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Preparation of Actinide Specimens for the US/UK Joint Experiment in the Dounreay Prototype Fast Reactor

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Operations Division

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PREPARATION OF ACTINIDE SPECIMENS FOR THE US/UK JOINT EXPERIMENT IN THE DOUNREAY PROTOTYPE FAST REACTOR

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ABSTRACT

A joint research program involving the United States and the United Kingdom was initiated about four years ago for the purpose of studying the fuel behavior of higher actinides using in-core irradiation in the fast reactor at Dounreay, Scotland. Simultaneously, determination of integral cross sections of a wide variety of higher actinide isotopes (physics specimens) was proposed. Coincidental neutron flux and energy spectral measurements were to be made using vanadium encapsulated dosimetry materials in the immediate region of the fuel pellets and physics samples. The higher actinide samples chosen for the fuel study were ^{241}Am and ^{244}Cm in the forms of Am_2O_3, Cm_2O_3, and $Am_6Cm(RE)_7O_{21}$, where (RE) represents a mixture of lanthanides. Milligram quantities of actinide oxides of ^{248}Cm, ^{246}Cm, ^{244}Cm, ^{243}Cm, ^{243}Am, ^{241}Am, ^{244}Pu, ^{242}Pu, ^{241}Pu, ^{240}Pu, ^{239}Pu, ^{238}Pu, ^{237}Np, ^{238}U, ^{236}U, ^{235}U, ^{234}U, ^{233}U, ^{232}Th, ^{230}Th, and ^{231}Pa were encapsulated to obtain nuclear cross section and reaction rate data for these materials. The technology development and its application in the preparation of the fuel specimens and the physics specimens that are an integral part of this cooperative program are described in this report.

I. INTRODUCTION

Following earlier discussions, Mr. C. W. Blumfield (Director, Dounreay Experimental Reactor Establishment, Thurso, Caithness, Scotland) wrote in November 1977 to Dr. G. W. Cunningham (then Acting Program Director for Nuclear Energy, Department of Energy, Washington, D. C.) proposing a joint experiment on the irradiation of higher actinides in the Dounreay Prototype Fast Reactor (PFR) at Dounreay, Scotland (UK). Dr. Cunningham agreed to set up a technical meeting to draft a detailed proposal. This meeting was held at the Department of Energy (DOE) Headquarters, Germantown, Maryland, in March 1978 and was attended by K. M. Swanson and A. L. Mills (UK, Dounreay); P. B. Hemmig, J. Lewellen, S. Whetstone, and R. D. Walton (DOE); S. Raman, J. E. Bigelow, and M. L. Williams (ORNL, Oak Ridge National Laboratory); T. E. Weber and W. Little (HEDL, Hanford Engineering Development Laboratory); S. Beamans and C. Cowan (General Electric Company); W. Maeck and W. Emel (Idaho National Engineering Laboratory); and C. Johnson (Argonne National Laboratory). At this meeting a proposal for an experiment was drafted that was subsequently the basis of the "Higher Actinides Agreement" signed in May 1979 as part of the long-term cooperation in the field of Liquid Metal Fast Breeder Reactor (LMFBR) technology between the United States and the United Kingdom under the LMFBR agreement of 1976.

The original proposal from the UK (presented by Mills and Swanson) was primarily aimed at a preliminary evaluation of materials behavior for higher actinides in a fuel-type irradiation of gram quantities of selected actinides (fuel pellets). During the March 1978 meeting, Raman (ORNL) proposed an additional experiment in which milligram quantities of several actinides (physics specimens) would be irradiated simultaneously with actinide "fuels" in order to improve our knowledge of basic nuclear cross sections and reaction rates. Both the "fuel" and "physics" experiments were incorporated into the Higher Actinides Agreement.

Major responsibilities for implementing this Agreement were assigned to HEDL (E. T. Weber) and ORNL (S. Raman) by Dr. P. B. Hemmig, Chief, Physics Section, DOE Reactor Research Technology. As shown in Fig. 1,

ORNL was responsible for selecting and providing the isotopes, for fabricating the fuel pellets, for fabricating the ORNL and HEDL dosimeters and ORNL physics samples, and for loading and seal-welding the pin segments (with HEDL assistance). HEDL was responsible for test pin design, pin fabrication, final assembly of pin sections, quality assurance, and shipping to the UK. ORNL responsibilities pertaining to test pin preparation were successfully met by the middle of June 1981.

The US/UK Higher Actinide Irradiation Test consists of four fuel pins (FP) to be irradiated in the Dounreay PFR. The fuel pin configuration is shown in Fig. 2. The tentative irradiation scheme based on full power days is as shown in Table 1. Pins FP1 and FP2 are identical; pins FP3 and

Table 1. Tentative Irradiation Scheme for US/UK Higher Actinide Irradiation Test

Pin	Bottom Section	Top Section	Irradiation Time
FP1	Physics	Fuel	90 Full Power Days
FP2	Physics	Fuel	180 Full Power Days
FP3		Fuel	>180 Full Power Days
FP4	———Physics———		To be determined

FP4 are modified versions of FP1 and FP2 which were designed to permit extended irradiation periods, provided several other conditions are also met (for example, safety, reactor space availability, irradiation rig availability, etc.). The engineering test plan and fuel pin designs are contained in two reports issued by Basmajian of HEDL[1,2].

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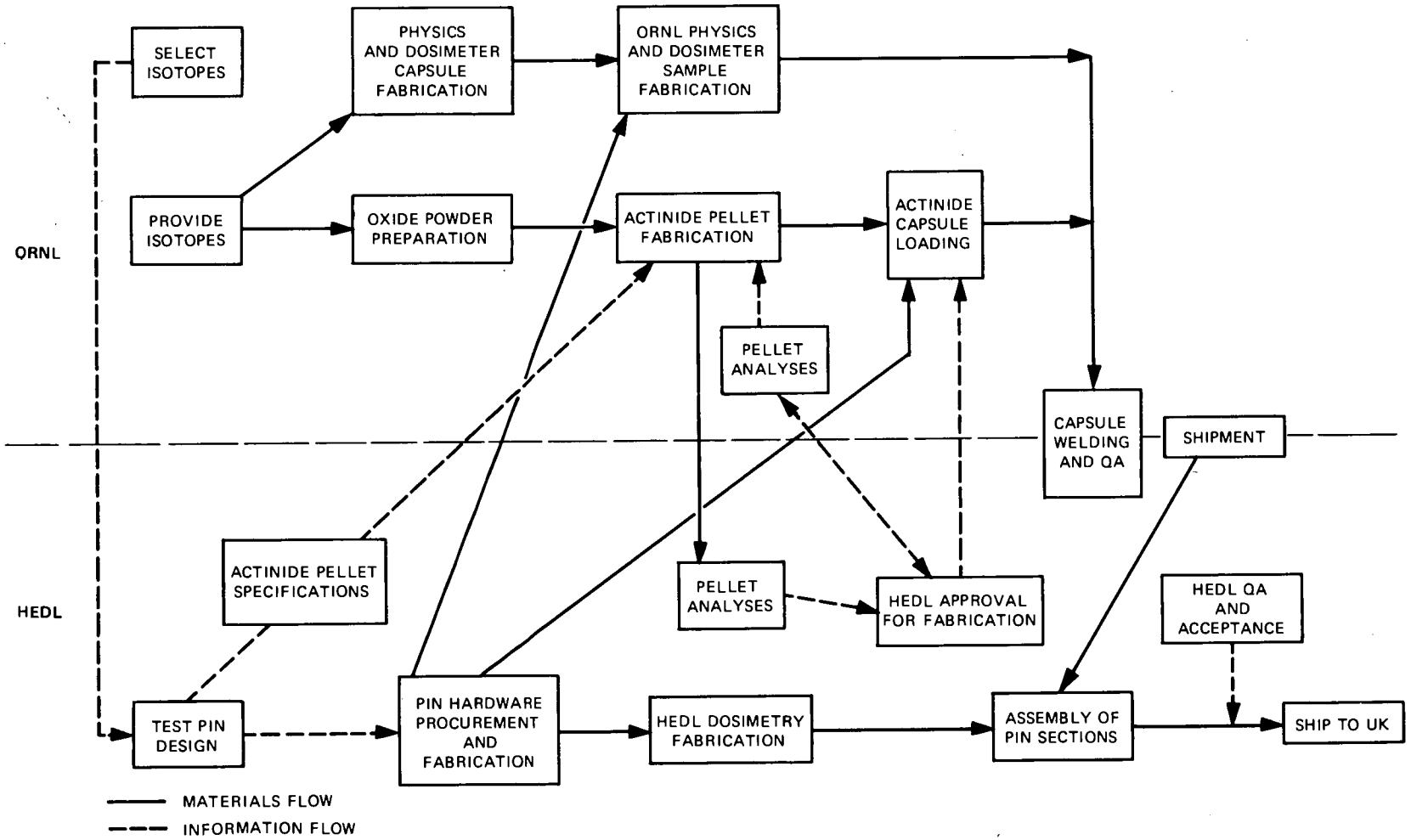

Fig. 1. US/UK Higher Actinides Irradiations Logistics for Test Pin Preparation

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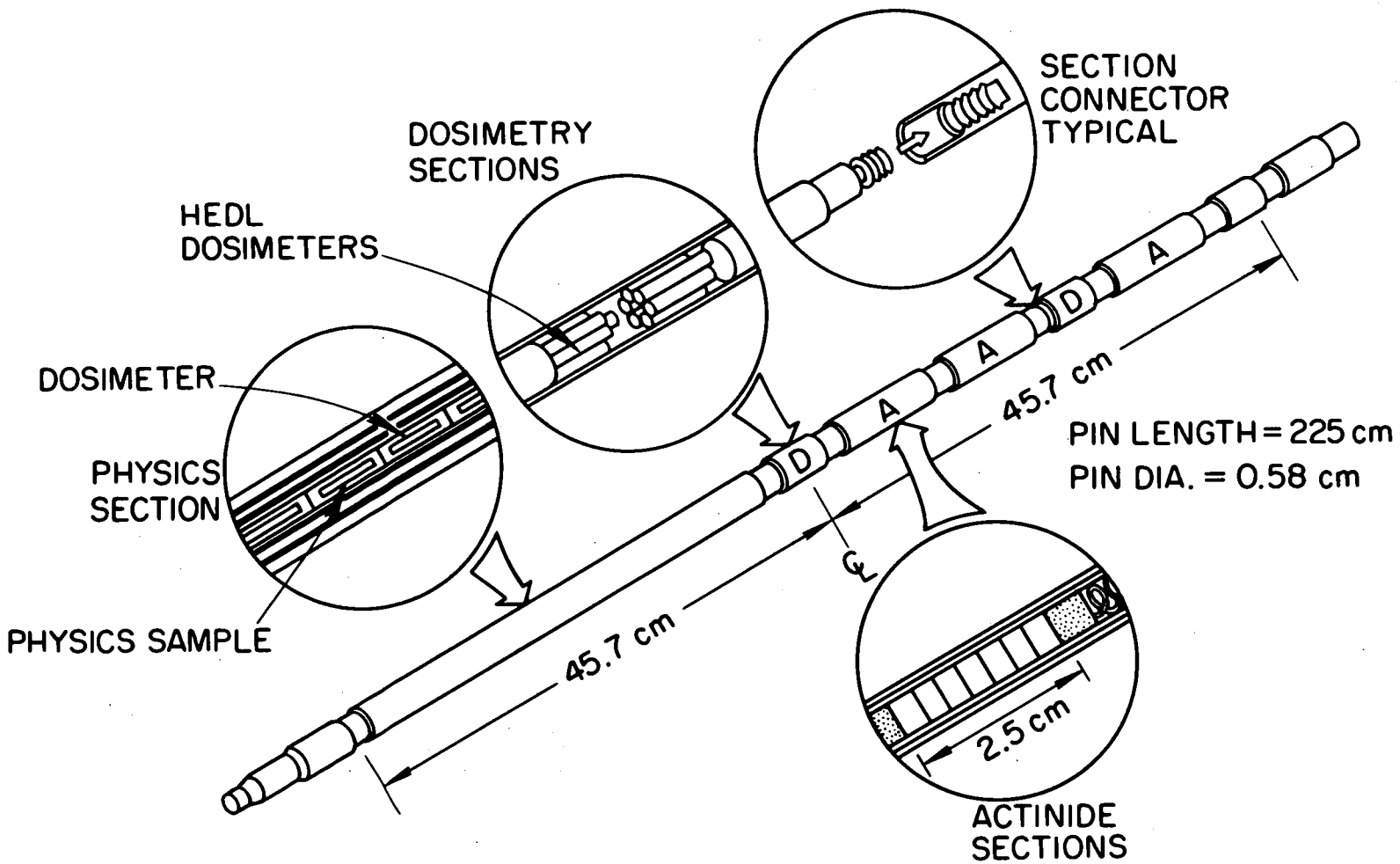

Fig. 2. Higher Actinides Pin for PFR Irradiation

The present report is a detailed description of the ORNL efforts in fabricating the fuel pellets and the physics specimens. In many respects, the fuel pellet fabrication efforts were of a pioneering nature and therefore warranted extended documentation and sharing of the experience. This report deals only with a special facet of the US/UK Higher Actinides Program. Additional information concerning the main objectives of this program, design of the experiment, more details on pin fabrication, post-irradiation examination, and experimental results will be described in future reports originating from ORNL, HEDL, and the UK. A more detailed characterization of the unirradiated samples will be contained in a separate ORNL report.

II. TECHNOLOGY REQUIREMENTS

Involvement of the Isotope Research Materials Laboratory (IRML) at ORNL in the cooperative US/UK Higher Actinide Experiment resulted from its historic experience in fabricating actinide oxides into many physical shapes for research purposes. Of greatest importance was the IRML development of the urea coprecipitation process.[3] This process permits the formation of pure or mixed oxides in an atomically homogeneous form with highly controlled particle size. Precipitated oxides prepared by this process have the additional quality of being densified to nearly theoretical values at temperatures of 150-200 K less than the temperatures normally required for densifing unprecipitated materials.

IRML personnel have many years of experience in operating shielded facilities (water-shielded hot cells) and glove box complexes with internal inert atmosphere control. Most of these facilities were fabricated in-house so that design and selection of structural materials for this project would be of minimal concern and would permit relatively low expenditures for equipment fabrication. In past years, IRML personnel have produced extruded and sintered oxide rods, ribbons, and "wire" of <0.5 to 1.0-mm diam as well as many hot-pressed forms (spheres, cylinders, dishes) of actinide and stable isotope compounds. This experience was directly applicable to the formation of fuel pellets needed for this program.

Finally, specific IRML experience with americium and curium oxide sample fabrication precluded an extensive learning period of trial-and-error manipulations. Expertise in building and operating miniature hot presses also reduced time and cost considerations. In general, most quality assurance was already available and in action for an operation of the type required to fabricate the fuel pellets.

With regard to preparation of neutron dosimetry materials and physics specimens for cross-section measurements during the UK irradiations, IRML has been the developer and sole supplier of high purity vanadium-encapsulated stable and radioactive isotopes used for dosimetry at the FFTF and EBR-II reactors for more than ten years. More than 8000 dosimeters containing powdered oxides or extruded and sintered "oxide wires" of actinide oxides have been produced and an associated quality assurance package was written by IRML personnel to qualify and quantify these samples and their contained isotopes.

The physics specimens used in this program were all prepared by loading small quantities (1-20 mg) of the actinide oxides as powder into 1.5-mm o.d. vanadium capsules. IRML facilities and capabilities, as related to dosimetry, permitted the use of already existing glove boxes, microbalances, related hardware, counting equipment and manpower for the preparation of these samples; such availability significantly reduced the cost and time compliments of this overall task.

III. ACTINIDE MATERIAL SPECIFICATIONS

A. Fuel Pellet Specifications

Materials selected and samples fabricated for the Joint US/UK Higher Actinide Irradiation Experiment were required to meet stringent specifications. The specifications for the actinide fuel and physics specimen materials are detailed in HEDL Report TC-1933.[1] Requirements for the ^{241}Am and ^{244}Cm fuel materials are summarized in Tables 2-5.

Table 2. Three Actinide Oxide Pellet Types Required for US/UK Experiment

Pellet Type	Actinide Oxide
I	$^{244}Cm_2O_3$
II	$^{241}Am_2O_3$
III	M_2O_3*

*$M = {}^{241}Am_6\ {}^{244}Cm\ RE_7$ where RE_7 corresponds to a mixture of stable lanthanide elements: La, Ce, Nd, and Sm in the atomic ratios of 1:2:3:1, respectively.

Table 3. Specifications for Fissile Actinide Content, Isotopic Content, and Oxygen-to-Metal (O/M) Ratio for US/UK Fuel Materials

Pellet Type	Fissile Actinide to Total Metal	Isotopic Content	O/M
I	>85%	^{244}Cm >85%	1.50 $\pm$0.02
II	>85%	^{241}Am >85%	1.50 $\pm$0.02
III	45% $^{+5\%}_{-3\%}$	^{244}Cm >85% of Cm ^{241}Am >85% of Am	1.50 $\pm$0.02

Table 4. Maximum Impurities for US/UK Fuel Materials

Impurity	Quantity (μg/g)
Iron + Nickel + Chromium	3000
Aluminum + Calcium	750
Boron	20
Carbon + Sulfur	500
Cadmium	20
Chlorine + Fluorine	65
Europium, Gadolinium, Dysprosium	1000
Moisture	50
Total Impurities Should be Less Than	5000

Table 5. US/UK Higher Actinide Irradiation Experiment Pellet Specifications

Pellet Type	Density[a]	Weight	Length (cm)[b]	Diameter (cm)
I	90 ±5%	Determined to ±0.5% (2 σ)	0.381 ±0.038	0.376 $^{+0.005}_{-0.003}$
II	90 ±5%	Determined to ±0.5% (2 σ)	0.381 ±0.038	0.381 ±0.003
III	90 ±5%	Determined to ±0.5% (2 σ)	0.381 ±0.038	0.381 ±0.003

(a) Theoretical density for pellet types I, II, and III assumed to be 11.67, 11.89, and 10.06 g/cm^3, respectively.

(b) The column length of seven pellets to be 2.54 ±0.25 cm.

B. Physics Specimen and Dosimeter Specifications

Physics specimens and dosimeters to be used with the physics specimens were required to be encapsulated in a high purity material that would not produce an undesirable background after irradiation. Capsules made from high purity vanadium were used for this purpose (Figures 3, 4). A spark source mass spectrographic (SSMS) analysis of the vanadium is shown in Table 6.

Table 6. SSMS Analysis of High Purity Vanadium Used for US/UK Physics Specimens and Dosimeters[c]

Element	Weight (ppm)
Ag	5
As	2
B	5
Ca	0.3
Fe	100
K	1

Table 6 (Cont'd)

Element	Weight (ppm)
Mg	10
Mo	50
Na	2
Nb	10
Ni	20
P	20
Si	300
Ta	100
Ti	20
V	Major
W	40
Zr	10
S	100
F	0.5

[c]Elements not shown are below detectable limits.

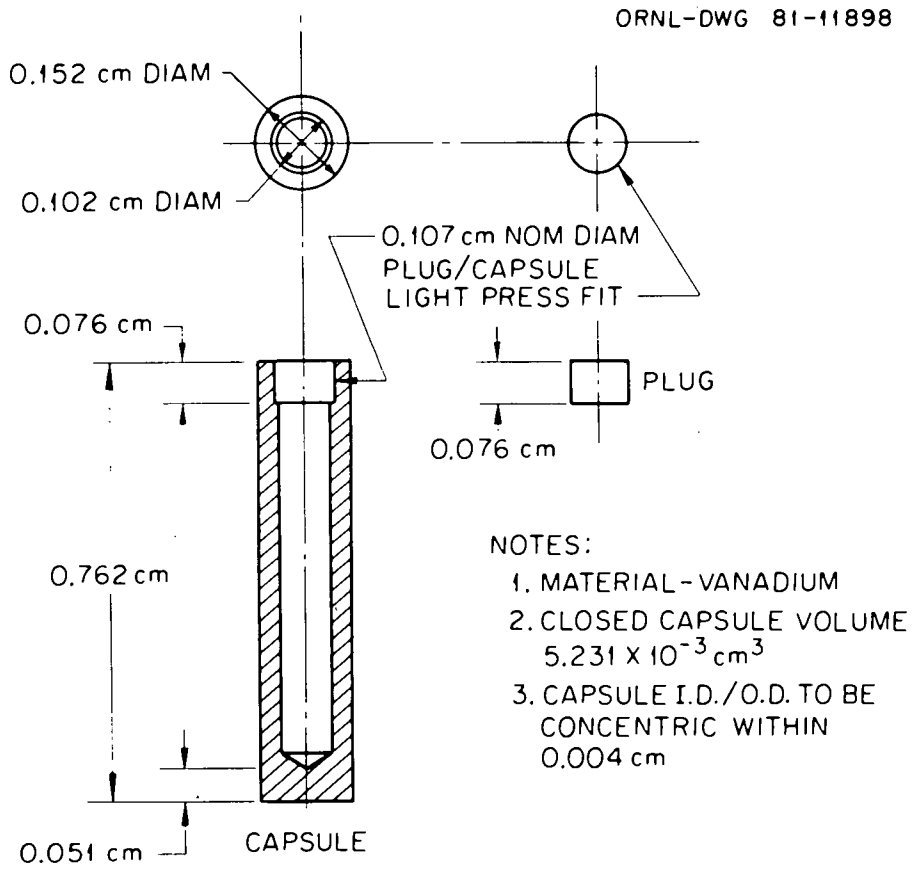

Fig. 3. High Purity Vanadium Capsules Used for Encapsulating Physics Specimen Materials

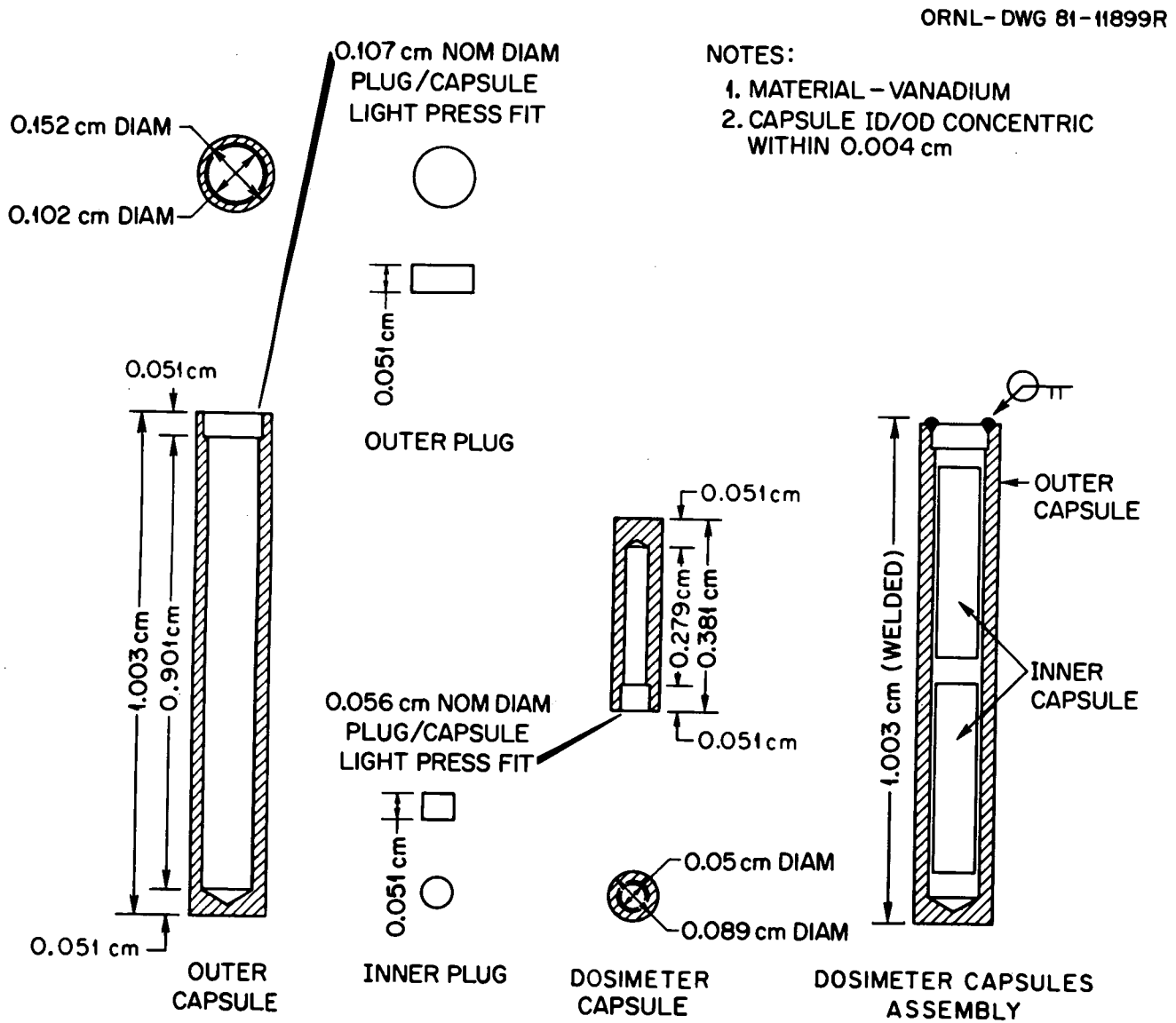

Fig. 4. High Purity Vanadium Capsules Used for Encapsulating Physics Specimen and Dosimeter Materials

Additional requirements for the vanadium capsules and encapsulated materials are detailed below:

a. All encapsulated materials are to be characterized by SSMS, isotopic, and concentration analyses,

b. All materials are to be loaded in a helium atmosphere,

c. Weight of encapsulated material is to be determined to the nearest microgram,

d. All vanadium capsules are to be identified by a dot code system (Table 7),

e. Each of the three sets of vanadium capsules is to be identified by a number scribed on the bottom of the capsule,

f. Each vanadium capsule is to be welded closed by tungsten-inert gas (TIG) fusion welding in a helium atmosphere,

g. Each welded capsule is to be heated to 1075 K in vacuum for one hour and then leak tested, and

h. The primary weight of actinide material in each capsule is to be verified by gamma-counting where applicable.

Table 7. Vanadium Capsule Dot Code Identification System
(Dots are placed on capsule near base. Capsules should be oriented horizontally with base of capsule located on the left and dots read from left to right for capsule identification. The ordinal numbers refer to capsules listed later in Tables 13-15.)

1.	.	10.	∴	19.	⁝.	28.	∴
2.	..	11.	∴	20.	∴	29.	⁝.
3.	⁝	12.	.⁝	21.	∴	30.	⁝.
4.	∴	13.	⁝	22.	.⁝	31.	∴
5.	.∴	14.	∴	23.	⁝.	32.	∴
6.	⁝	15.	⁝	24.	⁝⁝	33.	⁝⁝
7.	∴	16.	∴	25.	⁝⁝	34.	⁝⁝
8.	⁝	17.	⁝	26.	⁝.	35.	⁝⁝
9.	⁝∴	18.	⁝	27.	⁝⁝		

IV. FUEL PELLET FABRICATION AND CHARACTERIZATION

A. Equipment

Facilities utilized for the preparation of actinide powders, fuel pellet fabrication, characterization and welding included an existing manipulator cell and two stainless steel glove boxes, which were specifically designed and equipped for this project. Each of these facilities featured accessory equipment necessary for the containment of radioactivity by filtration and the maintenance of an internal negative pressure.

Manipulator Cell. The manipulator cell was a water-shielded enclosure equipped with Central Research Laboratory Model L manipulators and designed primarily for handling neutron-emitting materials. This facility was utilized only for the production of the three types of actinide powders used for fuel pellet fabrication.

Pellet Fabrication Glove Box. The pellet fabrication glove box, Figs. 5 and 6, was a commercially available vacuum glove box modified for this program. Utility services required for the operation of this facility consisted of electrical power, vacuum, inert gas purge, cooling water, atmospheric analysis equipment, pressure control and temperature monitoring equipment. Contained within this facility was a vacuum hot press, a tube furnace for oxide reduction, an analytical balance, a water-shielded storage facility and assorted metrology equipment. All inlet and exit gas lines to the glove box were filtered; cooling water which did not come in contact with the box interior surfaces was not filtered.

The vacuum hot press, Figs. 7 and 8, was fabricated from aluminum to achieve a low mass and superior heat conduction properties. The helical heating element in the hot press was machined from ATJ graphite; heat reflectors surrounding the element were fabricated from molybdenum sheet. Compressive force was supplied to the pressing die by a double-acting pneumatic cylinder mounted externally at the top center of the glove box. Force was transmitted through a water-cooled, double bellows assembly on the top flange of the vacuum shroud. Electrical power was supplied by a direct current power supply; 2.7 kW were required to attain the 1575 K pressing temperature required for this application. The equipment is capable of being operated at 2275 K. Temperature control and monitoring during operation of the press were accomplished using optical pyrometry through quartz viewing windows located on the vacuum shroud and on the glove box face. Primary cooling water was supplied to the hot press from a closed loop refrigerated system with a secondary emergency water supply using normal process water. The secondary supply system was automatically activated by a loss of flow in the primary system. Dies

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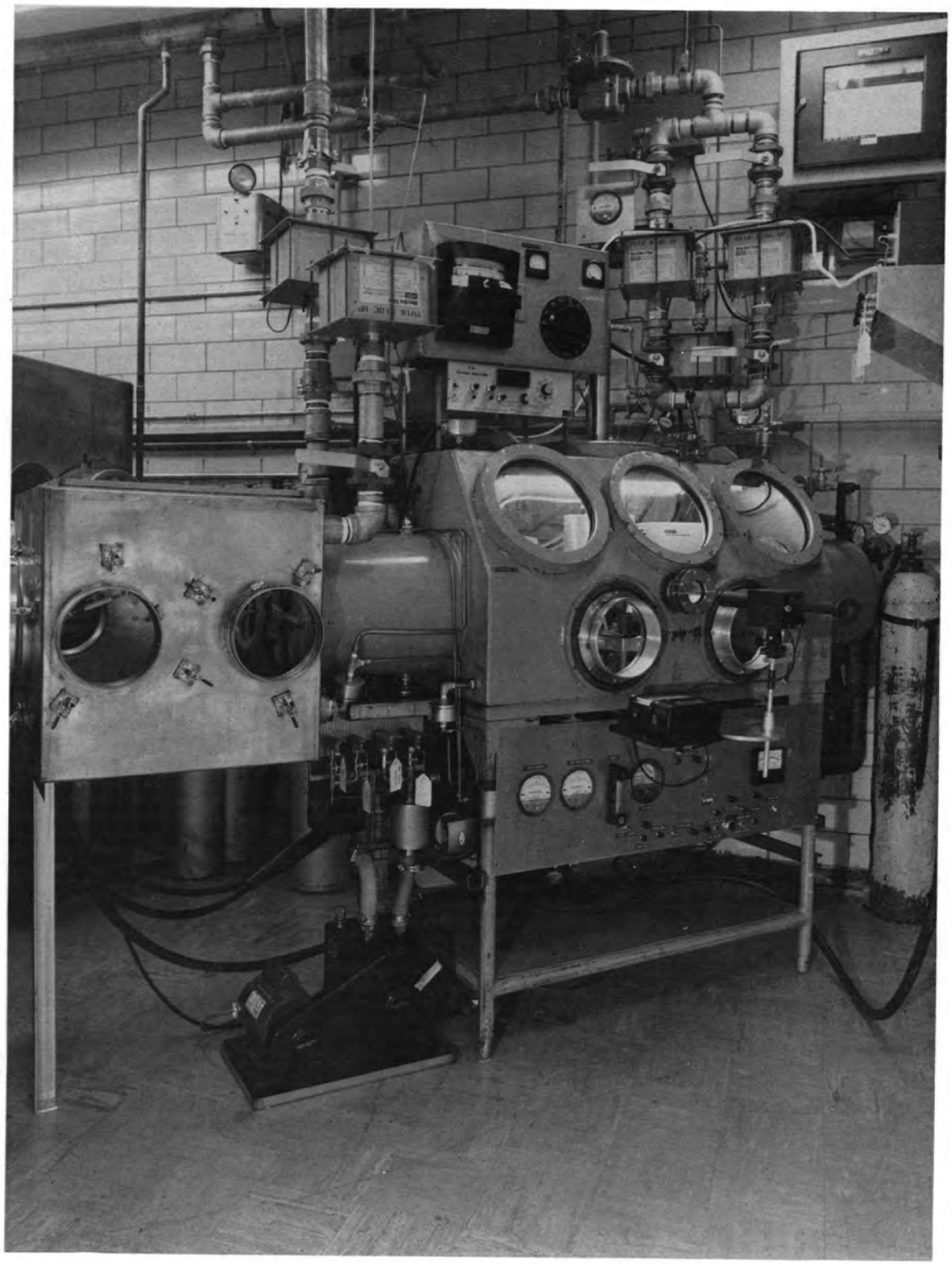

Fig. 5. US/UK Fuel Pellet Fabrication Glove Box

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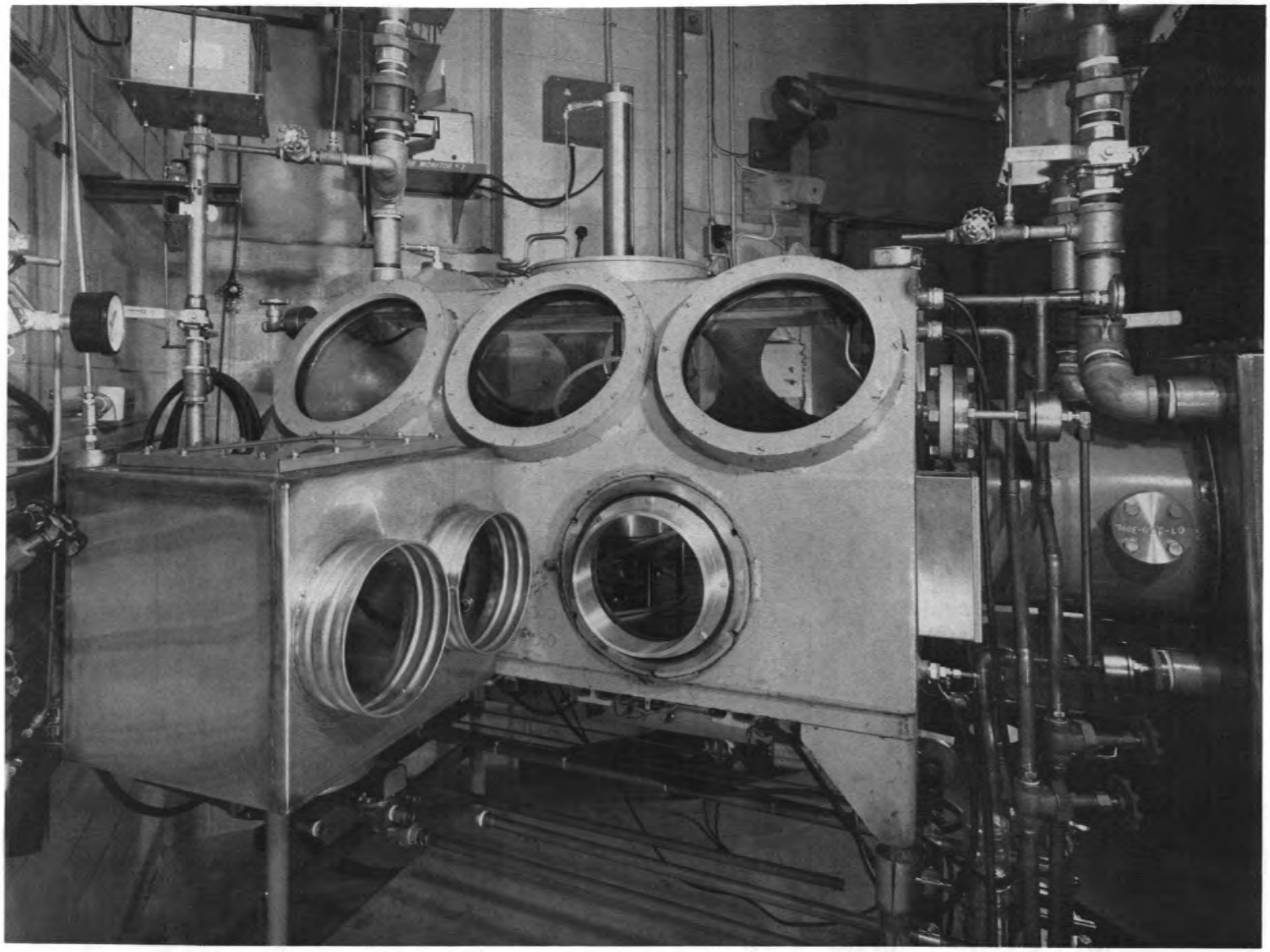

Fig. 6. US/UK Fuel Pellet Fabrication Glove Box and Appendage Loadout Glove Box

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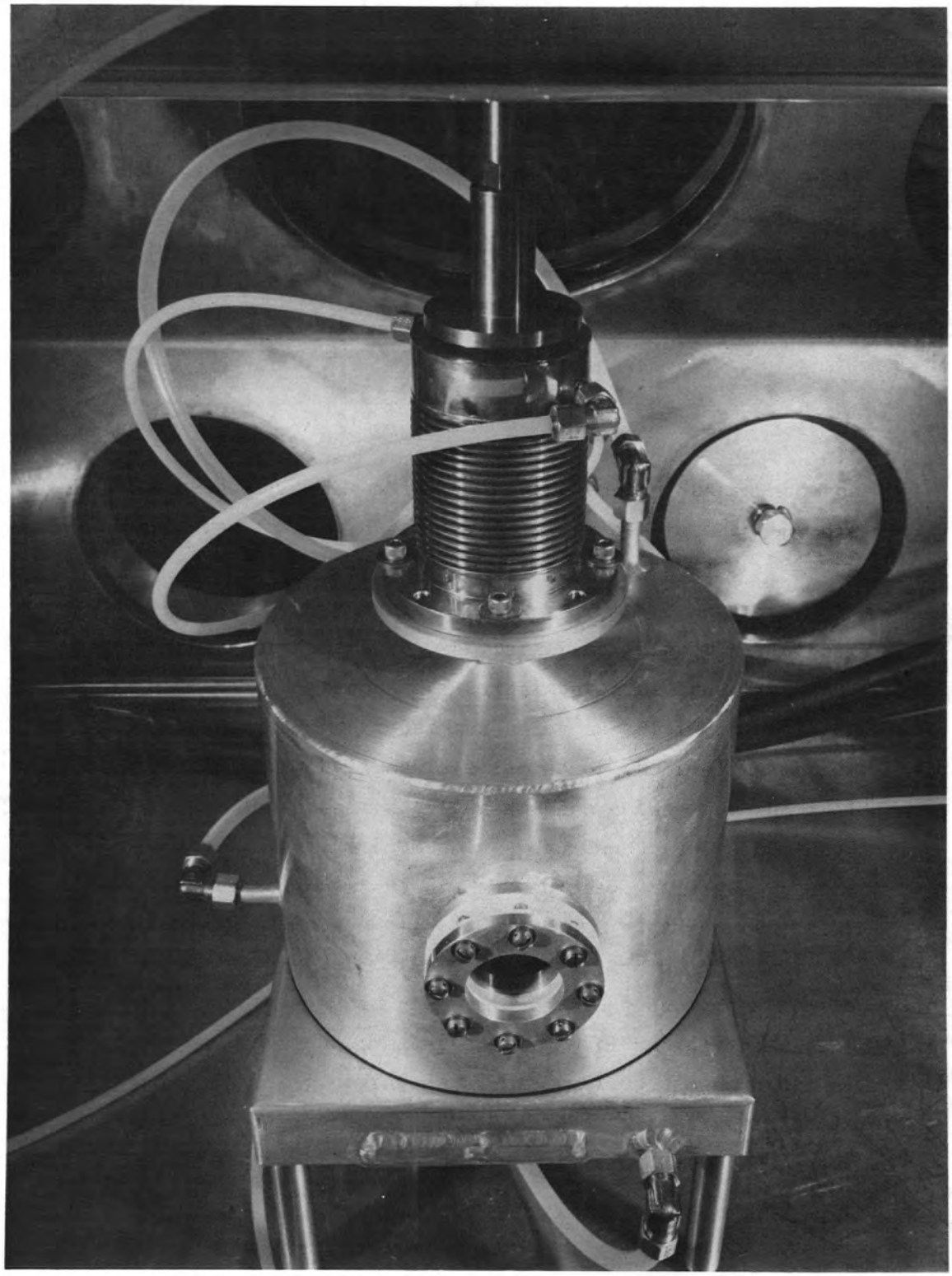

Fig. 7. Exterior View of Vacuum Hot Press Used in Preparing US/UK Fuel Pellets

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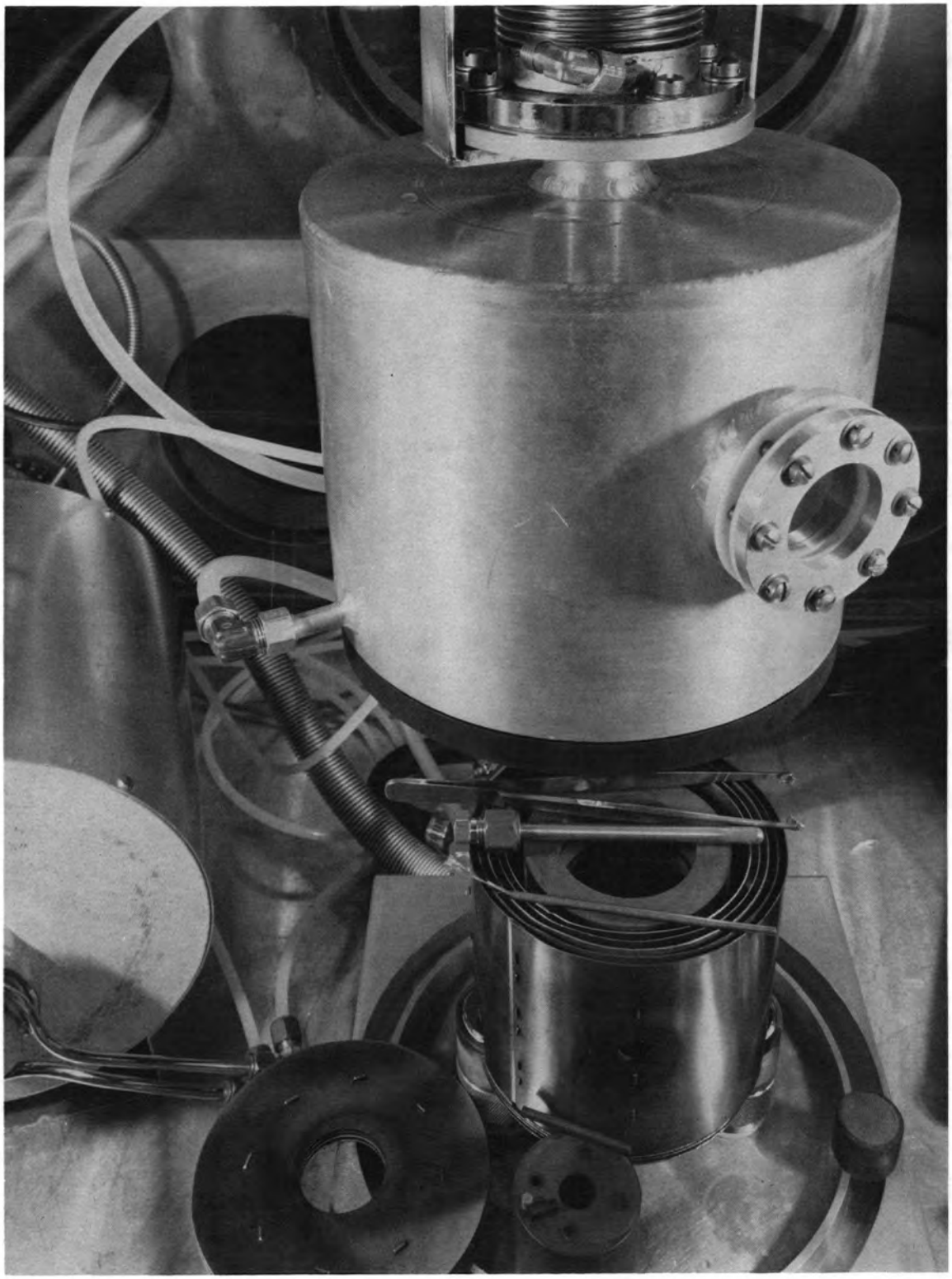

Fig. 8. Interior View of Vacuum Hot Press Showing the Helical ATJ Graphite Heating Element and the Molybdenum Heat Reflectors

for the hot press were fabricated from ATJ graphite, Fig. 9, and were constructed so that the densification of seven pellets could be performed simultaneously. Punches were fabricated from "Vitrecarb."[d]

The powder reduction furnace consisted of a platinum resistance heating element wound on an alundum center tube externally surrounded by packed ceramic fiber insulation. The required flow of reducing gas was supplied to the center tube interior through water-cooled "o-ring" seals at both ends of the furnace. The furnace temperature was maintained by a thermocouple-actuated power control.

The analytical balance, Fig. 10, was used for batching the powder that was eventually loaded into each individual die bore as well as for weighing individual finished pellets. The balance was modified to facilitate operation within the glove box.

The internal material storage facility, Fig. 10, consisted of a 38-cm diam by 38-cm long double-walled container attached to the right

ORNL Photo 3768-81

Fig. 9. ATJ Graphite Die and Vitrecarb Punches Used for Fabricating Seven Actinide Fuel Pellets Simultaneously

(d) Trade name of Fluorocarbon, Process Systems Division, Anaheim, CA.

ORNL Photo 1000-81

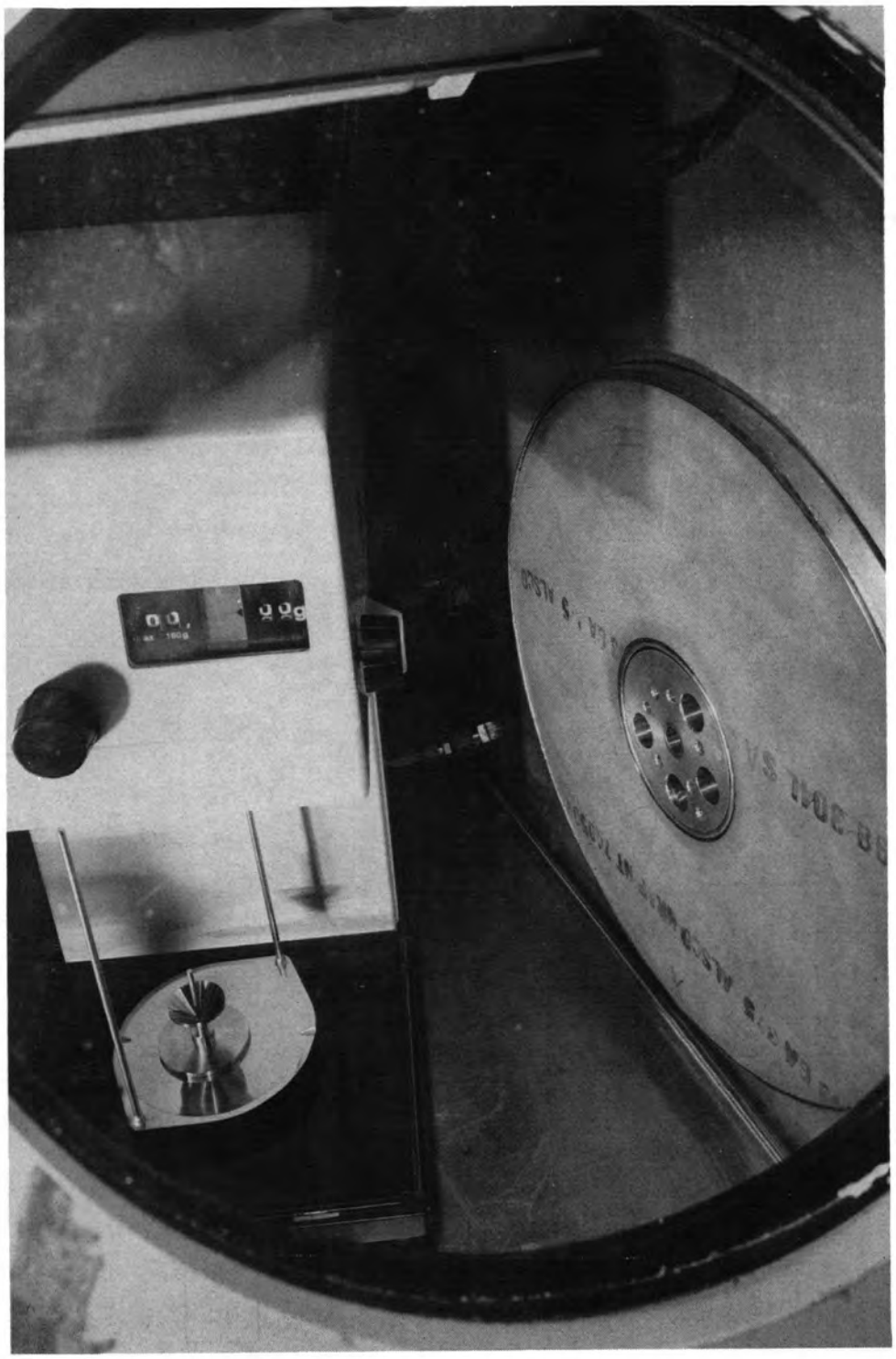

Fig. 10. Analytical Balance Used for Accurately Determining Weights of Fuel Pellets, and Storage Well Used for Storage of Actinide Materials to Minimize Radiation Exposure to Personnel

end of the glove box. The void between the double walls of this storage container was connected to the process water supply to provide 11.5 cm of water shielding (for neutrons) as well as for cooling to remove ^{244}Cm decay-heat. A 10-cm diam, 15.25 cm long copper block was fitted to the interior of the inner wall of the water shield and featured several bored holes to accept tubular containers of bulk actinide oxide powder and completed pellet storage cans.

A small appendage glove box was fitted to one side of the pellet fabrication glove box for the purpose of removing fully characterized pellets into a relatively contamination-free transfer container, Figs. 6, 11, and 12. This glove box was atmospherically isolated from the main pellet fabrication box and was equipped with separate negative pressure, inert gas purge and gas analyzer systems.

The welding glove box, Figs. 13 and 14, was a commercial system modified by the addition of an entry and exit glove box equipped with bag ports. Contained within the welding box was a tungsten inert gas welding torch (supplied by HEDL) and a secondary containment apparatus (Fig. 15) used for the transfer of pellets from the transfer containers to the fuel pins. This latter apparatus was connected to the building off-gas system to maintain an internal negative pressure of 2.5-7.5 kPa (10-30 in. H_2O), with reference to the welding box atmospheric pressure. The welding glove box was equipped with an evacuation pump and a bottled gas manifold system for backfilling the box after evacuation and subsequently to provide a continuous purge. Gas pressure inside the box, with respect to the ambient atmospheric pressure, was maintained negative to provide for containment of radioactive materials; internal box pressure was maintained by connection through filters to the cell ventilation system. An oxygen analyzer was installed to monitor the composition of the internal atmosphere. All entry and exit service lines were equipped with absolute filters and vacuum valving to isolate the glove box during evacuation.

Pellet transfer containers, Fig. 11, were fabricated from type 316 stainless steel. The internal through-bore was dimensioned to accept seven finished fuel pellets. Each end of the container was internally and externally threaded to provide a double seal using an internal screw

ORNL Photo 3769-81

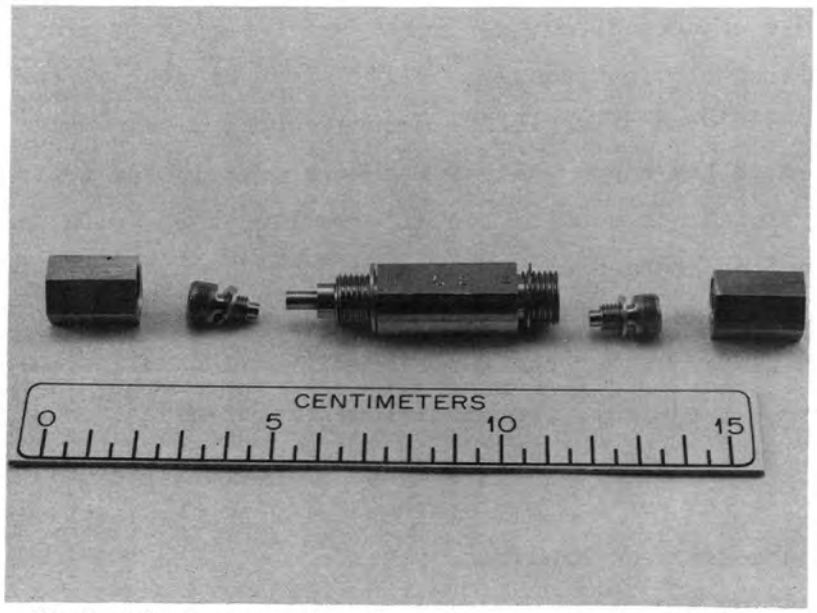

Fig. 11. Fuel Pellet Transfer Container Used for Moving Fuel Pellets to Welding Glove Box

ORNL Photo 5518-80

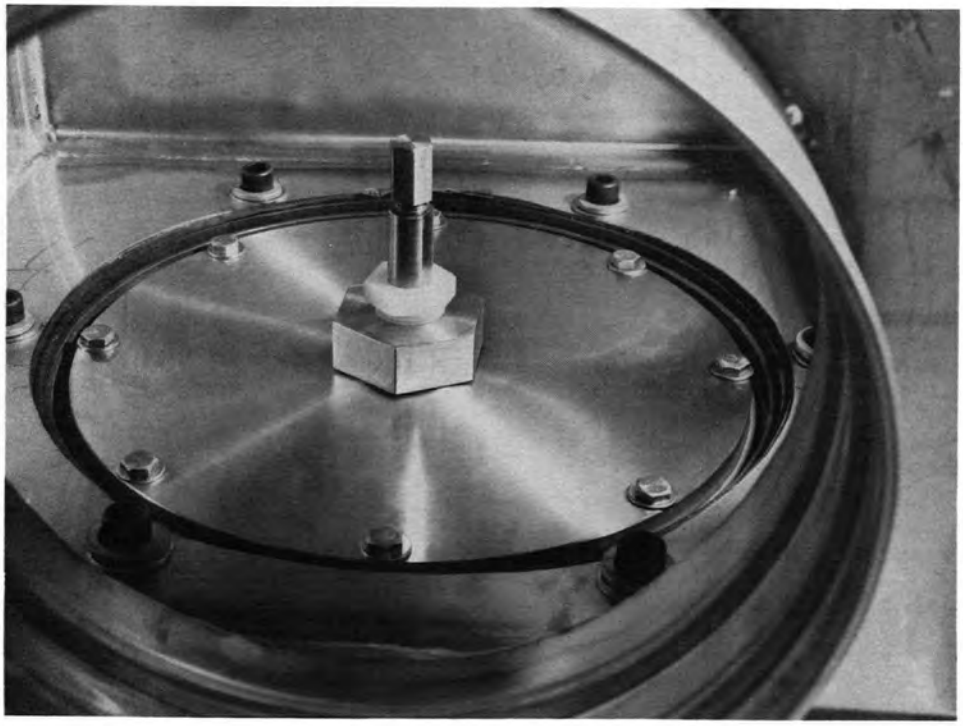

Fig. 12. Fuel Pellet Transfer Container Positioned for Loadout of Fuel Pellets From Fuel Pellet Fabrication Glove Box

ORNL Photo 3547-81

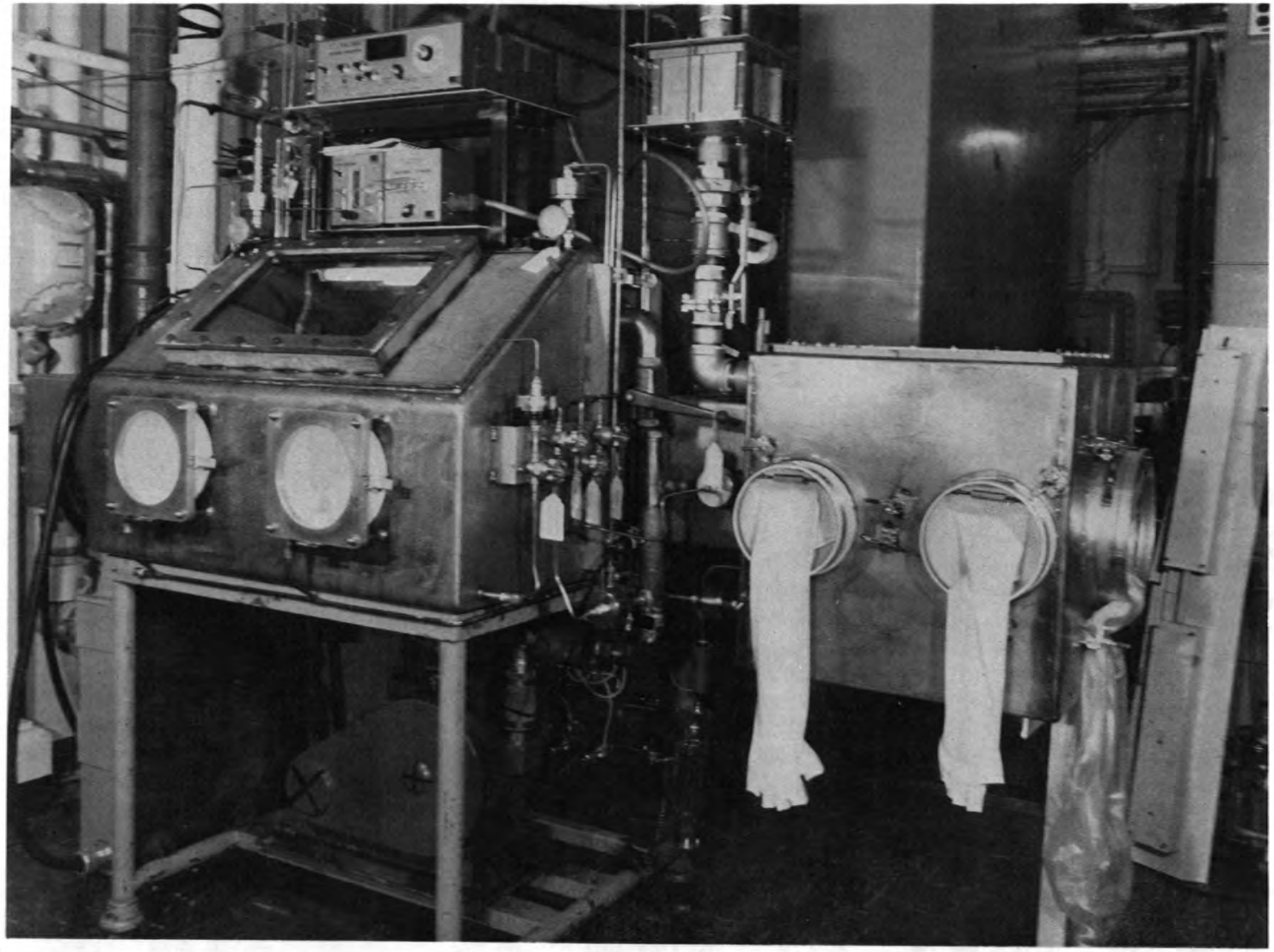

Fig. 13. Exterior View of Welding Glove Box Showing Entry Box and Oxygen Analyzer System

ORNL Photo 3546-81

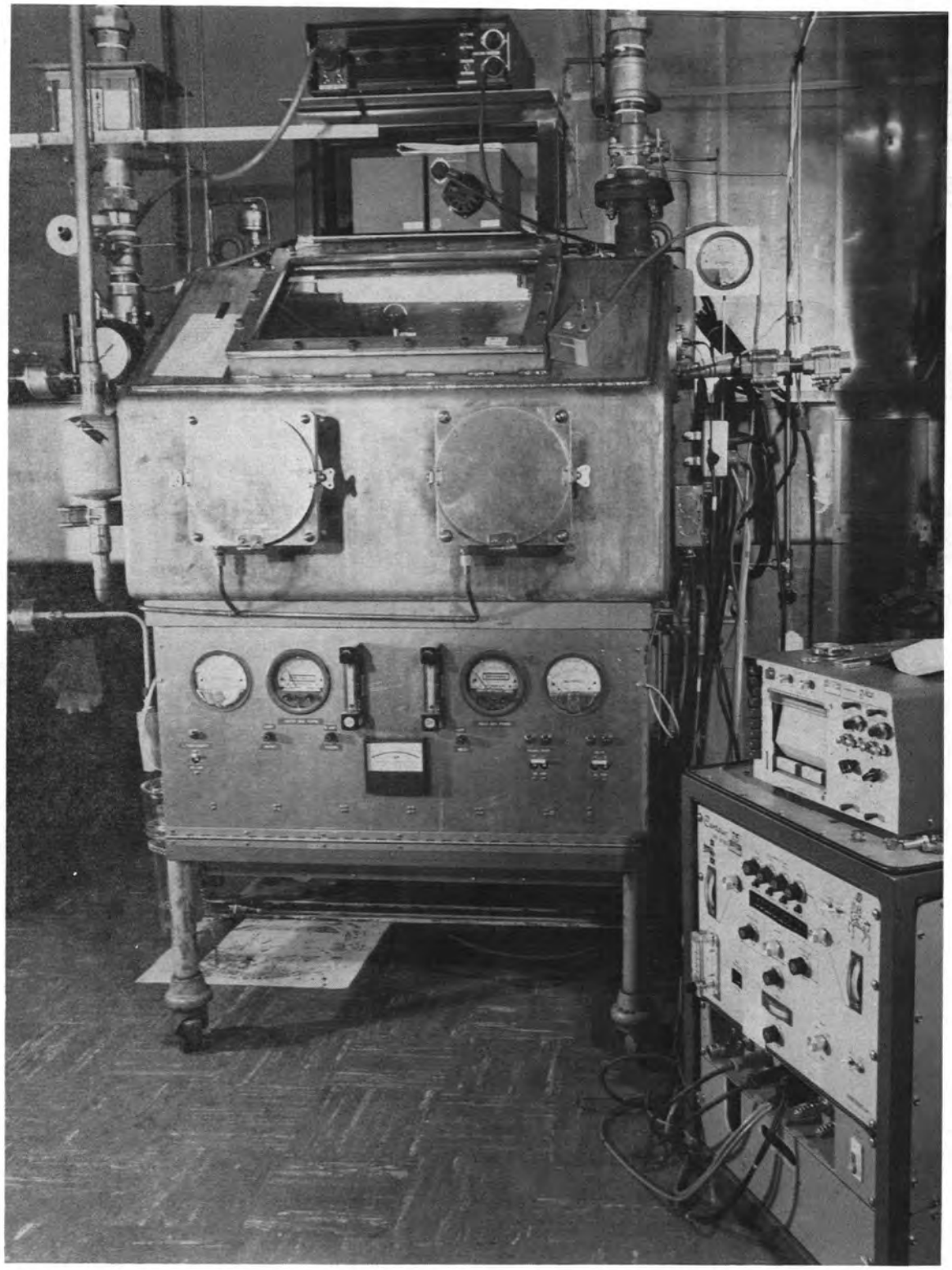

Fig. 14. Exterior View of Welding Glove Box Showing Welding Power Supply and Glove Box Atmosphere Controls

ORNL Photo 3545-81

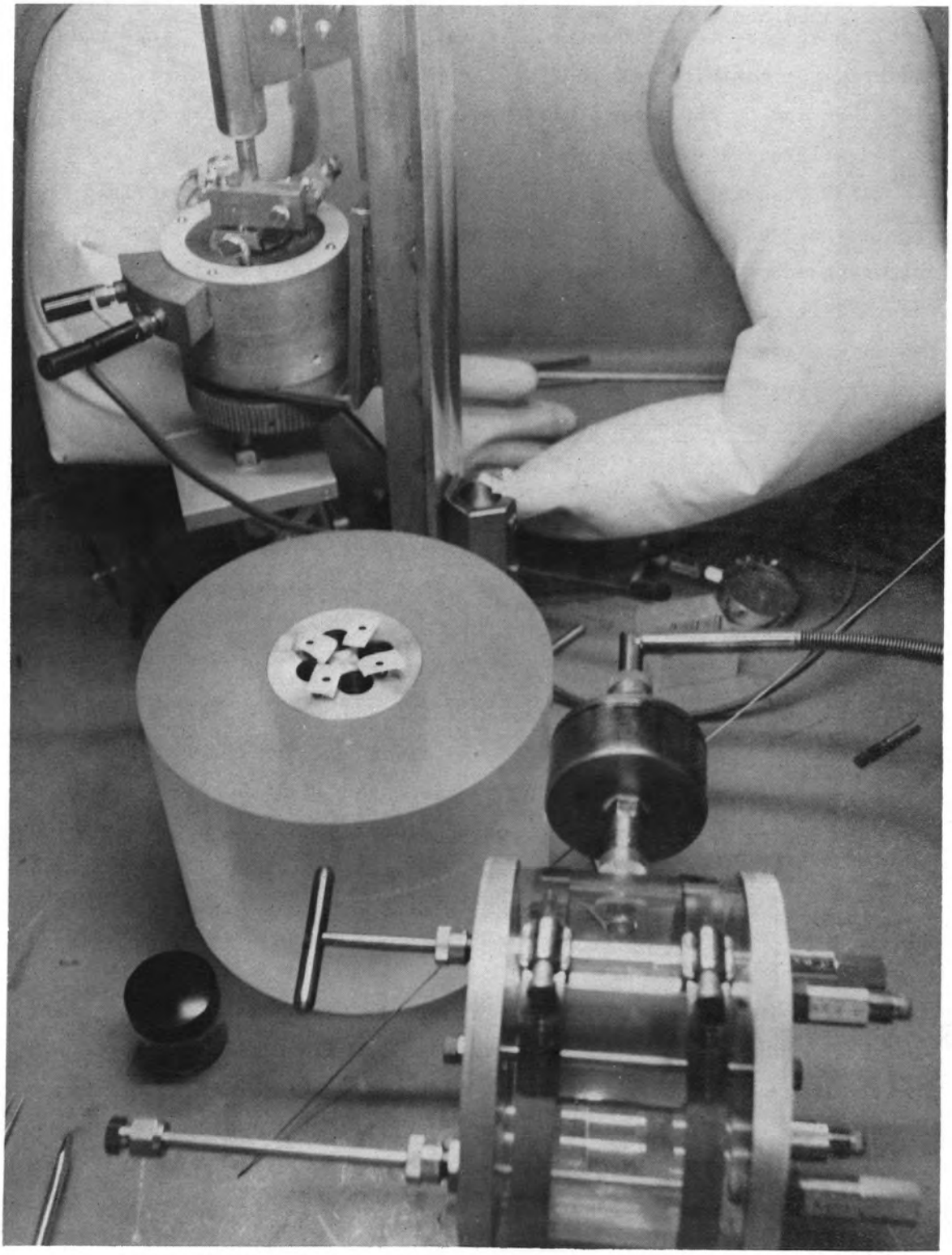

Fig. 15. Internal View of Welding Glove Box Showing Welding Setup, Storage Container for Fuel Pins, and Fuel Pin Loading Apparatus

plug and an external hexagonal cap closure; both seals were formed by a soft copper gasket. The external thread was also used for attachment of the transfer container to the loading port in the pellet fabrication glove box and to the flange plate of the secondary containment apparatus in the welding glove box.

Each transfer container was labeled to identify the contained fuel type as well as the loading and unloading ends. A precisely machined unloading adaptor was provided which mated with the transfer container internal thread and the counterbored open end of the fuel pins so as to facilitate transfer of fuel pellets prior to welding and to maintain a contamination-free weld zone on the pin.

B. Actinide Powder Preparation

The homogeneity and microstructural specifications for the three actinide-containing powders required that each material be coprecipitated to achieve appropriate particle size. The procedure selected to meet these requirements is generally known as the "urea solidification process."[3,4] This process has been used by IRML personnel and others for the production of oxide powders which are required to have very limited and controlled particle size and be easily sintered to high density. In addition, the process allows the production of mixed oxide powders which approach absolute chemical homogeneity. The ability of the process to produce powders with these characteristics has led to its use in the production of neutron dosimeter materials, cermets and several solid radioisotope waste storage forms. Oxide powder particle size is predictable by variation of the ratio of the cations added and the urea concentration in the precipitation medium.

To attain identical ceramic characteristics with all three types of actinide powders, all types were produced under identical conditions. The procedure described below is therefore representative of that used in producing all types of materials; appropriate chemical processing was conducted in a manipulator cell.

In each case the appropriate weight of starting material, as oxide, (for the Type III material, a mixture of ^{241}Am, ^{244}Cm and stable rare earths) was dissolved in a solution of 70 cc of 16 N HNO_3 and a small

amount of 30% H_2O_2 in a quartz, one-liter beaker. After dissolution occurred, 350 g of granular urea was slowly added to the acid solution while heating to a temperature sufficient to melt the urea. After all the urea was molten, the temperature of the mixture was held at approximately 425 K for one hour. During this digestion period, water and waters of hydration of the soluble nitrates present were removed according to the following reaction:

$$NH_2 \cdot CO \cdot NH_2 + H_2O \xrightarrow{\Delta} 2NH_3\uparrow + CO_2\uparrow.$$

The melt temperature was then raised to approximately 475 K for about 45 minutes which caused subsequent solidification. It is believed that this transition occurs through the sequence of compound formations noted below:

$$\text{Urea} \xrightarrow{\Delta} \text{Biuret} \xrightarrow{\Delta} \text{Cyanuric acid} \xrightarrow{\Delta} \text{Melamine}\ .$$

Heat was supplied by a laboratory hot plate to cause these reactions. During the digestion of the molten mixture containing the ^{244}Cm for the Type I material, a beautiful orange noctilucent glow was observed, Fig. 16, which was caused by the intense radiation.

Solid material resulting from the urea solidification process was subsequently calcined in air at 1075 K for 8 hrs. The resultant oxide powder was transferred to stainless steel powder storage cans; four cans were loaded for each material type, each containing 3.5 to 4 g of powder. Material division minimized hand exposure during later operations in the pellet fabrication glove box.

After weighing and canning, the oxide powder was transferred to the water-shielded storage facility within the pellet fabrication glove box. A hydrogen reduction was then performed in the tube furnace to convert each material to its respective sesquioxide. This was accomplished using a nickel boat at 1275 K in a flowing dry mixture of 4% H_2 - 96% He for two hours. Completion of powder reduction was substantiated gravimetrically. In all cases the quantity of reduced powder was within >99% of the theoretical weight. After reduction, the powders were again stored in the water-shielded storage well to await pellet fabrication. During all

ORNL Photo 3352-81

Fig. 16. ^{244}Cm Radiation-Induced Fluorescence of Molten Urea During Precipitation Process

operations, an inert atmosphere was maintained and monitored within the pellet fabrication glove box.

Pellets of Type II material (Am_2O_3) were prepared initially, followed by Type III (mixture) and then Type I; this sequence was consistent with the increasing radiation exposure potential of the three materials and would minimize the possibility of cross contamination. The pelletization, characterization and load-out of each material type was completed and a glove change and cleaning operation undertaken before the next type of powder was handled in the facility.

C. Actinide Fuel Pellet Preparation

Pelletization of all actinide materials was conducted by hot-pressing seven pellets simultaneously. Seven pellets met the requirements for all specified analytical aliquots as well as archive samples. Secondly, this number of pellets at the specified density would yield a total stack length commensurate with that specified for a single fuel pin loading, while also creating an individual pellet length-to-diameter ratio very near 1:1. This ratio was desirable to assure uniform densification in the hot-pressing process. In the case of the Type I material ($^{244}Cm_2O_3$) seven pellets also represented the maximum allowable amount of material which could be handled without undue personnel exposure.

In practice, the first seven pellets pressed of each material type were used for the required analytical and archive samples since this would ascertain that specifications were being met before pellets were pressed for actual fuel pin insertion. Since no additional material was available for the experimental establishment of densification parameters, this procedure was mandatory. Initial parameters were selected based on previous experience with similar materials. Fortunately, this experience proved to be adequate in meeting all physical parametric requirements.

Ultimate pellet dimensions were predicted by the calculation of the appropriate weight of powder which would be required to produce a pellet with the specified dimensions at an assumed density of 90% of the theoretical value for each particular actinide oxide. The theoretical densities of $^{244}Cm_2O_3$ and $^{241}Am_2O_3$ are well established. A "theoretical" density for the Type III material was calculated by proportional density-averaging of the individual component oxides weighted by their respective concentration in the compound.

Die loading was accomplished by using a specially fabricated stainless steel funnel equipped with a stainless steel center bore plug, Fig. 10. The appropriate amount of oxide powder was added to the funnel, while resting on the balance pan with the plug inserted. The funnel tip was then inserted in one of the seven die bores into which a short "Vitrecarb" punch had previously been inserted. The bore plug of the funnel was then removed allowing the powder to drop into the die

bore. This method of transfer virtually eliminated loss of material during the loading operation. After the loading procedure was repeated seven times, the longer top punches were installed. The center force distributor of the die was inserted and the assembly placed in the hot press. Following evacuation, the die assembly was heated to 1575 K while maintaining a pressure from the pneumatic driver sufficient to produce 13.8 MPa (2000 psi) on each pellet. Once the temperature of 1575 K was reached, the pressure was increased to 27.6 MPa (4000 psi) and maintained for one hour. At the end of this period, pressure was removed and the assembly cooled. After removal of the die assembly from the hot press, pellets were individually removed by pushing on the long punches by hand. Die assemblies were reusable by reversing punch positions to place the powder load in an unused portion of the die bores. Following densification, the pellets were stored in identifiable containers which were placed in the water-shielded storage well and heat sink to await characterization and load-out operations.

D. Actinide Fuel Pellet Characterization

Pellet characterization was conducted within the pellet fabrication glove box after the densification of each set of seven pellets was completed. Characterization consisted of a visual examination of each pellet and dimensional metrology. Pellets were required to pass through a ring gauge with a certified bore size. The diameter, height and final weight of each pellet were measured and recorded, and the pellet density was calculated. During this operation each pellet of the set was assigned a number which ultimately was indicative of its position in the pellet stack during storage, transfer and fuel pin loading.

From the first pressing of each material type, two pellets were taken out of the glove box for subsequent carbon and impurity analyses as well as for determination of oxygen-to-metal ratios by the ORNL Analytical Chemistry Division. In addition, three pellets were shipped to HEDL for further analysis and ceramographic examination. One pellet was used for the determination of high temperature dimensional stability. This test consisted of a two-hour heating period at a temperature of 1825 K with subsequent examination to determine pellet volume or weight change. The

hot press within the pellet fabrication glove box was used for this test with the pellet resting on an ATJ graphite support. This test could not be made accurately on the pellet of the Type I material because of apparent surface reaction between the $^{244}Cm_2O_3$ and the graphite support resulting in strong adherence of the pellet to the support. Based only on diameter measurements of the latter pellet, the volume reduction was approximately 3%. The one remaining pellet from the initial pressing of each material type served as an archive sample and ultimately was welded in a fuel pin for storage at HEDL.

As the three subsequent pressings of each material type were made, dimensional measurements were performed and recorded; each set of seven pellets were loaded into an identifiable transfer container which was attached to the load-out port from within the appendage glove box. Pellets in numerical order were placed on a loading trough which screwed into the internal thread of the transfer container, Fig. 17. The pellets

ORNL Photo 0998-81

Fig. 17. Loading Trough Used for Moving Completed Actinide Fuel Pellets Into the Transfer Container (Fig. 10)

were then pushed into the bore of the transfer container with a stainless steel rod, the loading trough removed and the internal gasketed plug inserted. Prior to removal of the loaded transfer container from the port, a threaded plug was inserted from within the pellet fabrication glove box into the load-out port to prevent the transfer of contamination into the appendage box. After removal of each sealed transfer container from the appendage glove box, the container exterior was decontaminated by abrasive blasting in an inflated hood bag equipped with gloves.

Analytical results for all fuel pellet materials are given in Appendix 1. The improvement in the sinterability of oxide powders produced by the urea process is made obvious by the comparison of the densities listed at pressing temperatures which were 150-200 K lower than those required to reach comparable densities with similar unprecipitated actinide oxide powders. Seven hot-pressed $^{244}Cm_2O_3$ pellets are shown in Fig. 18.

ORNL Photo 35-4381

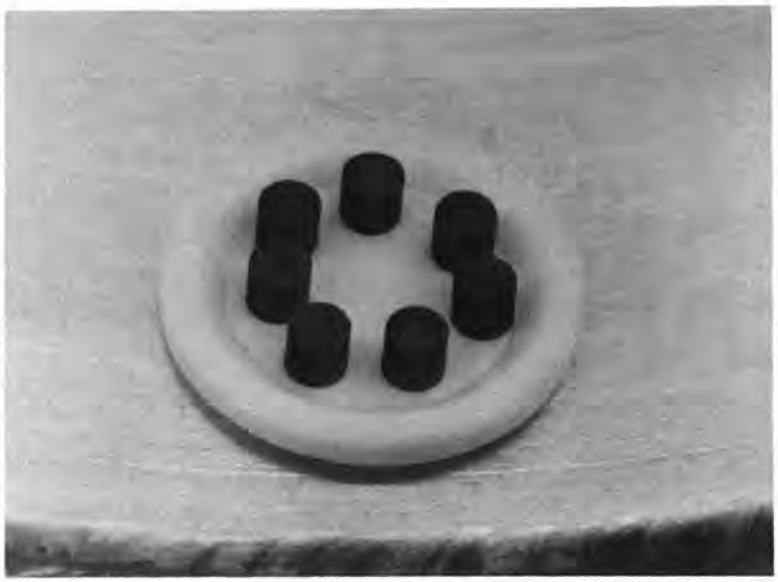

Fig. 18. Seven $^{244}Cm_2O_3$ Fuel Pellets Fabricated in a Single Pressing Displayed on a Thoria Planchet

E. Associated Hazards

The quantities of ^{241}Am and ^{244}Cm processed during fuel pellet preparation required the use of special handling equipment and techniques (described above) to minimize personnel exposure. Both ^{241}Am and ^{244}Cm decay by the emission of alpha particles. In addition ^{244}Cm decays by spontaneous fission (1.35×10^{-4} %) with the emission of neutrons. Although ^{244}Cm represented the most serious problem relating to personnel exposure, the 60-keV gamma ray emitted by the ^{241}Am daughter product, ^{237}Np, decaying to the ground state could not be ignored. However, personnel exposure to the gamma radiation was virtually eliminated by using small amounts of lead shielding.

The problems associated with the handling of ^{244}Cm arose mainly from the heat and neutron radiation resulting from ^{244}Cm decay.

a. Heat Problems

Curium-244 has a half-life of 18.1 years and generates approximately 2.8 watts per gram of ^{244}Cm as a result of its decay. Each $^{244}Cm_2O_3$ pellet contained approximately 0.3 grams of ^{244}Cm and thus generated approximately 0.8 watts of heat. Since each curium fuel pin contained seven pellets, approximately 5.6 watts of heat were generated by the ^{244}Cm inside the fuel pin. A measurement on one of the curium-containing pins in air indicated that the outer surface temperature was approximately 525 K.

Precautions were taken when handling ^{244}Cm in the glove box environment to prevent the possibility of burning a hole in a glove (with possible release of major contamination) and/or burning the hands of the operating personnel. However, the heat generated by ^{244}Cm decay created another problem that was not anticipated. The ^{244}Cm decay heat caused the threads on the first transfer capsule to seize making it impossible to unload the pellets according to the prescribed procedure. The pellets were necessarily removed in reverse order from the opposite end of the transfer capsule (the end through which the pellets were loaded into the capsule). A

slight change in handling procedure corrected the problem and the remaining transfers were made as planned. In any future work involving radioactive heat sources loaded in containers with screw caps, consideration should be given to the use of chill blocks for removing decay-generated heat to prevent thread seizure.

b. Radiation Exposure

Radiation exposure was a major consideration in designing equipment and developing procedures for preparing the fuel pellets. It was mandatory that close surveillance of all operating personnel be maintained by Health Physics personnel to keep personnel exposure records current. Operating personnel were required to wear special film badges to measure neutron exposure levels. Pocket dosimeters, film badges and finger rings were used to monitor exposure caused by beta- or gamma-radiation. In addition to daily pocket dosimeter readings, film badges were processed at the midpoint of the project to determine actual exposure as compared to estimated individual personnel exposure.

Monitoring personnel for exposure to neutron radiation was a problem since reliable pocket dosimeters for neutron detection are not available. Neutron exposure was estimated by using predetermined neutron-to-gamma radiation ratios. Since the neutron-to-gamma ratios were constantly changing, a conservative ratio of 5 mRem neutrons to 1 mRem gamma was used for estimating exposure. Personnel exposure levels were updated on a daily basis by this method. Radiation readings obtained from fuel pins loaded with seven fuel pellets of Type I (Cm_2O_3), Type II (Am_2O_3) and Type III (M_2O_3) are given in Table 8.

Table 8. Radiation Measurements for Cm_2O_3, Am_2O_3, and M_2O_3 Pellets Loaded in Fuel Pins

Sample Identification	Distance (cm)	Gamma Radiation (mRem/hr)	Neutron Radiation (mRem/hr)	Total Radiation (mRem/hr)	Comments
Type I Fuel Pin (Cm_2O_3) 7 Pellets	10.2	400	2200	2600	Readings made perpendicular to fuel pin
Type I Fuel Pin (Cm_2O_3) 7 Pellets	10.2	100	1000	1100	Readings made parallel to fuel pin
Type II Fuel Pin (Am_2O_3) 7 Pellets	3.8	1100	0	1100	Readings made perpendicular to fuel pin
Type III Fuel Pin (M_2O_3) 7 Pellets	3.8	580	500	1080	Readings made perpendicular to fuel pin
Type III Fuel Pin (M_2O_3) 7 Pellets	3.8	100	150	250	Readings made parallel to fuel pin

V. PHYSICS SPECIMEN FABRICATION AND CHARACTERIZATION

A. Selection of Actinide Materials

A total of twenty-one actinide materials were selected for use as physics specimen materials for the US/UK Actinide Experiment. Criteria used in selecting the materials included availability, isotopic enrichment, and chemical purity. The physics specimen materials chosen are shown in Table 9.

Table 9. Physics Specimen Materials Selected for US/UK Higher Actinide Experiment

Isotope	Batch Number	Isotope Enrichment (Wt. %)
Cm-248	1116	92.148[e]
Cm-246	C59SHIP	66.53 [f]
Np-237	24HP	99.99
Cm-244	C57CM46	92.35 [g]
Cm-243	1011	57.67 [h]
U-238	Q1	99.955
Am-243	HIP1018	99.987
Am-241	79AMB4	99.995
Pu-242	290A	99.745
Pu-244	297-C	87.47
Pu-240	HIP1068	99.93
Pu-239	453-BO	99.10
Pu-241	307-A	97.80
Th-232	4151	100.00
U-236	201DMR	88.96
U-234	M9	99.764
U-235	264C	99.89
Pa-231	Pa-F-1	100.00
Pu-238	06HP014	99.39 [i]
Th-230	256A	89.39
U-233	240A	99.886

[e] February 11, 1981.
[f] August 5, 1980.
[g] March 19, 1979.
[h] July 18, 1980.
[i] June 20, 1972.

B. Selection of Dosimeter Materials

Accurate characterization of the irradiated physics specimens requires that the neutron energy spectra, flux and fluence to which the physics specimens are exposed be accurately determined. The six neutron dosimeter materials used for this purpose are shown in Table 10. The approximate neutron energy ranges covered by these dosimeter materials are shown in Table 11. The ORNL dosimeter package described here is distinct from the HEDL dosimeter package (Fig. 2).

Table 10. Dosimeter Materials Selected for Use With Physics Specimens in US/UK Higher Actinide Experiment

Isotope	Batch Number	Isotope Enrichment (Wt. %)
U-235	264C	99.89
U-238	ESZ	99.999
Pu-239	453-BO	99.10
Np-237	24HP	99.99
Cu	N	Normal
Co	Co-1	Normal

Table 11. Approximate Neutron Energy Ranges Detected by Specific Dosimetry Materials

Isotope	Approximate Neutron Energy Range (MeV)	Reaction
^{59}Co	0.01- 1.0	$^{59}Co\ (n,\gamma)\ ^{60}Co$
^{235}U	0.03- 1.3	^{235}U (n,f) F.P.
^{239}Pu	0.05- 1.3	^{239}Pu (n,f) F.P.
^{237}Np	0.5 - 4.0	^{237}Np (n,f) F.P.
^{238}U	1.1 - 6.0	^{238}U (n,f) F.P.
^{63}Cu	6.0 -10.0	$^{63}Cu\ (n,\alpha)\ ^{60}Co$

C. Encapsulation of Physics Specimen and Neutron Dosimeter Materials

a) Choice of Capsule Material

All physics specimen and dosimeter materials were encapsulated in vanadium. Factors that influenced the use of vanadium as the encapsulation material included the following:

(1) Low neutron cross section,

(2) Ease of fabrication,

(3) Availability of very high purity metal, and

(4) High melting point.

In addition, capsules were required to be capable of withstanding large internal pressures created by gases resulting from neutron-induced fission and/or alpha decay. Calculations of anticipated gas pressure and maximum gas pressure that could be tolerated by the vanadium capsules are given in Appendix 2.

b) Loading and Welding of Vanadium Capsules

All physics specimens and dosimeters were encapsulated in vanadium containers shown in Figures 3 and 4. All physics specimen dosimeters listed in Table 10 were double-encapsulated in vanadium containers as detailed in Figure 4. Physics specimen materials (Table 9) were loaded into vanadium capsules shown in Figure 3 or into the "outer" vanadium capsules shown in Figure 4. Physics specimen materials loaded into the larger "outer" vanadium capsules provided the option of a longer reactor irradiation time for this series of actinide materials, since a larger quantity of fission gas could be accommodated in the larger capsule volume.

The quantities of isotopic material required for each dosimeter or physics specimen capsule are shown in Table 12. Since most of the materials loaded into the small vanadium capsules were in oxide form, special techniques and equipment were used to prevent gross external contamination of the capsules and to permit accurate determination of the weight of each encapsulated material. The amount of material in each vanadium capsule was determined by physical weighing. The weight of material per capsule was reported to the nearest microgram and the 2σ uncertainty of each weight was recorded. The substitution weighing scheme that was used and the associated computations are described in Appendix 3. Capsule contamination was reduced by using funnel loaders, shown in Figure 19, for insertion of the actinide oxide powders. The empty vanadium capsule was weighed, according to the weighing scheme described in Appendix 3 and then placed in the funnel loader. The actinide oxide powder to be loaded was weighed, poured into the funnel loader, and packed into the previously weighed vanadium capsule. The capsule was then re-weighed by the scheme described in Appendix 3 and the weight difference between the empty and loaded vanadium capsule was determined. The funnel loader was not needed for loading the dosimeter

Table 12. Quantities of Isotope Required in Physics Specimen and Dosimeter Capsules

Isotope	Amount of Compound Per Capsule (mg)
Physics Specimen Materials	
Th-230	3.5 - 4.0
Th-232	20.0 - 25.0
Pa-231	2.5 - 4.0
U-233	9.0 - 10.0
U-234	4.5 - 5.0
U-235	9.0 - 10.0
U-236	12.0 - 12.5
U-238	12.0 - 12.5
Np-237	13.5 - 14.0
Pu-238	3.5 - 4.0
Pu-239	9.0 - 10.0
Pu-240	13.5 - 14.0
Pu-241	5.5 - 6.0
Pu-242	2.0 - 2.5
Pu-244	2.5 - 3.0
Am-241	11.0 - 14.0
Am-243	11.0 - 14.0
Cm-243	1.0 - 1.2
Cm-244	11.0 - 14.0
Cm-246	11.0 - 14.0
Cm-248	2.0 - 2.5
Dosimeter Materials	
U-235	1.0 - 1.5
U-238	1.0 - 1.5
Np-237	1.0 - 1.5
Pu-239	1.0 - 1.5
Fe-54	1.0 - 1.5
Cu-63	1.0 - 1.5
Co-59 (0.1% Co-MgO)	1.0 - 1.5

ORNL Photo 8346-81

Fig. 19. Actinide Oxide Powder Loaders Used for Loading Physics Specimen Materials Into Vanadium Capsules

materials and a small number of the physics specimens since the materials were in the form of a ceramic oxide wire or metal foil. Non-powder samples were weighed directly by the scheme described in Appendix 3 and loaded into the vanadium capsules. Loaded vanadium capsules were placed in a copper chill block, Figure 20, and hermetic closure was made by tungsten-inert gas (TIG) fusion welding. All vanadium encapsulated materials were loaded and welded in a helium atmosphere.

c) Characterization of Physics Specimen and Dosimeter Materials

All encapsulated isotope materials were or will be characterized by spark source mass spectrographic, isotopic and concentration analyses. These analyses are essential in interpreting the analytical data to be obtained upon post-irradiation examination. The quantities of material encapsulated in each physics specimen and in each dosimeter together with the available isotopic and concentration analyses are shown in Tables 13, 14, and 15 for capsules loaded into fuel pin 1 (FP1), fuel pin 2 (FP2) and fuel pin 4 (FP4), respectively. It should be noted that the analytical data in Tables 13-15 will be updated as analyses on archive samples are completed.

Table 13. Analytical Data for Physics Specimen and Dosimeter Materials Contained in FP1.

Position	Capsule Material	Batch Number	Isotope Enrichment (wt. %)	Concentration Analysis (mg/g)	Compound Weight (mg)	Isotope Weight (mg)
1	Dos (U-235)	264C	99.89	879.7	1.245	1.094
	Dos (U-238)	ESZ	99.999	877.5	1.154	1.013
2	Dos (Pu-239)	453-BO	99.10	864.2	1.451	1.243
	Dos (Np-237)	24HP	99.99	874.0	1.300	1.136
3	Dos (Cu)	N	Normal	999.93	1.231	1.231
	Dos (Co)	CO-1	Normal	1.23	1.300	0.00160
4	Cm-248	1117	92.15[j]	910.0[j]	2.051	1.720
5	Cm-246	C59SHIP	66.53[k]	871.0[l]	11.488	6.657[k,l]
6	Cm-246	C59SHIP	66.53[k]	871.0[l]	11.284	6.539[k,l]
7	Np-237	24HP	99.99	874.0	13.674	11.950
8	Cm-244	C57CM46	92.35[m]	794.9[m]	11.108	8.154[m]
9	Cm-244	C57CM46	92.35[m]	794.9[m]	11.303	8.297[m]
10	Cm-243	1011	57.67[n]	576.0	1.010	0.336
11	U-238	Q1	99.96	814.6	12.108	9.859
12	Am-243	HIP1018	99.99	881.7	11.121	9.804
13	Am-243	HIP1018	99.99	881.7	11.275	9.940
14	Am-241	79AMB4	99.99	862.0	12.046	10.383
15	Am-241	79AMB4	99.99	862.0	11.081	9.551
16	Pu-242	290A			2.470	
17	Dos (U-235)	264C	99.89	879.7	1.193	1.048
	Dos (U-238)	ESZ	99.999	877.5	1.286	1.129
18	Dos (Pu-239)	453-BO	99.10	864.2	1.387	1.188
	Dos (Np-237)	24HP	99.99	874.0	1.286	1.124
19	Dos (Cu)	N	Normal	999.93	1.056	1.056
	Dos (Co)	CO-1	Normal	1.23	1.378	0.00169
20	Pu-244	297-C	87.47[o]		2.716	
21	Pu-240	HIP1068	99.93		13.906	
22	Pu-240	HIP1068	99.93		13.590	
23	Pu-239	453-BO	99.10	864.2	9.330	7.990
24	Pu-241	307-A	96.83[p]		5.705	
25	Th-232	4151	100.00	877.5	20.252	17.771

Table 13 (Cont'd)

Position	Capsule Material	Batch Number	Isotope Enrichment (wt. %)	Concentration Analysis (mg/g)	Compound Weight (mg)	Isotope Weight (mg)
26	U-236	201DMR	88.96	730.1	12.173	7.906
27	U-234	M9	99.764	720.0	4.792	3.442
28	U-235	264C	99.89	879.7	9.708	8.531
29	Pa-231	Pa-F-1	100.00	798.0	3.615	2.885
30	Pu-238	06HP014	99.38(q)	841.3(q)	3.504	2.930(q)
31	Th-230	256A	89.39	822.5	3.973	2.921
32	U-233	240A	99.886	797.8	9.939	7.920
33	Dos (U-235)	264C	99.89	879.7	1.218	1.070
	Dos (U-238)	ESZ	99.999	877.5	1.149	1.008
34	Dos (Pu-239)	453-B0	99.10	864.2	1.407	1.205
	Dos (Np-237)	24HP	99.99	874.0	1.290	1.127
35	Dos (Cu)	N	Normal	999.93	1.358	1.358
	Dos (Co)	CO-1	Normal	1.23	1.337	0.00164

(j) February 11, 1981
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(l) October 1, 1980
(m) June 16, 1981
(n) July 18, 1980
(o) September 14, 1973
(p) December 30, 1981
(q) June 20, 1972

Table 14. Analytical Data for Physics Specimen and Dosimeter Materials Contained in FP2.

Position	Capsule Material	Batch Number	Isotope Enrichment (wt. %)	Concentration Analysis (mg/g)	Compound Weight (mg)	Isotope Weight (mg)
1	Dos (U-235)	264C	99.89	879.7	1.296	1.139
	Dos (U-238)	ESZ	99.999	877.5	1.314	1.153
2	Dos (Pu-239)	453-BO	99.10	864.2	1.406	1.204
	Dos (Np-237)	24HP	99.99	874.0	1.229	1.074
3	Dos (Cu)	N	Normal	999.93	1.405	1.405
	Dos (Co)	CO-1	Normal	1.23	1.309	0.00161
4	Cm-248	1117	92.15[j]	910.0[j]	2.120	1.778
5	Cm-246	C59SHIP	66.53[k]	871.0[l]	11.748	6.808[k,l]
6	Cm-246	C59SHIP	66.53[k]	871.0[l]	11.317	6.558[k,l]
7	Np-237	24HP	99.99	874.0	13.903	12.150
8	Cm-244	C57CM46	92.35[m]	794.9[m]	11.249	8.258[m]
9	Cm-244	C57CM46	92.35[m]	794.9[m]	11.442	8.399[m]
10	Cm-243	1011	57.67[n]	576.0	1.135	0.377
11	U-238	Q1	99.96	814.6	12.692	10.335
12	Am-243	HIP1018	99.99	881.7	11.491	10.131
13	Am-243	HIP1018	99.99	881.7	11.347	10.004
14	Am-241	79AMB4	99.99	862.0	11.252	9.698
15	Am-241	79AMB4	99.99	862.0	10.892	9.388
16	Pu-242	290A			2.470	
17	Dos (U-235)	264C	99.89	879.7	1.338	1.176
	Dos (U-238)	ESZ	99.999	877.5	1.234	1.083
18	Dos (Pu-239)	453-BO	99.10	864.2	1.201	1.029
	Dos (Np-237)	24HP	99.99	874.0	1.027	0.898
19	Dos (Cu)	N	Normal	999.93	1.388	1.388
	Dos (Co)	CO-1	Normal	1.23	1.249	0.00154
20	Pu-244	297-C	87.47(o)		2.784	
21	Pu-240	HIP1068	99.93		13.652	
22	Pu-240	HIP1068	99.93		13.738	
23	Pu-239	453-BO	99.10	864.2	9.251	7.923
24	Pu-241	307-A	96.83[p]		5.621	
25	Th-232	4151	100.0	877.5	20.382	17.885

Table 14 (Cont'd)

Position	Capsule Material	Batch Number	Isotope Enrichment (wt. %)	Concentration Analysis (mg/g)	Compound Weight (mg)	Isotope Weight (mg)
26	U-236	201DMR	88.96	730.1	12.195	7.921
27	U-234	M9	99.764	720.0	4.558	3.274
28	U-235	264C	99.89	879.7	9.232	8.113
29	Pa-231	Pa-F-1	100.0	798.0	3.249	2.593
30	Pu-238	06HP014	99.39(p)	841.3(q)	3.939	3.293(q)
31	Th-230	256A	89.39	822.5	3.986	2.931
32	U-233	240A	99.886	797.8	9.759	7.777
33	Dos (U-235)	264C	99.89	879.7	1.316	1.156
	Dos (U-238)	ESZ	99.999	877.5	1.103	0.968
34	Dos (Pu-239)	453-BO	99.10	864.2	1.179	1.010
	Dos (Np-237)	24HP	99.99	874.0	1.100	0.961
35	Dos (Cu)	N	Normal	999.93	1.247	1.247
	Dos (Co)	CO-1	Normal	1.23	1.169	0.00144

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(n) July 18, 1980
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(p) December 30, 1981
(q) June 20, 1972

Table 15. Analytical Data for Physics Specimen and Dosimeter Materials Contained in FP4.

Position	Capsule Material	Batch Number	Isotope Enrichment (wt. %)	Concentration Analysis (mg/g)	Compound Weight (mg)	Isotope Weight (mg)
1	Dos (U-235)	264C	99.89	879.7	1.339	1.177
	Dos (U-238)	ESZ	99.999	877.5	1.345	1.180
2	Dos (Pu-239)	453-BO	99.10	864.2	1.396	1.196
	Dos (Np-237)	24HP	99.99	874.0	1.194	1.044
3	Dos (Cu)	N	Normal	999.93	1.353	1.353
	Dos (Co)	CO-1	Normal	1.23	1.221	0.00150
4	Cm-248	1117	92.15(j)	910.0(j)	2.124	1.781
5	Cm-246	C59SHIP	66.53(k)	871.0(l)	11.547	6.691(k,l)
6	Cm-246	C59SHIP	66.53(k)	871.0(l)	11.730	6.797(k,l)
7	Np-237	24HP	99.99	874.0	13.979	12.216
8	Cm-244	C57CM46	92.35(m)	794.9(m)	11.337	8.322(m)
9	Cm-244	C57CM46	92.35(m)	794.9(m)	11.239	8.250(m)
10	Cm-243	1011	57.67(n)	576.0	1.180	0.392
11	U-238	Q1	99.96	814.6	12.048	9.810
12	Am-243	HIP1018	99.99	881.7	11.152	9.832
13	Am-243	HIP1018	99.99	881.7	11.118	9.802
14	Am-241	79AMB4	99.99	862.0	10.931	9.422
15	Am-241	79AMB4	99.99	862.0	11.051	9.525
16	Pu-242	290A			2.421	
17	Dos (U-235)	264C	99.89	879.7	1.259	1.106
	Dos (U-238)	ESZ	99.999	877.5	1.119	0.982
18	Dos (Pu-239)	453-BO	99.10	864.2	1.331	1.140
	Dos (Np-237)	24HP	99.99	874.0	1.286	1.124
19	Dos (Cu)	N	Normal	999.93	1.406	1.406
	Dos (Co)	CO-1	Normal	1.23	1.279	0.00157
20	Pu-244	297-C	87.47(o)		2.741	
21	Pu-240	HIP1068	99.93		13.980	
22	Pu-240	HIP1068	99.93		13.951	
23	Pu-239	453-BO	99.10	864.2	9.890	8.470
24	Pu-241	307-A	96.83(p)		5.920	
25	Th-232	4151	100.0	877.5	21.516	18.880

Table 15 (Cont'd)

Position	Capsule Material	Batch Number	Isotope Enrichment (wt. %)	Concentration Analysis (mg/g)	Compound Weight (mg)	Isotope Weight (mg)
26	U-236	201DMR	88.96	730.1	12.258	7.962
27	U-234	M9	99.764	720.0	4.873	3.500
28	U-235	264C	99.89	879.7	9.528	8.373
29	Pa-231	Pa-F-1	100.0	798.0	3.148	2.512
30	Pu-238	06HP014	99.38(p)	841.3(q)	3.740	3.127(q)
31	Th-230	256A	89.39	822.5	3.921	2.883
32	U-233	240A	99.886	797.8	9.602	7.652
33	Dos (U-235)	264C	99.89	879.7	1.182	1.039
	Dos (U-238)	ESZ	99.999	877.5	1.224	1.074
34	Dos (Pu-239)	453-BO	99.10	864.2	1.383	1.184
	Dos (Np-237)	24HP	99.99	874.0	1.355	1.184
35	Dos (Cu)	N	Normal	999.93	1.433	1.433
	Dos (Co)	CO-1	Normal	1.23	1.159	0.00143

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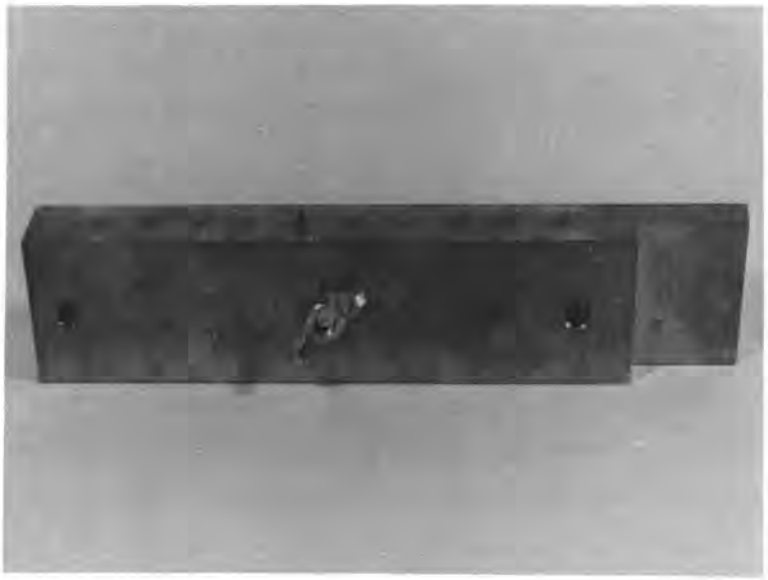

Fig. 20. Copper Chill Blocks Used During TIG Weld Closure of Vanadium Capsules to Maximize Heat Conductivity Away From the Actinide Oxide Powder

VI. LOADING AND WELDING FUEL PINS

A. Fuel Pellets

Loading of the actinide fuel pellets into the fuel pins was accomplished within the welding glove box, Figs. 13 and 14, using the secondary containment apparatus as shown in Fig. 15. After all fuel pins and loaded transfer containers from a particular actinide type were bagged into the welding glove box, the facility was isolated and evacuated. During this operation the transfer containers remained sealed to prevent the release of particulate contamination while the fuel pins were open to assure that they would ultimately contain no residual oxygen. After satisfactory evacuation of the facility, the welding glove box was backfilled with high purity helium gas (99.997% typical purity), and the facility was returned to the ventilation system to maintain negative pressure while a continuous purge of helium was maintained. After the glove box was purged to a sufficiently low oxygen level (200 ppm maximum) as indicated by the oxygen analyzer, the secondary containment apparatus,

Fig. 15, was valved into the off-gas system to effect an internal pressure of 2.5-7.5 kPa (10-30 in. H_2O) negative with reference to the glove box. The "loaded" ends of the four transfer containers were then screwed into the left rotating flange of the secondary containment apparatus, while still sealed by the internal screw plugs. Individual pin loading was accomplished by opening the "loaded" end of the transfer containers by removing the internal screw plug with the cross handle hexagonal wrench. The hexagonal wrench was sealed by an "o"-ring to the right rotating flange of the secondary containment apparatus. After plug removal, the wrench was withdrawn, the right side flange rotated and the "o"-ring sealed push rod was inserted into the opening of the transfer container to hold the pellets in position. At this point, the external and internal seals were removed from the exposed "unload" end of the transfer container and the pin loading adaptor screwed into the internal thread, Fig. 21. Under these conditions there was a relatively high flow of gas through the transfer container into the secondary containment apparatus which prevented escape of particulate contamination from within the transfer container to the welding box interior. After attachment of the appropriate fuel pin to the pin loading adaptor, the fuel pellets were pushed from the transfer container into the pin. The loaded pin was then removed from the adaptor, a tungsten spacer and spring were inserted, and the end plug was then welded in place. As each fuel pin was removed, the exposed end of the loading adaptor was covered and sealed with a plastic septum to prevent contamination and to maintain negative pressure within the secondary containment apparatus.

All peripheral closure welds were performed by personnel from the Core Technology and Safety Division of HEDL, using equipment and procedures developed and provided by that organization. All welding and inspection were performed in accordance with the requirements of Section 8, category la of specification RDT-F6-2T.

Three secondary containment apparatuses were constructed so that one could be used exclusively for each material type. The apparatus was removed intact from the welding box at the conclusion of the pin welding for a particular fuel pellet type. This system of material transfer proved

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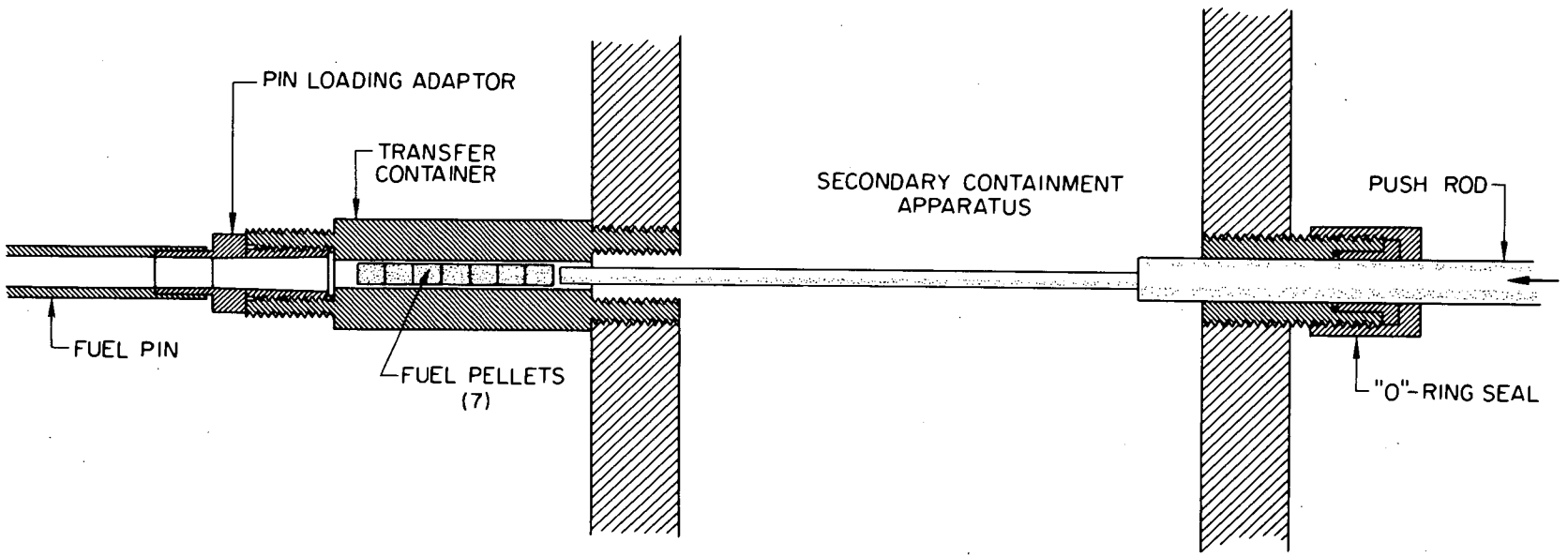

Fig. 21. Schematic of Secondary Containment Apparatus in Welding Box Detailing Transfer of Pellets From Transfer Container to Fuel Pin

very effective in providing welded fuel pins which were virtually free of surface contamination when removed from the glove box.

At the completion of the welding of all pins containing one material type, a gas sample was taken from the internal glove box atmosphere by filling an evacuated glass sampling container. These samples were subsequently analyzed by mass spectroscopy. Components of each sample are shown in Table 16 and represent the atmospheric composition in the completed pin segment.

After removal and inspection of the welded fuel pins, a helium leak test was performed on each pin using a commercial Veeco helium leak detector. The results are shown in Table 17. The leak test procedure is described in Appendix 4.

B. Physics Specimens and Dosimeters

The physics specimens and dosimeters were loaded into three fuel pins according to the arrangement shown in Table 18. The vanadium encapsulated materials were loaded into each fuel pin in five sections as shown in Fig. 22. Each section contained either physics specimens or dosimeters that were placed inside a stainless steel tube. The ends of the tube were crimped to prevent movement of the vanadium capsules. The five sections were then placed inside a longer stainless steel tube. Again, the ends of the tube were crimped to fix the location of the sections inside the tube. Each liner was loaded into a fuel pin and placed inside the welding glove box, Fig. 13. The welding box was evacuated and backfilled with helium. The lower end caps (lower end cap and spacer for FP4) were inserted in the fuel pins and the closure welds were made by HEDL personnel according to established procedures. After the pins were welded, a gas sample was taken of the glove box atmosphere and the results are shown in Table 16 as sample number 123. The welded fuel pins were leak checked according to the procedure described in Appendix 4, and the results are shown in Table 17.

C. Fuel Pin Radiography

Radiographic examination of the closure welds was accomplished by the use of 155 kV x-rays and with two films of different exposure speeds in each film holder. A flat block of 6.76-mm thick stainless steel containing a 5.94-mm diam hole along the length of the block was used as a shape correction mask.

Table 16. Gas Analyses of Glove Box Atmosphere During Welding Operations

Date	Sample Number	Compound	Atom Percent	Comments
5-21-81	123	H_2	0.0003	Gas sample taken after welding samples 4-7, 2-7, and 1-7. (Physics specimens and dosimeters)
		He	99.9744	
		CH_4	0.0001	
		H_2O	0.0000	
		Ne	0.0023	
		N_2	0.0168	
		O_2	0.0053	
		Ar	0.0004	
		CO_2	0.0000	
5-22-81	373	H_2	0.0002	Gas sample taken after welding samples 1-4, 2-4, 3-4, and 5-4. (Am_2O_3 fuel pellets)
		He	99.9743	
		CH_4	0.0001	
		H_2O	0.0001	
		Ne	0.0028	
		N_2	0.0159	
		O_2	0.0057	
		Ar	0.0004	
		CO_2	0.0001	
5-22-81	377	H_2	0.0008	Gas sample taken after welding samples 1-5, 2-5, 3-5, and 5-5. (M_2O_3 fuel pellets)
		He	99.992	
		CH_4	0.0002	
		H_2O	<0.0002	
		N_2+CO	0.0031	
		O_2	0.0009	
		Ar	0.0001	
		CO_2	0.0001	
5-23-81	385	H_2	<0.0001	Gas sample taken after welding samples 1-2 and 5-2. (Cm_2O_3 fuel pellets)
		He	99.9954	
		CH_4	<0.0002	
		H_2O	<0.0002	
		Ne	0.0004	
		N_2	0.0035	
		O_2	0.0005	
		Ar	<0.0001	
		CO_2	<0.0001	

Table 16 (Cont'd)

Date	Sample Number	Compound	Atom Percent	Comments
5-29-81	LG-NN	H_2	0.0001	Gas sample taken after welding samples 2-2, 3-2, and 5-1. (Cm_2O_3 and Am_2O_3 fuel pellets)
		He	99.9589	
		CH_4	0.0000	
		H_2O	0.0000	
		Ne	0.0014	
		N_2	0.0320	
		O_2	0.0075	
		Ar	0.0000	
		CO_2	0.0000	

Table 17. Helium Leak Test Results for Fuel Pellet and Physics Specimen Assemblies

Type	Capsule No.	Helium Leak Rate (Std. cc/s)
Physics Specimens	1-7	6.3×10^{-10}
Physics Specimens	2-7	8.9×10^{-10}
Physics Specimens	4-7	1.6×10^{-9}
Am_2O_3 Fuel Pellets	5-1	8.0×10^{-10}
Am_2O_3 Fuel Pellets	2-4	1.1×10^{-9}
Am_2O_3 Fuel Pellets	3-4	7.8×10^{-10}
Am_2O_3 Fuel Pellets	5-4	9.5×10^{-10}
Mixture Fuel Pellets	1-5	6.6×10^{-10}
Mixture Fuel Pellets	2-5	4.8×10^{-10}
Mixture Fuel Pellets	3-5	6.6×10^{-10}
Mixture Fuel Pellets	5-5	3.1×10^{-10}
Cm_2O_3 Fuel Pellets	1-2	1.1×10^{-9}
Cm_2O_3 Fuel Pellets	2-2	1.2×10^{-9}
Cm_2O_3 Fuel Pellets	3-2	1.3×10^{-9}
Cm_2O_3 Fuel Pellets	5-2	1.4×10^{-9}

Table 18. Vertical Ordering of Physics Specimens and Dosimeter Capsules for FP1, FP2, and FP4

Position	Isotope
1	Dos (U-235, U-238)
2	Dos (Pu-239, Np-237)
3	Dos (Cu, Co)
4	Cm-244
5	Cm-246
6	Cm-246
7	Np-237
8	Cm-244
9	Cm-244
10	Cm-243
11	U-238
12	Am-243
13	Am-243
14	Am-241
15	Am-241
16	Pu-242
17	Dos (U-235, U-238)
18	Dos (Pu-239, Np-237)
19	Dos (Cu, Co)
20	Pu-244
21	Pu-240
22	Pu-240
23	Pu-239
24	Pu-241
25	Th-232
26	U-236
27	U-234
28	U-235
29	Pa-231
30	Pu-238
31	Th-230
32	U-233
33	Dos (U-235, U-238)
34	Dos (Pu-239, Np-237)
35	Dos (Cu, Co)

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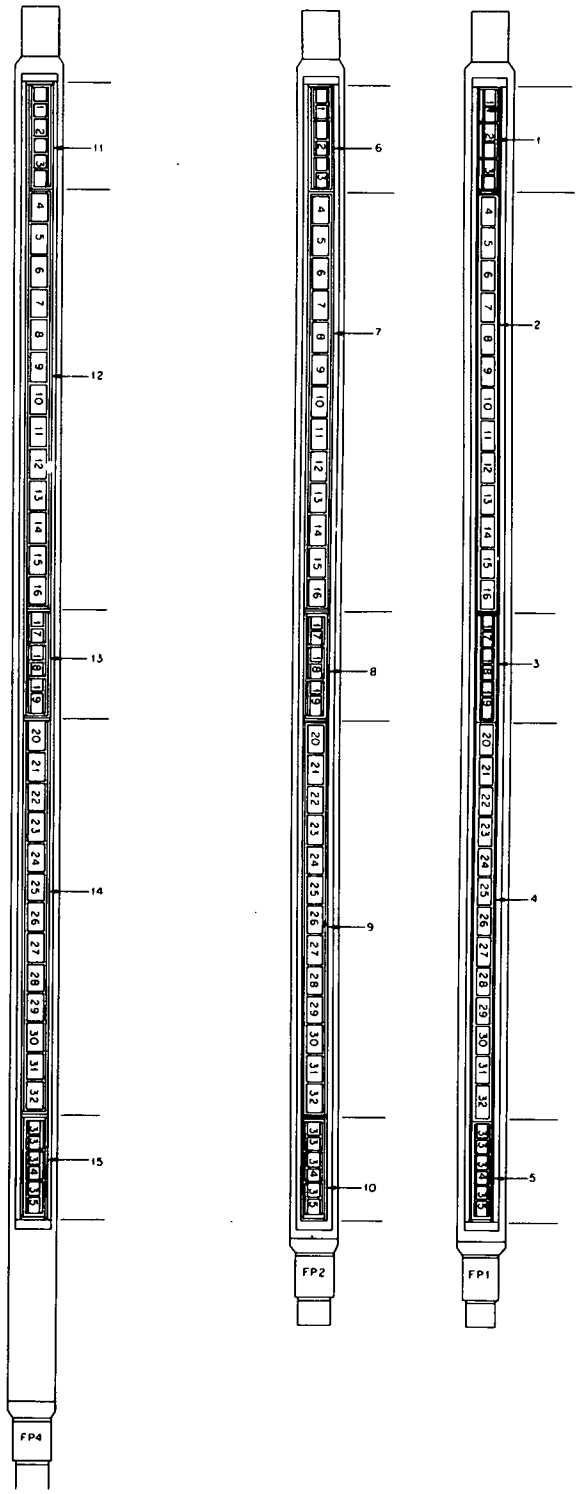

Fig. 22. Ordering of Physics Specimens and Dosimeters Inside Physics Specimen Sections of Fuel Pins (The numbers 1 through 35 refer to capsules listed in Tables 13-15. The numbers 1 through 15 refer to outer markings on short pin sections.)

The end of the pin having the closure weld was inserted into a plastic sleeve connected to a positioning device. This positioning device permitted indexed rotation of the pin and exposures were made on each pin every 36 degrees between 0 and 144 degrees around its circumference. The position of the weld in relation to the end cap required superimposing the source-side and film-side weld images.

Film viewing and interpretation were done under 10 X magnification. One film provided coverage of the thinner sections of the assembly. Radiographs revealed no indications of insufficient weld penetration nor lack of fusion; no indications of cracks or porosity were noted.

VII. SUMMARY

A cooperative US/UK research program was initiated approximately four years ago to investigate the neutronic and irradiation behavior of higher actinides in fast breeder reactors. This program eventually involved ORNL, HEDL, and laboratories in the UK at Dounreay, Scotland, and Windscale, England. The test "fuel" pin(s) designed to observe actinide behavior during irradiation in the Prototype Fast Reactor (PFR) at Dounreay contains three sections of actinide oxides (fuel pellets) in a normal fuel configuration and 21 actinide oxides (physics samples) encapsulated in vanadium. ORNL had specific responsibilities for selecting and providing the isotopes, for fabricating the fuel pellets, for fabricating the neutron dosimeters and physics samples, and for loading and seal-welding the fuel pin segments (with HEDL assistance). HEDL was responsible for test pin design, pin fabrication, final assembly of pin sections, quality assurance, and shipping to the UK. ORNL responsibilities pertaining to test pin preparation were successfully met by the middle of June 1981.

VIII. ACKNOWLEDGMENTS

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dosimeters, and fuel pellets for this experiment. The authors are indebted to the Office of Basic Energy Sciences, U. S. Department of Energy, for the transplutonium element production facilities at the Oak Ridge National Laboratory, which furnished the ^{240}Pu and all nuclides heavier than mass 242 that were used for the fabrication of physics specimens and for the ^{244}Cm used in the fabrication of fuel pellets. The authors gratefully acknowledge the extensive efforts of B. A. Caylor in the preparation of reports, papers, and all data presentations associated with this program. The diligent efforts of Harry Trotter and Ross Jones for craft and machine shop support during this program are appreciated. John Sites, Analytical Chemistry, performed all gas analyses reported in this operation--an invaluable service for which the authors extend their gratitude.

The present US/UK program is a joint one involving ORNL, HEDL and the UK. The authors have greatly benefited from discussions and interactions with J. A. Basmajian, K. R. Birney, R. L. Gibby, J. L. Jackson, E. T. Weber, and C. N. Wilson from HEDL and with B. C. Bateman, J. K. Butler, A. L. Mills, and K. M. Swanson from the UK. Finally, the authors thank G. F. Flanagan and U. Gat of ORNL, and J. W. Lewellen and P. B. Hemmig of DOE for their assistance and encouragement.

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APPENDIX 1

Analytical Data for Actinide Oxide Fuel Pellet Materials

I. Chemical Composition

A. Pellet Type I Material - $^{244}Cm_2O_3$ (Batch C57CM46)

Element or Atomic Weight	Quantity (ppm)	Element or Atomic Weight	Quantity (ppm)
Pb	0.04	245	~8000
Pt	1	244	Major
W	1	243	~10000
Ta	≤50	240	~70000
La	0.3	239	1
Ba	0.07	238	1
Sn	1	236	0.7
Cd	0.7	174	0.2
Rh	0.04	173	0.1
Zr	5	172	0.1
Y	0.005	171	0.1
Sr	0.1	170	0.7
Zn	0.1	169	1
Cu	1	168	60
Ni	40	167	5
Co	0.03	166	300
Fe	10	165	200
Mn	0.05	164	7
Cr	2	163	100
V	0.003	162	100
Ca	50	161	50
K	0.05	160	60
Cl	2	159	6
S	2	158	1
P	2	157	0.04
Si	5	156	8
Al	20	154	0.2
Mg	3	152	0.1
Na	5	150	0.1
F	20	148	0.6
B	0.1	147	0.05
Ce	20	146	0.9
Pr	0.2	145	0.08
248	200	144	1
247	300	143	0.01
246	~50000	Carbon	320

I. Chemical Composition (Cont'd)

B. Pellet Type II Material - $^{241}Am_2O_3$ (Batch 79AMB4)

Element or Atomic Weight	Quantity (ppm)	Element or Atomic Weight	Quantity (ppm)
U	10	Ni	~1000
Th	100	Co	10
Pt	500	Fe	300
Ho	0.7	Mn	60
Dy	100	Cr	300
Tb	1	V	0.3
Gd	4	Ti	8
Eu	5	Ca	20
Sm	50	K	0.2
Nd	300	S	20
Pr	40	Al	~200
Ce	100	B	0.1
La	40	Zn	0.2
Pd	3	239	10
Mo	4	237	~10000
Y	40		
Sr	0.2	Carbon	63
Cu	0.4		

II. Isotopic Composition

A. Pellet Type I Material - $^{244}Cm_2O_3$ (Batch C57CM46)

Curium[r]

Isotope	At. %
243	0.016 ±0.002
244	92.40 ±0.03
245	1.162 ±0.004
246	6.26 ±0.03
247	0.101 ±0.002
248	0.057 ±0.001

[r]June 16, 1981.

II. Isotopic Composition (Cont'd)

Plutonium[r]

Isotope	At. %
238	0.002 ±0.0005
239	0.048 ±0.001
240	99.922 ±0.01
241	0.004 ±0.001
242	0.024 ±0.001

[r]June 16, 1981.

Americium

Isotope	At. %
241	0.40 ±0.05
242	0.08 ±0.02
243	99.52 ±0.05

B. Pellet Type II Material - $^{241}Am_2O_3$ (Batch 79AMB4)

Isotope	At. %
241	99.995
242	<0.001
243	0.005

The remaining analytical data for the fuel pellet materials are summarized in Tables 1-1 through 1-3.

Table 1-1. Analytical Data for Type 1 Fuel Pellet Materials From Curium Batch C57CM46

Oxygen-to-Metal Ratio (O/M):	1.52
^{244}Cm Concentration	756.0 mg/g
^{240}Pu Concentration	67.0 mg/g
^{243}Am Concentration	1.6 mg/g

Pellet Number	Pressing(s) Sequence	Pellet Diameter (mm)	Pellet Length (mm)	Pellet Wt. (g)	Comments
1	1	3.747	3.813	0.4200	ORNL High Temperature Test Repeat Sample
2	1	3.744	3.785	0.4206	HEDL Analytical Samples (TC-M3)(t)
3	1	3.747	3.782	0.4216	HEDL Analytical Samples (TC-M3)(t)
4	1	3.741	3.815	0.4196	HEDL Archive Sample
5	1	3.744	3.790	0.4212	ORNL Analytical Sample
6	1	3.747	3.835	0.4218	ORNL Analytical Sample
7	1	3.741	3.830	0.4202	High Temperature Test Sample
		Column Length = 2.6721 cm			
1	2	3.739	3.406	0.4200	Loaded in Capsule No. 1-2
2	2	3.734	3.424	0.4189	Loaded in Capsule No. 1-2
3	2	3.736	3.475	0.4194	Loaded in Capsule No. 1-2
4	2	3.736	3.475	0.4196	Loaded in Capsule No. 1-2
5	2	3.741	3.457	0.4180	Loaded in Capsule No. 1-2
6	2	3.736	3.432	0.4185	Loaded in Capsule No. 1-2
7	2	3.736	3.429	0.4287	Loaded in Capsule No. 1-2
		Column Length = 2.3919 cm			
1	3	3.741	3.487	0.4239	Loaded in Capsule No. 2-2
2	3	3.741	3.493	0.4228	Loaded in Capsule No. 2-2
3	3	3.747	3.495	0.4236	Loaded in Capsule No. 2-2
4	3	3.741	3.493	0.4249	Loaded in Capsule No. 2-2
5	3	3.741	3.490	0.4233	Loaded in Capsule No. 2-2
6	3	3.744	3.508	0.4248	Loaded in Capsule No. 2-2
7	3	3.741	3.526	0.4244	Loaded in Capsule No. 2-2
		Column Length = 2.4407 cm			

Table 1-1 (Cont'd)

Pellet Number	Pressing(s) Sequence	Pellet Diameter (mm)	Pellet Length (mm)	Pellet Wt. (g)	Comments
1	4	3.736	3.475	0.4233	Loaded in Capsule No. 3-2
2	4	3.734	3.493	0.4227	Loaded in Capsule No. 3-2
3	4	3.736	3.467	0.4188	Loaded in Capsule No. 3-2
4	4	3.736	3.518	0.4213	Loaded in Capsule No. 3-2
5	4	3.734	3.513	0.4270	Loaded in Capsule No. 3-2
6	4	3.739	3.533	0.4246	Loaded in Capsule No. 3-2
7	4	3.734	3.523	0.4235	Loaded in Capsule No. 3-2

Column Length = 2.4534 cm

Table 1-2. Type II pellets - $^{241}Am_2O_3$ (Batch 79AMB4)

Oxygen-to-Metal Ratio (O/M): 1.492

^{241}Am Concentration: 888.0 mg/g

Actinide Oxide Weight, Pellet Diameter, Pellet Length and Column Length

Pellet Number	Pressing(s) Sequence	Pellet Diameter (mm)	Pellet Length (mm)	Pellet Wt. (g)	Comments
1	1	3.792	3.340	0.4132	ORNL Analytical Sample
	1	3.795	3.335	0.4132	ORNL Analytical Sample
3	1	3.807	3.378	0.4328	HEDL Analytical Sample (TC-M-1)(t)
4	1	3.795	3.373	0.4288	HEDL Analytical Sample (TC-M-1)(t)
5	1	3.797	3.373	0.4247	ORNL High Temperature Test Sample
6	1	3.805	3.307	0.4186	HEDL Archive Sample (TC-Am-1)(t)
7	1	3.795	3.401	0.4310	HEDL Analytical Sample (TC-M-1)(t)

Column Length = 2.3388 cm

Table 1-2 (Cont'd)

Pellet Number	Pressing(s) Sequence	Pellet Diameter (mm)	Pellet Length (mm)	Pellet Wt. (g)	Comments
1	2	3.792	3.510	0.4430	Loaded in Capsule No. 5-1
2	2	3.785	3.556	0.4410	Loaded in Capsule No. 5-1
3	2	3.795	3.523	0.4425	Loaded in Capsule No. 5-1
4	2	3.787	3.541	0.4411	Loaded in Capsule No. 5-1
5	2	3.790	3.546	0.4417	Loaded in Capsule No. 5-1
6	2	3.797	3.569	0.4417	Loaded in Capsule No. 5-1
7	2	3.792	3.523	0.4384	Pellet chipped and was removed from fuel pin
		Column Length = 2.4615 cm			
1	3	3.795	3.404	0.4263	Loaded in Capsule No. 2-4
2	3	3.785	3.353	0.4217	Loaded in Capsule No. 2-4
3	3	3.785	3.465	0.4366	Loaded in Capsule No. 2-4
4	3	3.785	3.376	0.4290	Loaded in Capsule No. 2-4
5	3	3.777	3.343	0.4072	Loaded in Capsule No. 2-4
6	3	3.787	3.340	0.4098	Loaded in Capsule No. 2-4
7	3	3.790	3.386	0.4291	Loaded in Capsule No. 2-4
		Column Length = 2.3594 cm			
1	4	3.805	3.526	0.4400	Loaded in Capsule No. 3-4
2	4	3.797	3.500	0.4407	Loaded in Capsule No. 3-4
3	4	3.810	3.493	0.4423	Loaded in Capsule No. 3-4
4	4	3.805	3.490	0.4400	Loaded in Capsule No. 3-4
5	4	3.800	3.498	0.4395	Loaded in Capsule No. 3-4
6	4	3.795	3.503	0.4391	Loaded in Capsule No. 3-4
7	4	3.800	3.513	0.4386	Loaded in Capsule No. 3-4
		Column Length = 2.4493 cm			

Table 1-3. Pellet Type III Material - M_2O_3

Oxygen-to-Metal Ratio (O/M): 1.532

^{241}Am Concentration 493.0 mg/g
^{244}Cm Concentration 64.4 mg/g

Actinide Oxide Weight, Pellet Diameter, Pellet Length and Column Length

Pellet Number	Pressing(s) Sequence	Pellet Diameter (mm)	Pellet Length (mm)	Pellet Wt. (g)	Comments
1	1	3.787	3.637	0.3719	ORNL High Temperature Test Sample
2	1	3.777	3.614	0.3723	HEDL Archive Sample
3	1	3.785	3.617	0.3724	HEDL Analytical Sample (TC-M-2)(t)
4	1	3.787	3.630	0.3746	HEDL Analytical Sample (TC-M-2)(t)
5	1	3.785	3.607	0.3725	HEDL Analytical Sample (TC-M-2)(t)
6	1	3.785	3.620	0.3749	ORNL Analytical Sample
7	1	3.785	3.599	0.3729	ORNL Analytical Sample
		Column Length = 2.5349 cm			
1	2	3.795	3.609	0.3726	Loaded in Capsule No. 1-5
2	2	3.792	3.635	0.3727	Loaded in Capsule No. 1-5
3	2	3.792	3.630	0.3747	Loaded in Capsule No. 1-5
4	2	3.795	3.597	0.3736	Loaded in Capsule No. 1-5
5	2	3.792	3.622	0.3738	Loaded in Capsule No. 1-5
6	2	3.805	3.622	0.3730	Loaded in Capsule No. 1-5
7	2	3.792	3.622	0.3719	Loaded in Capsule No. 1-5
		Column Length = 2.5334 cm			
1	3	3.795	3.635	0.3735	Loaded in Capsule No. 2-5
2	3	3.785	3.686	0.3746	Loaded in Capsule No. 2-5
3	3	3.790	3.673	0.3750	Loaded in Capsule No. 2-5
4	3	3.787	3.655	0.3726	Loaded in Capsule No. 2-5
5	3	3.785	3.688	0.3724	Loaded in Capsule No. 2-5
6	3	3.777	3.691	0.3723	Loaded in Capsule No. 2-5
7	3	3.785	3.627	0.3723	Loaded in Capsule No. 2-5
		Column Length = 2.5591 cm			

Table 1-3 (Cont'd)

Pellet Number	Pressing[s] Sequence	Pellet Diameter (mm)	Pellet Length (mm)	Pellet Wt. (g)	Comments
1	4	3.790	3.675	0.3732	Loaded in Capsule No. 3-5
2	4	3.802	3.653	0.3709	Loaded in Capsule No. 3-5
3	4	3.787	3.645	0.3733	Loaded in Capsule No. 3-5
4	4	3.785	3.691	0.3749	Loaded in Capsule No. 3-5
5	4	3.777	3.637	0.3716	Loaded in Capsule No. 3-5
6	4	3.787	3.650	0.3746	Loaded in Capsule No. 3-5
7	4	3.785	3.658	0.3730	Loaded in Capsule No. 3-5

Column Length = 2.5573 cm

[s]Seven pellets are pressed during each pressing operation.

[t]Transfer container number.

APPENDIX 2

M. L. Williams - Engineering Physics Division (ORNL)

Internal Gas Pressure Calculation for Encapsulated Physics Specimens

Calculations were made to predict fission-gas pressure generated by a 10-mg actinide sample irradiated in a fast reactor neutron flux of 6×10^{15}. Calculations were made using the computer code ORIGEN. In order to insure that the calculations would result in maximum pressure values, a fictitious material was assumed that had a spectrum-averaged fission cross section of 5.5b and gas yield corresponding to ^{239}Pu. The assumed fission cross section is higher than any of the actinide cross sections reported by HEDL based on ENDF/B-V data, and therefore it should provide an upper bound to the calculated gas pressure. The ^{239}Pu yield data are the only values currently available in the ORIGEN code.

Time-dependent concentrations in moles of the important gases produced in the irradiated sample are given in Table 2-1. The oxygen concentration shown results from release of oxygen from the oxide compound. The majority of oxygen as well as halogen fission products will combine with other materials and will not contribute to the gas pressure. In computing the total internal gas pressure it was assumed that 1% of these materials remain as pressure-contributing gases.

The time-dependent pressure in the capsule is given in Table 2-2. These values are based on a available volume of 4.2×10^{-3} cm^3 and a temperature of 920 K. The values in Table 2-2 were calculated from equation A2-2 derived below:

> At STP (T = 273 K, P = 101 kPa) 1 mole of ideal gas occupies 2.24×10^4 cm^3. Assuming the sample is well cooled and the capsule is thin enough so that the thermal gradient within the capsule is negligible, the temperature of the sample should be approximately equal to that of the coolant. The exit temperature of the coolant is approximately 923 K (650°C). Assuming ideal gas behavior, the pressure exerted by one mole of gas can be calculated according to the equation:

$$P_s = (101\ \text{kPa})\ \frac{(2.24) \times 10^4\ \ (923)}{(4.2) \times 10^{-3}\ \ (273)} = 1.82 \times 10^9\ \frac{\text{kPa}}{\text{mole}}\ . \qquad \text{(A2-1)}$$

Table 2-1. Time-Dependent Concentration (Moles) of Gases Released by a 10-mg Actinide[u] Sample for $\phi = 6 \times 10^{15}$

	Time, Days							
Element	30	90	120	150	200	250	300	370
He	4.78-11	9.11-10	1.98-9	3.58-9	7.58-9	1.33-8	2.08-8	3.40-8
Xe	7.04-7	1.95-6	2.50-6	3.00-6	3.73-6	4.37-6	4.91-6	5.54-6
I[v]	1.00-7	1.88-7	2.26-7	2.62-7	3.14-7	3.60-7	3.99-7	4.44-7
Kr	6.90-8	1.89-7	2.41-7	2.89-7	3.60-7	4.21-7	4.73-7	5.35-7
Br[w]	6.35-9	1.73-8	2.21-8	2.64-8	3.28-8	3.83-8	4.29-8	4.84-8
O[x]	6.80-6	1.87-5	2.39-5	2.87-5	3.57-5	4.18-5	4.70-5	5.31-5
Total Inert	7.73-7	2.14-6	2.74-6	3.29-6	4.10-6	4.80-6	5.40-6	6.11-6
Total	7.68-6	2.10-5	2.69-5	3.25-5	4.01-5	4.70-5	5.28-5	5.97-5

[u]Fictitious "worst case" actinide with ^{241}Pu decay constant $\sigma_f = 5.5b$, ^{239}Pu gas yield data.

[v]Boiling point = 457 K.

[w]Boiling point = 332 K.

[x]Produced by release of oxygen from PuO_2.

From the law of partial pressures: $P_{Tot} = \sum_i (m_i\ P) = P(\sum_i m_i)$, where m_i is equal to the number of moles of fission gas "i".

Therefore,

$$P_{Tot} = 1.82 \times 10^9\ m(t), \qquad \text{(A2-2)}$$

where m(t) is equal to the number of moles of gas at time t.

Table 2-2. Gas Pressure as a Function of Time

Days of Exposure	Available Gas[y] (Moles)	Pressure (MPa)
30	8.42×10^{-7}	1.54
90	2.33×10^{-6}	4.25
120	2.98×10^{-6}	5.45
150	3.58×10^{-6}	6.54
200	4.46×10^{-6}	8.15
250	5.22×10^{-6}	9.54
300	5.87×10^{-6}	10.7
370	6.65×10^{-6}	12.1

[y]Available gas assumed to be composed of 100% of inert fission product gases plus 1% of released oxygen and halogen fission product gases.

After 90 days the pressure in the capsule is about 4.25 MPa and after a full year of irradiation it is about 12.1 MPa. These values should be very conservative (i.e., high) for the actinide samples since the 5.5b fission cross section is much higher than the actual value for most of the nuclides used. The spectrum-averaged cross sections used by HEDL in predicting heat generation rates are given in Table 2-3. It can be seen from Table 2-3 that the assumed 5.5b cross section is more than an order of magnitude higher than the ^{241}Am value and is almost a factor of two higher than the ^{243}Cm and ^{245}Cm values. Therefore the calculated pressures shown should be regarded as an upper bound rather than pertaining to any specific actinide sample.

The allowable internal pressure for vanadium capsules is calculated below:

$$\text{Maximum Stress} = \text{Pressure}\left[\frac{(\text{Inside Radius})^2}{(\text{Outside Radius})^2-(\text{Inside Radius})^2}\right] \qquad \text{(A2-3)}$$

Table 2-3. HEDL ENDF/B-V Cross Sections

Isotope	Fission Cross Section, (Barns)	Capture Cross Section, (Barns)
^{241}Am	0.1959	2.4370 (heating 2.0422 transmutation)
^{242}Am	5.2646	0.63190
^{243}Am	0.1476	1.6738
^{242}Cm	0.0938	0.5993
^{243}Cm	3.3496	0.3870
^{244}Cm	0.3045	1.1799
^{245}Cm	3.1092	0.4094
^{246}Cm	0.1720	0.3667
^{238}Pu	1.1304	1.0916
^{239}Pu	2.1263	0.8503
^{240}Pu	0.2737	0.9104
^{241}Pu	2.1263	0.8503
^{242}Pu	0.1803	0.6371

Using an extrapolated yield stress of 206.84 MPa for vanadium at 923 K,[5] the maximum allowable pressure is:

$$\text{Pressure} = \frac{206.84}{\dfrac{(0.02)^2}{(0.03)^2 - (0.02)^2}} = \frac{206.84}{0.8} = 258.6 \text{ MPa.} \quad \text{(A2-4)}$$

Thus, the maximum allowable internal pressure for the smallest physics specimen vanadium capsule exceeds by more than a factor of 20 the calculated value for gas pressure generated from irradiating an actinide material with a cross section of 5.5b.

APPENDIX 3

Substitution Weighing Scheme Used for Determining Weights of Material Loaded into Physics Specimen and Dosimeter Capsules

The recommended weighing scheme is that described by the National Bureau of Standards in a paper, "Design and Test of Standards of Mass" in Precision Measurement and Calibration, Optics Metrology and Radiation Handbook 77, Vol. III, United States Department of Commerce (1961). In this scheme, three similar size specimens and a similar size standard weight are each weighed by themselves three times in a particular specified sequence. The balance reading is recorded after each weighing. The balance zero is not recorded or adjusted, but is allowed to drift. This can be allowed because the weights of the unknown specimens are determined solely by differences between themselves and the standard.

The following FORTRAN computer program is used for data reduction. The final output contains the three weights together with their 2σ uncertainties.

```
  1 I = 1
  5 J = 2
 10 K = 3
 20 READ (5, 30) TITLE
 30 FORMAT (20A4)
 40 READ (5, 50, END = 300) X, SEN, STD
 50 FORMAT (8F 10.5)
 60 A = SEN * (X(1) - X(2))
 70 B = SEN * (X(3) - X(4))
 80 C = SEN * (X(5) - X(6))
 90 D = SEN * (X(7) - X(8))
100 E = SEN * (X(9) - X(10))
110 F = SEN * (X(11) - X(12))
120 IF(ABS(X(2) - STD) . GE . 2.000) GO TO 300
130 W(I) = ((2.*A + B + C + D + E)/4.) + STD
140 W(J) = ((A + 2.* B + C - D + F)/4.) + STD
150 W(K) = ((A + C + 2.*C - E - F)/4.) + STD
```

```
160 R1 = A - (W(I) - STD)
170 R2 = B - (W(J) - STD)
180 R3 = C - (W(K) - STD)
190 R4 = D - (W(I) - W(J))
200 R5 = E - (W(I) - W(K))
210 R6 = F - (W(J) - W(K))
  C  U = UNCERTAINTY AT THE 95 PERCENT CONFIDENCE INTERVAL
     U = 3.182 (0.70711 * (SQRT((R1**2 + R2**2 + R3**2 + R4**2 +
         R5**2 1 + R6**2)/3.)
220 WRITE (6,230) TITLE
230 FORMAT(1H1, 'B DOS WGTS',//,1X,20A4,//,
    1'SAMPLE MASS (MG)',/)
240 WRITE (6,250) I, W(I),J, W(J), K, W(K)
250 FORMAT (I7, F12.4,/,I7,F12..4,/,I7,F12.4(
    WRITE (6,255) SEN, STD, U
255 FORMAT ('SENSITIVITY = ',F7.4./,'STANDARD = ',F8.4,//,
    1' UNCERTAINTY = ', 1PE12.5,/)
260 I = I + 3
270 J = J + 3
280 K = K + 3
290 GO TO 40
300 STOP
    END
```

All standard weights used in the dosimeter program are calibrated using a Class M weight set M65829. This set was calibrated by Y-12 plant personnel using weight set M53800 which was calibrated previously by comparison with NBS set 232.09 - 212230 - A.

Prior to loading the physics specimens, the vanadium capsules are weighed according to the above described weighing scheme. Actinide oxide powders are loaded into the capsules using funneled powder loaders and the capsules are weighed again according to the substitution scheme. The contained oxide powder weight is determined by difference.

Prior to loading the dosimeter specimens, small pieces of "oxide" wire (for the actinide dosimeter materials) or small pieces of metal wire

or foil for the stable dosimeter materials are cut. Each piece is roughly weighed to verify that it is within the required weight range. The substitution weighing scheme is then used to determine the final weight and the related 2σ uncertainty. All physics and dosimeter specimens are loaded in helium.

APPENDIX 4

Helium Leak Test Procedure Used for Actinide Fuel Pellets and Physics Specimen Assemblies

I. Calibration of Mass Spectrometer Leak Detector (MSLD)

A. The MSLD shall be calibrated using a standard leak in the 10^{-7} to 10^{-10} atm. cm^3/s range. Prior to calibration the machine background shall be adjusted to zero.

B. The sensitivity shall be calculated using the equation

$$\text{Sensitivity} = \frac{\text{Standard Leak Rate}}{\text{Output}} \qquad \text{(A4-1)}$$

where:

Sensitivity = sensitivity of the leak detector in atm. cc/s/division,

Standard leak rate = leak rate as specified on standard helium leak in atm. cc/s, and

Output = net divisions.

C. The MSLD is acceptable for quantitative work if the sensitivity is better than 1×10^{-9} atm. cc/s/division.

D. Calibration will be performed in accordance with the following schedule:

1. At the beginning and end of each four-hour period of continued use.
2. At the beginning of each operating period if operation is not continuous.
3. At any time the operator suspects the equipment is performing erratically.

II. System Calibration

A. The entire vacuum chamber testing system shall be calibrated.

B. The system background shall not be zeroed.

C. The background reading shall be less than one-tenth of the maximum leakage allowed for the part tested and not exceed 100 percent of the most sensitive scale.

D. The sensitivity of the system shall be calculated using the equation

$$\text{Sensitivity} = \frac{\text{Standard Leak Rate}}{\text{Total Output-Background}} \qquad \text{(A4-2)}$$

where:

Total output and background are determined in net divisions.

III. Total Leak Rate Measurement of Specimens Placed Inside a Vacuum Chamber Assembly

A. The system sensitivity shall be determined in accordance with procedures outlined in Part II above.

B. Place individual specimen assemblies that have been welded in a helium atmosphere in the vacuum chamber and evaluate the chamber.

C. Initiate the testing cycle.

D. When meter output stabilizes, compute the leak rate by using the equation

$$\text{Leak Rate} = \text{Sensitivity} \times \text{Output} \qquad \text{(A4-3)}$$

where:

Leak Rate = Leak rate of unknown leak in atm. cc/s,

Sensitivity = Sensitivity of leak detector in atm./s/division, and

Output = Net divisions.

E. Document results.

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