products, ^{90}Sr and ^{137}Cs , in the Waste Processing Facility (B Plant) for later encapsulation as SrF_2 and CsCl in the Waste Encapsulation and Storage Facility (WESF). These operations are under contract to the Atomic Energy Commission's Division of Production and Materials Management. ARHCO also has some engineering effort supported by the Division of Applied Technology (DAT) to provide current and projected information on the ^{90}Sr and ^{137}Cs production capabilities and product quality and availability. ARHCO has liaison responsibilities with Pacific Northwest Laboratory (PNL) and DAT to provide information which will aid possible licensing and commercial use of the $^{90}\text{SrF}_2$ capsules to be produced in WESF.

The Waste Encapsulation and Storage Facility (WESF) inert strontium fluoride capsule failed the Navy's 1000 bar (33,000 ft water) hydrostatic pressure test. The capsule was returned and sectioned. The failure is attributed to lack of capsule strength to withstand this pressure for which it was not designed, as well as the loss of ductility caused by the 1600°F heat treatment of the Hastelloy C-276 container material requested by the Navy.

ARHCO can supply these capsules with a thicker outer container, but the final tungsten inert gas welder would have to be replaced, and techniques devised to provide sufficient strength at the weld area with the thicker material. These costs would be funded on a full cost recovery basis by the user.

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