

FEB 23 1967

WAPD-TM-588
AEC RESEARCH AND
DEVELOPMENT REPORT

MASTER

FABRICATION OF FUEL RODS
CONTAINING U-233 PELLETIZED
OXIDE FUELS

FEBRUARY 1967

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

CONTRACT AT-11-1-GEN-14

BETTIS ATOMIC POWER LABORATORY, PITTSBURGH, PA.,
OPERATED FOR THE U. S. ATOMIC ENERGY COMMISSION
BY WESTINGHOUSE ELECTRIC CORPORATION



DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

UC-25: Metals, Ceramics, and Materials
Special Distribution

FABRICATION OF FUEL RODS CONTAINING
U-233 PELLETTIZED OXIDE FUELS

W. L. Frankhouser
H. H. Crain
R. L. Fischer
J. H. Suldán
J. H. Eyler
E. L. Speer

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

February 1967

Contract AT-11-1-GEN-14

Printed in the United States of America
Available from

Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia 22151

Price: Printed Copy \$3.00; Microfiche \$0.65

NOTE

This document is an interim memorandum prepared primarily for internal reference and does not represent a final expression of the opinion of Westinghouse. When this memorandum is distributed externally, it is with the express understanding that Westinghouse makes no representation as to completeness, accuracy, or usability of information contained therein.

BETTIS ATOMIC POWER LABORATORY, PITTSBURGH, PA.,
OPERATED FOR THE U. S. ATOMIC ENERGY COMMISSION
BY WESTINGHOUSE ELECTRIC CORPORATION

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission, makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor or contract disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

SPECIAL EXTERNAL DISTRIBUTION

	No. Copies
AEC, Washington, D.C., J. M. Simmons	1
Argonne National Laboratory	9
Atomics International	4
Babcock and Wilcox Company	2
Battelle Memorial Institute	2
Battelle-Northwest	6
Brookhaven National Laboratory	4
Bureau of Mines, Albany	1
Combustion Engineering, Inc. (NRD)	1
Dow Chemical Company, Rocky Flats	1
Du Pont Company, Aiken	4
General Atomic Division	2
General Electric Company, Cincinnati	2
General Electric Company, San Jose	2
IIT Research Institute	1
Iowa State University	2
Knolls Atomic Power Laboratory	3
Los Alamos Scientific Laboratory	3
Mound Laboratory	1
National Reactor Testing Station (INC)	4
Naval Research Laboratory	3
Nuclear Materials and Equipment Corp.	1
Union Carbide Corporation (ORNL)	5
University of California, Livermore	2
Westinghouse Electric Corporation	2
DTIE	3
Manager, PNR	7
Total	<hr/> 78

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

CONTENTS

	<u>Page</u>
I. INTRODUCTION	1
II. ZIRCALOY-2 CLADDING	2
III. URANYL (-233) NITRATE	3
IV. FABRICATION OF FUEL PELLETS	6
V. FABRICATION OF FUEL RODS	9
VI. SHIPPING OF FUEL RODS	12
VII. CONCLUSIONS	14
ACKNOWLEDGMENT	15

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Sketches of Blanket- and Seed-Type Rods for U-233 Program.	31
2	Natural Decay of U-232 in U-233 Fuel.	32
3	Buildup of Radioactivity in U-233 Fuel.	32
4	Typical Microstructures of Seed-Size Tubing.	33
5	Compositions of Solutions Introduced and Removed during Solvent Extraction.	34
6	Flow Sheet for Solvent Extraction of Uranyl Nitrate.	35
7	Shipping Container Used to Transport U-233 Nitrate to NFS.	36
8	UO ₂ -ThO ₂ Microstructure.	37
9	UO ₂ -ZrO ₂ Microstructure.	38
10	Radioactive Buildup of U-233 Fuels.	39
11	Tube Loading Fixture.	39
12	TIG Welding Setup for U-233 Rods.	40
13	Typical Blanket End-Closure Weld.	41

LIST OF FIGURES (Cont)

<u>Figure</u>		<u>Page</u>
14	Section through Typical Blanket End-Closure Weld.	41
15	Fuel Element Shipping Container.	42

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Inspection of Cladding Tubes	16
2	Results of Chemical Analysis for Impurities	17
3	Reported Chemistry for the Shipments of Uranyl (U-233) Nitrate and Bettis Recheck of U and Th Isotopic Analyses	19
4	Analyses of ThO ₂ Powder and 1 w/o U ²³³ O ₂ -ThO ₂ Pellet	20
5	Summary of U ²³³ O ₂ Powder Production	21
6	Summary of U-233 Pellet Fabrication	22
7	U ²³³ O ₂ -ThO ₂ Measurements of Density of Production Blanket Pellets	24
8	U ²³³ O ₂ -ZrO ₂ Measurements of Density of Production Seed Pellets	25
9	Summary of NFS U-233 Material Balances	26
10	Uranium Analyses of Preproduction (NFS) and Production Pellets	27
11	Abridged Process Outline for Processing of Fuel Rods	28
12	Exposure of Personnel during Fuel Rod Manufacture	29
13	Fuel Rod Yields	30
14	Activity Levels of Fuel Rod Shipping Containers	30

Approximately 13 kg of U-233 were fabricated into 1 w/o $\text{UO}_2\text{-ThO}_2$ and 26 w/o $\text{UO}_2\text{-ZrO}_2$ pellets and enclosed in Zircaloy-2 cladding tubes as 1299 blanket and 377 seed rods. The U-233 contained 38 ppm of U-232, and was purified as two batches of liquid uranyl nitrate immediately prior to processing into powder form. All fuel rods were completed within 95 days of this initial solvent extraction of the nitrate. Overall uranium yield of nitrate to usable materials was over 90 percent, and only 14 rods were rejected in welding - but without loss of contained fuel.

FABRICATION OF FUEL RODS CONTAINING U-233 PELLETIZED OXIDE FUELS

W. L. Frankhouser
H. H. Crain
R. L. Fischer
J. H. Suldan
J. H. Eyler
E. L. Speer

I. INTRODUCTION

Fuel rods containing U-233 ceramic pellets encapsulated in Zircaloy cladding were required for a physics critical experiment to be conducted at the Bettis Atomic Power Laboratory as part of the LSBR development program. The work demonstrated the feasibility of processing these fuels in pellet form rather than as powder which is vibratory-packed in cladding tubes. This program was completed within 10-1/2 months (including selection of vendors, placement of orders, and the actual fabrication of components and final product). Zircaloy-2 cladding tubes were supplied by Wolverine Tube Division (WTD) of Calumet Hecla, Inc. Fuel pellets were fabricated by Nuclear Fuel Services (NFS) and loaded into cladding tubes with one end being previously sealed. Nuclear Materials and Equipment Corporation (NUMEC) performed all welding and inspection of the rods and provided the fuel rod shipping containers. The initial U-233 product was supplied by the AEC from the Pilot Plant Facility at Oak Ridge National Laboratory (ORNL). This latter material was purified by solvent extraction immediately prior to its shipment to NFS in the form of liquid uranyl nitrate.

A total of 1299 blanket and 377 seed fuel rods, as illustrated in Figure 1, was fabricated and shipped. The blanket rods contained 1 w/o $\text{U}^{233}\text{O}_2\text{-ThO}_2$ ceramic pellets, and 26 w/o $\text{U}^{233}\text{O}_2\text{-ZrO}_2$ was the composition for the seed rods. Cladding

material was supplied as cut-length tubes, and as oversize rolled-rod stock which was subsequently machined into endplugs and spacers. This tubing is considered to be free-standing for intended applications to 500°F temperature and 650 psi pressure. As shown in Figure 1, each rod contained a 15.00 ± 0.06 -inch stack of fuel pellets which were fabricated by conventional dry-pressing and sintering. Two-inch spacer plugs separated fuel from endcaps, and an axial clearance of 0.02 to 0.13 inch was provided in each rod for differential expansion of fuel and cladding. Diametral assembly clearance between fuel and cladding was from 0.002 to 0.012 inch for the seed rods and from 0.003 to 0.018 inch for the larger blanket rods.

It was recognized that a potential problem area was the buildup of gamma activity associated with decay of U-232, as illustrated in Figures 2 and 3. For that reason, attention was directed both to maximum reduction of U-232 daughters in initial solvent extraction of uranium stock and to close scheduling among all participants in reducing overall production time. Accordingly, actions taken by Bettis were:

- (1) To complete all qualification and preproduction operations with depleted uranium prior to purification of U-233.
- (2) Following (1), above, and also prior to U-233 purification, to conduct a complete production run using depleted uranium with inspections at each stage as required by specification.
- (3) To ship U-233 immediately upon completion of purification, and in a separate batch for both blanket and seed fuel rods.
- (4) To seal one end of cladding tubes prior to loading of fuel pellets.
- (5) To provide tight control of processes, developed in (1) and (2) above, on the actual U-233 production run as the major guard for product quality, and to keep delays in U-233 processing to a minimum by relying on the prior inspection techniques and the process control of production.

These actions were expected to minimize both the fabrication period and the shielding of operations or rotation of personnel to avoid exceeding acceptable dose levels. Data concerning gamma activity levels and safety precautions required are presented where available.

II. ZIRCALOY-2 CLADDING

The Zircaloy-2 cladding tubes were fabricated by tube reducing from 3-inch OD shells, except for finish sinking and plug-draw passes on a bench for the

seed size. A final recrystallization anneal at 1200°F for 4 hours provided the fine-grained structure shown in Figure 4. Pieces were cut to final lengths by WTD; ID and wall dimensions were consistent with tolerances of Figure 1. A summary of other pertinent inspection information is provided in Table 1.

The tubing is considered to be adequate for the program described. More recently, tubing requirements, particularly for higher strengths, have been modified to satisfy new program and design requirements.

The Zircaloy rods used as machining stock for endplugs and spacers were fabricated by conventional hot-rolling of billets. The seed size material was finished with a final recrystallization anneal at 1300°F and air cooled, and the blanket size was cold reduced 5 to 10 percent by cold drawings and annealed at 1300°F for finishing. Microstructure of this material was similar to the tubing of Figure 4, and physical properties such as corrosion resistance, strength, etc., met the conventional requirements for Zircaloy.

III. URANYL (-233) NITRATE

Approximately 13 kg (12.989) of contained U-233 as uranyl nitrate were made available and solvent extracted by ORNL. The primary objective of this purification was to break the decay chain of Figure 2 between the U-232 and its daughters by removing the daughters with the associated gamma activity. The shipments of the purified material were divided into batches of 6.950 and 6.039 kg of U-233 which were delivered two weeks apart.

The solvent extraction was performed by the Pilot Plant at ORNL, as shown in Figures 5 and 6. This operation consisted of a two-column system, comprised of an extraction-scrub column and a stripping column. Each column was 34-feet long with feed and take-off points such that 14 feet of column height were available for uranium extraction, and 19 feet for scrubbing in the extraction-scrubbing column, while 20 feet were available for stripping in the second column. The extraction column was fitted with orificed pulse or contactor plates (23 percent open area) at 2-inch intervals. The process involved the use of an aqueous solution, uranyl nitrate, and an organic solution, di-secondary butyl phenyl phosphonate (DSBPP) in di-ethyl benzene, as the immiscible solvent pair. A 2-1/2 percent solution of the DSBPP in the benzene was introduced near the bottom of the extraction column counterflow to the uranyl nitrate feed solution which was introduced near the middle of the column. Natural thorium (Th-232) nitrate solution had been added to the uranyl nitrate solution to adjust the concentration

of this aqueous solution to approximately 200 grams of thorium and 5 grams of uranium per liter of solution. The DSBPP solution, being lighter than the uranyl nitrate-thorium nitrate solution, passed upwards through the extraction column while taking the uranyl nitrate into solution. The thorium nitrate, being insoluble in the organic solution, passed down the column. At the top of the scrubbing section of the column, a concentrated aqueous phase solution of aluminum nitrate was added. This aluminum nitrate loaded the aqueous phase within the column, forcing the uranyl nitrate to remain in the organic phase, and washed out any entrained impurity. As the aluminum nitrate passed down the column into the extraction section it aided the uranyl nitrate to pass into the organic phase.

For the removal of uranyl nitrate, the DSBPP was introduced near the bottom of the second (or stripping) column with pure water entering at the top of the column. The uranyl nitrate, being more soluble in the water, was stripped from the organic solvent. The stripped DSBPP was washed and returned to the first column for reuse, while the uranyl nitrate solution was concentrated by evaporation to approximately 125 grams of U-233 per liter of solution.

The original introduction of the thorium nitrate to the feed material not only loaded the aqueous solution, forcing the uranyl nitrate to pass into the organic solvent, but also diluted the Th-228 (the first daughter element of the U-232) in the uranyl nitrate. The two isotopes of thorium, being the same chemically, reacted similarly in the extraction column; thus, whatever thorium was carried into the final product of the extraction process was quite dilute in Th-228 (see Figure 5).

Because of criticality as well as health and safety requirements, a special cask (Figure 7) had to be used for the shipment of the uranyl nitrate solution to NFS. This was an existing AEC unit, and contained a 180-liter bottle of annular shape (approximately 2-inches wide x 3-1/4 feet in diameter x 4-feet high) and was shielded externally with 6 to 8 inches of concrete. Because of a total unloaded weight of approximately 8 tons, it was fastened to the bed of a large truck for transporting.

Analyses for uranium isotopes and impurity content of the uranyl nitrate shipped are reported on the left side of Tables 2 and 3. It was reported by ORNL that the two batches were more pure in levels of impurities and gamma activity than any other batch processed previously. This was attributed to the close control of the concentrations of the feed solution maintained by the operating personnel and to the fact that the two batches, approximately 6.9 and 6.1 kg,

were larger than normally processed. Criticality limitation had previously restricted the batch size to 5 kg, but this limit was raised to 8 kg prior to processing the two Bettis batches. The level of gamma activity buildup was slower than anticipated since one of the elements in the decay chain of the U-232 is radon, a gas which is drawn off while maintaining a vacuum in the shipping cask.

Many of the detection limits for impurities in the chemical analyses were at relatively high levels. Recently, it was desired to know the actual impurity levels, and impurity analyses of representative UO_2 powder and of 1 w/o UO_2 - ThO_2 pellets were performed on the spark-source mass spectrometer at Bettis with considerably lower detection limits. These results are published on the right-hand side of Tables 2 and 4 for UO_2 powder and 1 w/o UO_2 - ThO_2 pellets, respectively. In the latter table these results can be compared with analyses of the original ThO_2 powder and with analyses of uranyl nitrate in Table 2. In addition, Bettis rechecked the isotopic uranium analyses of the UO_2 powder and the thorium content, and the results are reported on the right-hand side of Table 3.

A comparison of the two sets of analyses for liquid nitrate and UO_2 powder in Table 2 provides a comparison in some cases which indicates the contamination introduced during powder preparation. The increase in Fe, Co, Cr, Mo, Ni, Si, and W are not unexpected since these are elements found in molybdenum, Inconel, steel, tool steel, and stainless steel processing equipment. In addition, the reported segregation of five of these elements indicates that the contamination was introduced in particulate form. The segregation of Ca and Ti is not readily explained, nor is the observed pickup of Al and Mg, but these latter elements must have been introduced from shop equipment also. It can be noted also that Na is reduced during powder making.

The comparison of ORNL and Bettis isotopic analyses in Table 3 shows good agreement except in the case of U-236. Again, there is no ready explanation for this difference, but it probably results from a difference in analytical procedures.

The differences in analyses of ThO_2 powder and pellets of Table 4, when comparisons can be made, provide an insight into contamination introduced during pellet making. Again, the steel and Inconel constituents Fe, Co, Cr, Ni, and Si were picked up in significant amounts, as expected. Ca, Ti, and Mg levels were also raised as before, indicating that there must be a source of these contaminants in the vendor's shop. There was no opportunity in these comparisons to

determine whether Na was reduced, as in Table 2, but it is clear that Cl was reduced during pellet making. The increases in rare earth contents, such as Dy, Gd, and Nd, are not believed to be real since there have been many problems in analyzing for these elements with the mass spectrometer, and segregations of the elements have been easily detected in the thorium starting stock (thorium nitrate tetrahydrate crystals).

IV. FABRICATION OF FUEL PELLETS

The U-233 was processed from the solution of uranyl nitrate into high-density seed and blanket fuel-pellet stacks and loaded into fuel tubes by NFS. These fuel-tube assemblies were loaded with 1 w/o $U^{233}O_2$ - ThO_2 (blanket) fuel or with 26 w/o $U^{233}O_2$ - ZrO_2 (seed) fuel. Process descriptions for NFS operations are presented as a summary outline in Tables 5 and 6 for powder and pellet production, respectively.

Due to the high levels of radioactivity encountered during the processing of U-233, decontamination of equipment becomes an important consideration. It was therefore decided that NFS would perform a complete preproduction run of a number of fuel pellet stacks equal to the production quantity of each type using depleted UO_2 , and perform all chemical analyses. One lot each of ThO_2 and ZrO_2 powder was blended which would be large enough for both preproduction and production, thus assuring no variations due to materials. Based on the preproduction results, the production run was then made with extremely close process control and a minimum of analyses. This presented a very definite cost savings for this test, by eliminating procurement of special equipment, such as spectrographic equipment for impurity analysis and autoclaves for corrosion testing, which it was felt could not be successfully decontaminated at reasonable cost. A separate purchase order was issued to ORNL for performance of uranium content and isotopic analysis on representative production pellets as an additional control. At the completion of the preproduction program, all inspection data were evaluated and considered acceptable. Production was then initiated.

Visual standards for surface defects, color, and metallography were selected from the preproduction pellets for use in production inspection. Geometric density of the $U^{233}O_2$ - ThO_2 pellets averaged 94.07 percent of theoretical (10.04 g/cc) which was within the specified nominal of 96.0 ± 3 percent. Average geometric densities for the various batches are shown in Table 7. The maximum and minimum values recorded for the 500 samples measured were 96.81 and 90.53 percent of theoretical, respectively.

Because of the size of the seed pellets (0.210-inch diameter) and the fact that handling inside glove boxes was required, it was extremely difficult to obtain accurate dimensions for density calculations. Therefore, the water displacement method was used. Each sample consisted of five pellets to increase the accuracy of the weighings because of vibrations within the glove box. As shown in Table 8, the density for the $U^{233}O_2$ - ThO_2 pellets averaged 93.42 percent of theoretical (6.94 g/cc), with maximum and minimum measurements of 95.82 and 88.47 percent of theoretical, respectively.

Perpendicularity and flatness of the end-faces of the pellets were maintained below 0.004 and 0.003 inch, respectively, at an assurance level of 95 x 95 percent. Pellet length measurements were not required since only stack height of the fuel in the tube was considered to be important. The UO_2 - ThO_2 blanket pellets were fabricated to a length-to-diameter ratio of 1.2/1.0, while the smaller diameter UO_2 - ZrO_2 seed pellets were fabricated to a length-to-diameter ratio of approximately 2.5/1.0. Diameters were 100 percent inspected with go, no-gogages, and the oversize pellets were ground (approximately 50 percent of each type). Pellets having rejectable end chips were salvaged by reducing their lengths on a cut-off wheel; this aided in obtaining stack heights and in increasing pellet yield to more than 90 percent as shown in Table 9.

Two pellets from each batch were sent to ORNL and were analyzed for uranium content and isotopic uranium. The results, in Table 10, showed an average of 0.939 percent uranium in the UO_2 - ThO_2 pellets and 24.47 percent uranium in the UO_2 - ZrO_2 pellets. Isotopic analysis showed 97.19 percent of the uranium as U-233 in the UO_2 - ThO_2 and 97.29 percent as U-233 in the UO_2 - ZrO_2 . The production results for the UO_2 - ThO_2 (0.939 percent total uranium) are slightly higher than the preproduction results, due to a misinterpretation by the vendor. Batch calculations for production were based on 0.91 w/o U-233 instead of 0.91 w/o total uranium as required per the specification.

No corrosion testing was performed on the U-233 pellets. Samples from each batch of the preproduction pellets were tested at Bettis, and showed an average weight change of -0.0337 mg/cm² and a maximum weight change of +0.9952 mg/cm² for the UO_2 - ThO_2 pellets, and an average and maximum of +0.1366 mg/cm² and +1.190 mg/cm², respectively, for the UO_2 - ZrO_2 pellets. All corrosion tests were performed at 750°F and 2000 psi in steam for 3 days.

Two metallographic samples were examined from each batch of preproduction and production pellets for internal porosity and to assure that the material was single phase. Typical microstructures and internal porosity samples of each

composition are shown in Figures 8 and 9. In addition, two pellets of each composition of the preproduction were analyzed for uranium heterogeneity at Bettis with the electron microprobe. The microprobe analyses showed the material to be homogeneous without detectable areas of uranium segregation.

The decay of U-232 presented problems with increasing radioactivity during processing. To minimize the contamination of buildings and work areas, all processing was done within air-tight glove boxes. The glove boxes were specially designed and arranged in such a fashion as to permit a constant flow of material with a minimum number of transfers from one line to another. The material was hand-transferred for all operations from each box to the next adjoining box on the line via air-tight tunnels through final pellet production and loading into cladding tubes. Special ports were included in each line to facilitate enclosing all materials, when necessary to be removed from the box, in plastic bags without contaminating the exterior of the bags. A negative air pressure was maintained within the boxes and tunnels at all times to insure against radioactive effluents.

The collection system used in conjunction with the dry box production lines consisted of fiberglass primary filters within each box, a secondary external filter of an activated charcoal bed, and a final fiberglass filter within the central collector. To assure against failure of electrical power required to operate the collector system and subsequent contamination of the area, an auxiliary generator was maintained for emergency use.

The increase in radioactivity of the U-233 material did not present any problems during the first portion of the fabrication process, although, as shown in Figure 10 (curves 1 and 2), a substantial increase in activity was observed in the uranyl nitrate solution from the time of solvent extraction until processing. Curves 3 and 4 of Figure 10 show the increase in activity for the $\text{UO}_2\text{-ThO}_2$ blanket pellets and the $\text{UO}_2\text{-ZrO}_2$ seed pellets, respectively, from the time of compacting until the finished pellets were loaded into the tubes. A delay in sintering of the seed pellets after CO_2 pretreatment was experienced due to limited furnace capacity, and a considerable amount of shielding became necessary around the pellet storage area. The activity was monitored daily, and the shielding (concrete blocks) was increased as required to maintain a minimum of personnel exposure.

A special glove box was designed for tube loading to prevent contamination of the fuel rod exterior surface. The loading box consisted of two individual

chambers, with a machined fixture, shown in Figure 11, extending from the pellet stacking chamber into the clean tube chamber. The tubes were inserted into the loading fixture and clamped tightly against the back edge of the fixture as indicated. The pellet stacks were thus inserted into the tube without abrading against the edge of the tube and with no contamination of the outside tube surface. Loading fixtures were utilized in 0.001-inch ID increments to accommodate the variation in tubing diameter. Lead bricks were employed within the loading box for shielding. During loading of the seed pellets, the activity had increased to such a level that the personnel were rotated every four hours to prevent overexposure.

V. FABRICATION OF FUEL RODS

NUMEC fabricated, inspected, and delivered to Bettis 1299 blanket and 377 seed rods. One end-closure was welded on each tube prior to loading of pellets. This tube assembly was inspected and then shipped to the pellet vendor. Upon the return to NUMEC of the loaded cladding, the second end-closure was completed and the rods were subjected to final inspection. An abridged process outline for the rod processing is provided in Table 11.

The weld design consisted of a recessed endplug which was fitted into the ends of the tube as shown in Section A-A of Figure 12. This creates a lap-joint weld of balanced wall thickness. Welding was performed by the TIG process in a chamber in which the open-end rods were previously evacuated. After evacuation, the chamber was backfilled with the welding gas, the endplug was inserted, and welding commenced. As shown in Figure 12, a group of rods was held in a lazy susan inside the chamber, and lifted individually into a holder for welding. Typical blanket welds are shown as a photograph and a microsection in Figures 13 and 14, respectively.

Major integrity requirements for the fuel rods included:

- (1) Achievement of a weld penetration equivalent to at least 100 percent the supplied tube wall thickness and freedom from other weld defects.
- (2) Helium leak tightness at the standard 1×10^{-7} cc/sec. level.
- (3) Weld area corrosion resistance equivalent to the parent material.
- (4) Assurance that external radioactive contamination was within standard health and safety requirements.
- (5) Dimensions per applicable drawing.

Weld quality of fuel rods was determined radiographically. Both ends of each rod were fitted with steel absorption-compensation pins, encased in a Zircaloy correction block, and exposed in three circumferential positions. This practice was unable to obtain resolution, in all possible instances, of the unwelded interface between the OD of the inserted plug and the ID of the tube wall. The limit for resolution appeared to be fit-ups of plug in tube which resulted in a diametral difference of less than 0.001 inch. Additional assurance of weld penetration was obtained by modifying external visual examination of fuel rods to include comparison with standards which represented the limiting conditions of weld parameters which could be tolerated during end-closure welding.

A helium leak test was applied to both the first end-closure and the finished fuel element. In the former case, the open end of the tube was attached to an evacuation manifold and pumped down to 0.03 microns of helium pressure. Helium was then sprayed around the welded end-closure, and the exhaust was monitored with an ion gage. The finished fuel elements were tested by both immersion in water and evacuation in a helium-monitored chamber after pressurizing to 300 psi in helium.

In lieu of a corrosion test on the rods, a test sample, which consisted of a short length of production tubing joined with two production endcaps, was prepared and tested. The first end-closure on the test sample was made just prior to the start of production welding in a box load of rods, and the second end-closure was made after completion of the last production weld in the box. In this manner, all production welding conditions, particularly the weld chamber atmosphere and also welding speed, were reproduced on the test sample. The corrosion resistance of all samples satisfied standard requirements for appearances.

Shielding and rotation of personnel were required during the manufacture of the rods in order to limit the exposures as required by Code of Federal Regulations 10-CFR-20. Individual workers were limited to exposures of 100 mr per week. Total exposures of the seven men engaged in the receiving-inspection and preparation of fuel rods for welding are shown in Table 12. Exposure of the men engaged in welding and inspection of the finished fuel rods was considerably lower, and well within the requirements of Code of Federal Regulations 10-CFR-20.

All operations involving open-end U²³³-bearing fuel rods were conducted within either a plastic tent under negative pressure or a welding chamber of vacuum quality in order to minimize the possibility of airborne contamination. These operations included opening of the inner shipping container (see discussion under SHIPPING OF FUEL RODS), removal of the plastic shipping cap and plastic shipping bag from each fuel rod, a wipe check for removable contamination, a gamma scan to determine the gross concentration of U-233, determination of the fuel rod weight and end-gap dimension, swab-cleaning of the weld area, placement of a clean plastic cap over the open end, and loading into the lazy-susan weld box fixture (Figure 12). The loaded fixture was then placed into the cylindrical vertical stand of the weld box on blocks which raised the top of the fuel rods to within reach of the operator's glove ports. The box was closed, and the plastic caps removed from the open end of the fuel rods to allow evacuation and back-filling of the rods with helium/argon gas prior to the final end-closure. Air monitors and smears were used to check for contamination external to the tent and weld box. Operator's hands and gloves were also monitored, and no external contamination was incurred.

Gamma radiation from the decay of U-232 was present at the levels shown in Table 11. Personnel working through ports in the receiving-inspection tent were protected from excessive exposure by a 2-inch wall of lead bricks. Shielding for the welding operators was afforded by the 3/8-inch wall of the 12-inch diameter stainless steel pipe which formed the vertical stand of the weld box. This shielding was augmented by wrapping the OD of the pipe with a 1/4-inch lead sheet, and by utilization of leaded glass and leaded gloves in the sight and working ports, respectively.

As-recieved, loaded tubes were contaminated to a level of 50 to 100 cpm, as determined by a wipe-check on the outer surface of plastic shipping bags. One inner container showed a removable contamination level of 90 ppm on receipt, and required decontamination prior to shipment to Bettis. All other containers were within the loose-removable specification limits of Federal Regulations.

An unusual observation of contamination occurred during evacuation of the rods and weld box prior to welding. After vacuum pump-down of the initial lot of rods, smear tests disclosed that the box and its contents were contaminated at a level of 2000 to 3000 cpm. Attempts to reduce this level by lowering the rate of evacuation, and by using glass-wool filters in the open ends of the fuel rods, were unsuccessful. This led to the conclusion that the contamination coming

from the rods was gaseous radon rather than particulate matter. Radon is one of the daughter products of U-232 decay (see Figure 2). Further support of this theory was the diminution of contamination levels observed when the box was allowed to stand idle. The reduction in activity was ascribed to further decay of the radioactive contaminants without replenishment by radon.

Table 13 summarizes the fuel rod losses incurred during manufacture. Rejections in all rod categories were lower than were expected, and only one of the seed rods was rejected. In fact, the only cause for rejection were inclusions in the welds. These were detected primarily by the radiographic examination. Subsequent experience has indicated that this rejection level can be further reduced by modification of electrode design and weld parameters.

VI. SHIPPING OF FUEL RODS

Several factors contributed to the formulation of requirements for shipping fuel rods. Among these were nuclear health and safety considerations, the geographical separation between the principal vendors and Bettis, the radioactive behavior of U²³³-bearing fuels, and the resultant scheduled manufacturing sequence. The legal movement of radioactive materials between licensed users is subject to regulation by local, state, and federal governmental agencies. These regulations are designed to prevent dangerous criticality incidents, harmful exposure of humans, animals, or foodstuffs to radioactivity, and radioactive contamination of the surroundings while in transit.

The geographic separation of the manufacturers of the pellets, and fuel elements, and the Bettis plant required the movement of fuel materials from NFS to NUMEC and from NUMEC to Bettis. The production schedule for the program was keyed to completion and delivery to Bettis of the U²³³-bearing fuel elements within 90 days following purification by solvent extraction. These considerations resulted in the following ground rules for shipping containers:

- (1) Since the requirements include control of both criticality and total activity, the design selected represents a compromise of mass and geometry control, and sets the quantity of fuel rods contained in the individual units. For these reasons, the container manufacturer was requested to specify the number of containers required to ship the fuel rods.
- (2) The anticipated manufacturing schedule precluded the reuse of shipping containers for the three-way shuttle service between plants.

- (3) In the interest of reducing the handling, packing, and re-inspection of pellets manufactured by NFS, these pellets were loaded in cladding tubes supplied by NUMEC with one end-closure affixed. The open end of the fuel-loaded tube was capped with plastic and sealed with plastic tape when returned to NUMEC for final fuel rod end closure and testing. This arrangement required shipment of fuel-loaded tubes in the vertical position only, with adequate hold-down and lateral support to preclude spillage of, or damage to, the pellets in transit.
- (4) In the interest of satisfying requirements for both storage during the Bettis program, and return of the U-233 materials afterward, a requirement was added to allow addition of shielding materials to the U²³³-bearing containers. This assured adherence to the regulations for external activity at the maximum fuel activity anticipated up to approximately 30 months after purification.
- (5) To assure that the containers would be free from either effluence of radioactive materials from the container (especially during the one-way shipment of open-end fuel rod assemblies) and the influx of contaminants into the container in transit, a hermetic seal was required in each container.

NUMEC was responsible for design and fabrication of 18 shipping containers. Two separate container designs, of the same basic geometrical configuration, were submitted for handling the two fuel rod types. Rod support in each design is illustrated in Figure 15 and consists of an upper and lower aluminum grid plate, separated by threaded rods, and confined between steel compression plates. The pressure on the top and bottom of the fuel rods is transmitted from the compression plates through the rubber pads as shown. This grid assembly slips inside a welded steel right-circular cylinder, which is provided with a fire-proof hermetic seal by compression of a stainless steel O-ring in a machined circular groove between a flange welded to the top of the cylinder OD and a coverplate bolted to the flange. The containers controlled criticality by spacing fuel rod openings within the grid plates, and by the "birdcage" support of the containers within the drum and, in part, by the thickness of the steel in the inner and outer cans. The seed containers hold 54 rods, and 144 rods fit in the blanket containers.

A lead shield was added between the inner and outer cans. Additional gamma shielding may be added by filling the annulus between the outer container and the steel drum with concrete. This option was exercised on two blanket and two seed containers in which case absorber-bearing concrete was used. Addition of this 2000 psi, minimum compressional-strength concrete, which contained magnetite replacement of the sand and pig iron replacement of the aggregate per Bureau of Explosives requirements, assured that the activity levels anticipated for the fuel rods 30 months after purification could be attenuated to acceptable levels at the outside of the drum. When the concrete shielding was added, the gross weight of the containers exceeded the BE regulations for distribution of the mass over the supporting deck areas of a carrier. This condition required the addition of skids to the containers, as illustrated in Figure 15.

Since removability of the inner can from the drum for vault storage or confined-area handling was a design requirement, top shielding was provided by thicker steel in the coverplates. Shielding requirements in this direction were reduced by the presence of more than two inches of Zircaloy spacers between the ends of the fuel rod and the start of the fuel, as illustrated in Figure 1.

In addition to the containers, NUMEC supplied disposable plastic caps and plastic insert plugs used in the one-way shipment of open-end fuel element assemblies from NFS. The insert plug provided a length interference with the fuel column plus Zircaloy spacer and was restrained by the compressional interference of the plastic cap. This outer cap extended down the OD of the tube for approximately 1/2 inch. This cap was taped to assure freedom from radioactive contamination of the container interior and of the fuel rod exterior by leakage through the unwelded end.

Activity levels of the shipments are summarized in Table 14. Shipment of the blanket rods to Bettis was completed 95 days after solvent extraction of the U-233. Shipment of the seed rods was completed 83 days after solvent extraction.

VII. CONCLUSIONS

Ceramic pellets containing up to 26 w/o $U^{233}O_2$, with 38 ppm of U-232, were successfully fabricated and loaded into Zircaloy cladding within 95 days after batch purification of uranyl nitrate. On a full-production basis, the outputs of solvent extraction, powder and pellet production, and fuel rod operations would be integrated at a common capacity to minimize time of buildup of radioactivity following purification of uranyl nitrate. On this basis, there is no reason to believe that actual core fuels could not be recycled and fabricated

in pellet form. Since these fuels would have considerably higher levels of U-232 than the 38 ppm of this program, it would be necessary to provide additional shielding and to modify certain procedures for remote operation. In no case does this modification appear to be an insurmountable task. Additional advantages can be foreseen, in modification of the techniques used in the program, by designing the fuel rod operations on an assembly-line basis. If welding and radiography, for example, were performed on single rods, there is a major reduction of operator exposure attendant to the reduction of mass.

The material balance for U-233 revealed that a total of 4.71 percent must be considered in the category as irrecoverable, questionably recoverable (largest portion), or unidentified in accountability. Additional work is planned in this area to make a realistic determination of irrecoverable loss to be expected in large production runs.

ACKNOWLEDGMENT

In a program of this magnitude, personnel from many disciplines in the Bettis organization were required to join in the effort. The authors are indebted to each of these persons who are obviously too numerous to list. Additional acknowledgment of efforts of those participants external to Bettis is also mentioned with a feeling of gratitude and satisfaction. The program was completed only with major assistance from Wolverine Tube Division, Nuclear Fuel Services, Nuclear Materials and Equipment Corporation, and Oak Ridge National Laboratory.

TABLE 1. INSPECTION OF CLADDING TUBES*

1. Ultrasonic Test - All tubes were tested against artificial longitudinal and transverse defect standards of 0.002-inch depth x 0.030-inch length for seed size and 0.003-inch depth x 0.062-inch length for blanket size.
2. Internal Pressure Test - The seed-size tubes were tested hydrostatically at 5920 psi for 1 minute and the blanket tubes were tested at 5325 psi. Both types of tubing were also air tested at 100 psi for 1 minute.

3. Room-Temperature Tensile Properties

	Ultimate Strength (psi)	0.2% Offset Yield Strength (psi)	% Elongation over 2 in. Gage Length
Blanket Size			
Min.	68,600	47,000	22
Max.	90,400	67,000	35
\bar{X}	76,300	56,200	26.8
Seed Size			
Min.	59,100	40,800	20
Max.	78,400	63,800	29.5
\bar{X}	67,900	50,500	24.6

4. Surface Finish (AA) (OD and ID)

	Seed Size	Blanket Size
Min.	8	17
Max.	27	31
\bar{X}	18.5	25

5. Chemical Analyses - The tubing satisfied the conventional requirements for Zircaloy-2. The following analyses were reported on final product.

	Seed Size (ppm)			Blanket Size (ppm)		
	Min.	Max.	\bar{X}	Min.	Max.	\bar{X}
H ₂	2	20	7.0	2	20	7.8
N	22	52	32.7	21	65	35.9

Oxygen analyses in the original ingots ranged from 670 to 1390 ppm.

6. Corrosion Properties (Weight Gains in mg/dm²)

	Seed Size			Blanket Size		
	Min.	Max.	\bar{X}	Min.	Max.	\bar{X}
14 days, 680°F water	13	21.2	17.1	14.2	19	17.0
14 days, 750°F steam	20.5	36	27.2	22	30	28.6

7. Grain Size (ASTM)

Seed Size	Blanket Size
8.5 - 11.5	10 - 11.5

*This summary provides only the highlights.

TABLE 2. RESULTS OF CHEMICAL ANALYSIS FOR IMPURITIES

Element	Original Analyses of Liquid Nitrate*		Recent Bettis Analysis of UO ₂ Powder† (ppm)
	Batch No. 1 (ppm)	Batch No. 2 (ppm)	
Ag	<0.6	<0.6	-
Al	23	16	37
As	<62	<62	0.04
Au	<12	<12	-
B	<6	<6	6
Ba	<125	<124	3
Be	<0.01	<0.01	-
Bi	<31	<31	-
Ca	51	31	32 ‡
Cd	<12	<12	-
Ce	<16	<16	-
Cl	-	-	4
Co	<12	<12	122 ‡
Cr	6.3	5.4	141 ‡
Cs	-	<15	-
Cu	1.9	1.5	-
Dy	<15	<16	-
Er	<62	<62	-
Eu	<3	<3	-
F	-	-	0.1
Fe	22	16	1090 ‡
Ga	<5	<5	0.2
Gd	<3	<3	-
Ge	<1	<1	-
Hf	<6	<6	-
Hg	<125	<124	37
Ho	<31	<31	-
In	<9	<9	-
Ir	<62	<62	-
K	<62	<62	15 ‡
La	<12	<12	-
Li	12	<12	-
Lu	12	<12	-
Mg	12	4	20 ‡
Mn	<0.6	<0.6	-
Mo	<7	<7	9
N	-	-	43
Na	89	40	4
Nb	<16	<16	0.6
Nd	<78	<78	-

TABLE 2 (Cont)

Element	Original Analyses of Liquid Nitrate*		Recent Bettis Analysis of UO ₂ Powder† (ppm)
	Batch No. 1 (ppm)	Batch No. 2 (ppm)	
Ni	1.6	1.5	262 ‡
Os	<31	<31	-
P	<156	<155	6
Pb	<12	<12	0.1
Pd	<6	<6	-
Pr	<31	<31	-
Pt	<12	<12	-
Pu	-	-	15
Rb	<62	<31	0.1
Re	<6	<6	-
Rh	<12	<12	-
Ru	<6	<6	-
S	-	-	7
Sb	<6	<6	-
Sc	<3	<3	-
Si	1	<1	428 ‡
Sm	<6	<6	-
Sn	<12	<12	-
Sr	<6	<6	0.05
Ta	<31	<31	-
Tb	<31	<31	-
Tc	<3	<3	-
Ti	-	-	23 ‡
Tl	<16	<16	-
Tm	<6	<6	-
V	<1.2	<1.2	0.7
W	<16	<16	33
Y	<1	<1	-
Yb	<3	<3	-
Zn	-	-	0.04
Zr	-	-	27

*Emission spectrometer

†Spark-source mass spectrometer. Elements for which no number is reported do not exceed 0.1 ppm, if present, except for C and O which were not checked.

‡One spectrum indicated Mg 100 times this level. Ni, Co, Fe, Cr, Ti, Ca, K, and Si are also segregated; however, averages appear to be representative of the sample.

TABLE 3. REPORTED CHEMISTRY FOR THE SHIPMENTS OF URNAYL (U-233) NITRATE
AND BETTIS RECHECK OF U AND Th ISOTOPIC ANALYSES

Item	Original Analyses of Liquid Nitrate		Recent Bettis Analysis of UO ₂ Powder*
	Batch No. 1	Batch No. 2	
Total Volume	54.115 Liters	48.406 Liters	-
Solution Analysis			
U-Total	131.94 mg/ml UNH	128.18 mg/ml UNH	-
U-233	128.43 mg/ml UNH	124.76 mg/ml UNH	-
Th	0.86 mg/ml UNH	1.13 mg/ml UNH	1.43 w/o
	or 0.67 w/o (U-223)	or 0.89 w/o (U-233)	
HNO ₃	0.0375 molar	0.0365 molar	-
Weight			
U-Total	7,140 grams	6,205 grams	-
U-233	6,950 grams	6,039 grams	-
Uranium Isotopic			
U-232	0.05 a/o (ORNL)	0.05 a/o (ORNL)	-
	38 ppm (U-233)*	38 ppm (U-233)*	39 ppm** (+4)
U-233	97.24 w/o	97.22 w/o	97.20 w/o (±0.04)
U-234	1.53 w/o	1.56 w/o	1.56 w/o (±0.02)
U-235	0.05 w/o	0.05 w/o	0.06 w/o (±0.01)
U-236	0.05 w/o	0.05 w/o	0.014 w/o (±0.005)
U-238	1.12 w/o	1.12 w/o	1.16 w/o (±0.02)
Thorium Isotopic			
Th-228	0.02 a/o†	0.03 a/o†	-
Th-229	0.02 a/o	0.03 a/o	-
Th-230	0.02 a/o	0.03 a/o	-
Th-231	0.02 a/o	0.03 a/o	-
Th-232	100. a/o	100. a/o	-
Th-233	Masked by U-233	Masked by U-233	-
Th-234	Masked by Th-232	Masked by Th-232	-
Gamma Activity			
1 day after purification	1.2 x 10 ³ c/m/mg	7.24 x 10 ² c/m/mg	-
7 days after purification	1.5 x 10 ³ c/m/mg	1.28 x 10 ³ c/m/mg	-

*Based on U-233 and on previous irradiation history as well as activity spectrum of the uranium with respect to time.

†No difference is intended between the two batches. This is merely the limit of detection for the two analyses.

*Spark-source mass spectrometer.

**Determined by counting technique.

TABLE 4. ANALYSES OF ThO₂ POWDER AND
1 w/o U²³³O₂-ThO₂ PELLETT

	Bettis Analyses of ThO ₂ Powder*	Recent Bettis Analyses of Pellet†
Al	<50	40
As	-	0.04
B	1	0.1
Ba	-	3
Be	-	1
C	186	-
Ca	<50	54
Cd	<0.5	-
Ce	-	29
Cl	10	0.8
Co	<5	7
Cr	<30	57
Cu	<20	0.06
Dy	2.2	6
Er	-	2
F	<10	0.07
Fe	95	183
Ga	-	0.6
Gd	2.9, 2.9	5
Ge	-	0.2
Hf	<35	-
Hg	-	0.3
Ho	-	1
K	<25	0.3
La	-	12
Li	-	0.007
Mg	<50	64
Mn	<5	0.05
Mo	<25	2
N	<50	11
Na	<50	0.1
Nb	-	0.8
Nd	(D)	12
Ni	75	278
P	-	1
Pb	<5	0.5
Pr	-	3
Pu	-	0.2
S	-	2
Sc	-	0.3

TABLE 6. SUMMARY OF U-233 PELLET FABRICATION

1. Weigh $U^{233}O_2$ and ThO_2 or ZrO_2 powders.
2. Load into blender, blend for 1 hour (2-ft³ blender for UO_2-Th_2 , 8-qt blender for UO_2-ZrO_2).
3. Discharge blends into doubled, tare-weighed polyethylene bags. Seal bags with tape and identify.
4. Load one blend into ball jar and add appropriate weight of cylindrical borundum balls (5-gal. jar for UO_2-ZrO_2 blend). Add 700 ml of acetone to each UO_2-ZrO_2 blend and seal jar.
5. Transfer to ball-mill racks and mill for 4 hours.
6. Dry each ball-milled UO_2-ZrO_2 blend at 100°F (38°C) for 2 hours.
7. Transfer ball-milled batch to Hobart planetary mixer.
8. Agglomerate powder by adding a previously-prepared solution of PVA (polyvinyl alcohol) whiler mixer is running slowly. Total addition of PVA is equivalent to 1.5 w/o of powder.
9. Transfer agglomerated batch to Stokes granulator and granulate through an 18-mesh screen.
10. Spread uniformly in 3/4-inch thick layers on stainless-steel trays.
11. Vacuum dry at 100°F for 6 hours with a vacuum of 30 inches of Hg.
12. Transfer dry granules to Stokes granulator and granulate through a 20-mesh screen.
13. Transfer three batches of UO_2-ThO_2 granules to the 2-ft³ twin shell blender, and one batch of UO_2-ZrO_2 granules to the 8-qt twin shell blender.
14. Add 0.4 w/o of -200 mesh Sterotex and blend for 10 minutes.
15. Package each blend in a polyethylene-lined can (5 gal. for UO_2-ThO_2 , 1 gal. for UO_2-ZrO_2) and transfer to press.
16. Compact into pellets, checking green density of every 12th pellet.
 UO_2-ThO_2 : 6.45 to 6.55 g/cc
 UO_2-ZrO_2 : 3.95 to 4.05 g/cc
17. Load acceptable pellets in a single layer on stainless steel trays. Transfer to furnace for binder renewal.

TABLE 4 (Cont)

	Bettis Analyses of ThO ₂ Powder*	Recent Bettis Analyses of Pellet†
Se	-	0.08
Si	45	188
Sm	-	4
Sn	<5	1
Sr	-	0.3
Tb	<5	1
Ti	<5	97
U	<1	-
V	-	0.5
W	-	11
Y	(D)	49
Zr	-	21

(D) = Detected only

* = Emission spectrograph.

† = Spark-source mass spectrometer. Elements for which no number is reported do not exceed 0.1 ppm, if present, except for C and O which were not checked.

TABLE 5. SUMMARY OF U²³³O₂ POWDER PRODUCTION

1. Transfer as-received uranyl nitrate solution from storage tank to feed vessel and subsequently to the boil-down evaporator.
2. Evaporate until concentration is 250 grams of U-233/liter.
3. Transfer to precipitation station.
4. Precipitate to form ammonium di-urate (ADU) as follows:
 - (a) All material for UO₂-ThO₂ and 1/2 of material for UO₂-ZrO₂ composition, precipitate with anhydrous ammonia.
 - (b) 1/2 of material for UO₂-ZrO₂ composition, precipitate with liquid ammonia.
5. Dry at 250°F for 4 hours.
6. Transfer to hydrogen reduction furnace.
7. Reduce to UO₂ at 1500°F.
8. Screen through a 20-mesh sieve.
9. Blend and cross-blend into two uniform lots; one for UO₂-ThO₂, and one for UO₂-ZrO₂ compositions.

TABLE 6 (Cont)

18. Load trays into furnace muffle for binder removal. Insert muffle into furnace at established rate of 19 inches per hour. Temperature at center of furnace will be held at 1250°F (675°C). Total cycle requires 11 hours.
19. Remove muffle from furnace; transfer pellets to sintering station.
20. Load pellets into molybdenum boats, two layers deep for $\text{UO}_2\text{-ThO}_2$ and a single layer for $\text{UO}_2\text{-ZrO}_2$.
21. Sinter in hydrogen furnace:

$\text{UO}_2\text{-ThO}_2$: 3125°F (1720°C) for approximately 12 hours at temperature.

$\text{UO}_2\text{-ZrO}_2$: 3200°F (1760°C) for approximately 48 hours at temperature.
22. Remove trays from furnace and transfer to inspection station.
23. Perform 100 percent inspection for physical defects and diametrical tolerances (go, no-go gage).
24. Centerless grind OD surface if necessary.
25. Dry at 40°C for 2 hours and with a vacuum of 30 inches of Hg.
26. Assemble entire batch for sampling.
27. Randomly select sample pellets from each batch:

	<u>$\text{UO}_2\text{-ThO}_2$</u>	<u>$\text{UO}_2\text{-ZrO}_2$</u>	
(a)	20	10	pellets for density measurement
(b)	2	2	pellets for analysis by ORNL
(c)	2	2	pellets for metallography
(d)	5	5	pellets for retainer sample
28. Assemble pellets into stacks 15.000 ± 0.060 inches in length. No pellets shall be cut to less than 1/2 the diameter, and cut pellets shall be at least four pellet lengths from the end.
29. Weigh metallic tube components and fuel stack separately to nearest 0.1 gram and record.
30. Check tube height and end-clearance gap, adjust stack height if necessary.
31. Load pellet stack into tube and seal with plastic cap.
32. Check-weigh entire tube assembly to nearest 0.1 gram and verify with weights in 29, above.

TABLE 6 (Cont)

33. Monitor each tube and verify that radiation levels do not exceed following limits:

(a) Loose removable contamination - 25 dpm/tube

(b) Fixed contamination - 50 dpm/61 cm²

34. Load into shipping container, ship to NUMEC for second end welding.

TABLE 7. U²³³O₂-ThO₂ MEASUREMENTS OF DENSITY OF PRODUCTION BLANKET PELLETS

<u>Batch No.</u>	<u>Avg. Density (20 Samples)*</u>	<u>Minimum Individual Pellet*</u>	<u>Maximum Individual Pellet*</u>
01	9.57	9.44	9.68
02	9.37	9.23	9.46
03	9.36	9.24	9.50
04	9.50	9.27	9.61
05	9.55	9.47	9.63
06	9.59	9.49	9.72†
07	9.53	9.26	9.72
08	9.42	9.27	9.57
09	9.53	9.39	9.62
10	9.43	9.27	9.60
11	9.40	9.25	9.56
12	9.41	9.18	9.58
13	9.44	9.32	9.60
14	9.57	9.40	9.71
15	9.46	9.29	9.60
16	9.40	9.24	9.59
17	9.35	9.22	9.56
18	9.36	9.24	9.50
19	9.37	9.20	9.49
20	9.41	9.29	9.53
21	9.49	9.34	9.60
22	9.37	9.09†	9.60
23	9.44	9.33	9.60
24	9.36	9.22	9.44
25	9.43	9.28	9.61

Overall Average = 9.445 g/cc
= 94.07% TD

*Values given in g/cc

†Minimum individual = 9.09 g/cc = 90.53%

#Maximum individual = 9.75 g/cc = 96.81% TD

TABLE 8. $U^{233}O_2-ZrO_2$ MEASUREMENTS OF DENSITY OF PRODUCTION SEED PELLETS

<u>Batch No.</u>	<u>Avg. Density (10 Samples)*</u>	<u>Minimum Individual Measurement*</u>	<u>Maximum Individual Measurement*</u>
01	6.46	6.41	6.55
02	6.43	6.38	6.50
03	6.29	6.20	6.34
04	6.33	6.23	6.40
05	6.46	6.28	6.59
06	6.53	6.29	6.65 ‡
07	6.62	6.60	6.64
08	6.58	6.53	6.60
09	6.57	6.46	6.63
10	6.33	6.24	6.40
11	6.29	6.14†	6.43
12	6.36	6.26	6.49
13	6.39	6.36	6.46
14	6.43	6.35	6.56
15	6.57	6.45	6.60
16	6.50	6.29	6.63
17	6.49	6.32	6.59
18	6.59	7.58	6.61
19	6.59	6.55	6.62
20	6.57	6.51	6.61
21	6.59	6.49	6.62
22	6.59	6.57	6.63
23	6.58	6.47	6.62

Overall Average - 6.484 g/cc = 93.42% TD=

*Values given in g/cc

†Minimum individual = 6.14 g/cc = 88.47% TD

‡Maximum individual = 6.65 g/cc = 95.82% TD

TABLE 9. SUMMARY OF NFS U-233 MATERIAL BALANCES

<u>Category</u>	<u>% of Input</u>	
A. <u>Usable Materials</u>		<u>91.22</u>
1. Pellets shipped to Bettis in fuel rods	86.11	
2. Residues in usable powder form	4.97	
3. Retainer samples	0.14	
B. <u>Recyclable Process Scrap</u>		<u>3.17</u>
1. Reject Pellets	2.61	
2. Reject Powder	0.56	
C. <u>Recoverable Scrap</u>		<u>0.90</u>
1. Grinder Sludge	0.90	
D. <u>Buried Scrap Residues (Estimate)</u>		<u>3.87</u>
1. Miscellaneous minor equipment, SS granulation screens, cans, filters, and combustible wastes	3.87	
E. <u>Scrap Liquors</u>		<u>0.30</u>
1. Condensate, weak nitric acid, wash liquors, and ammoniacal filtrate	0.30	
F. <u>Residues Remaining in Capital Equipment (Estimate)</u>		<u>0.04</u>
G. <u>Material Unaccounted For (Estimate)</u>	0.50	<u>0.50</u>
		100.00

TABLE 10. URANIUM ANALYSES OF PREPRODUCTION (NFS) AND PRODUCTION PELLETS

I. <u>Depleted (Preproduction)</u>	<u>Avg. w/o U</u>	<u>No. Analyses</u>
A. <u>1 w/o UO₂-ThO₂</u>		
1. NFS		
2. Bettis (1)	0.925	60
(2)	0.895	21
	0.912	21 (recheck)
3. ORNL	0.932	4
B. <u>26.8 w/o UO₂-ZrO₂</u>		
1. NFS	23.40	63
2. Bettis	23.70	21
3. ORNL	23.67	4
II. <u>U-233 (Production)</u>		
A. 1 w/o UO ₂ -ThO ₂ - ORNL	0.939* (w/o U-233 - 0.913)	52
B. <u>26.8 w/o UO₂-ZrO₂ - ORNL</u>	24.47* (w/o U-233 - 23.81)	46
III. <u>Isotopic Analysis (ORNL)</u>	<u>UO₂-ThO₂</u> <u>(26 Analyses)</u>	<u>UO₂-ZrO₂</u> <u>(23 Analyses)</u>
U-232	<0.05	<0.05
U-233	97.19	97.29
U-234	1.55	1.56
U-235	<0.05	<0.05
U-236	<0.05	<0.05
U-238	1.23	1.14

*Corrected to U-233 - $\frac{(233)}{(238)}$

TABLE 11. ABRIDGED PROCESS OUTLINE FOR PROCESSING OF FUEL RODS

1. Receiving inspect cut-to-length tubes and rod stock.
2. Machine endplugs and inspect.
3. Clean tubes and endplugs in hot sodium carbonate, tri-sodium phosphate solution. Rinse and air dry.
4. Insert first endplug flush with tube end.
5. Insert in weld box; evacuate to 0.03 micron; back-fill with argon - 10 v/o helium to atmosphere pressure.
6. TIG-weld with high frequency arc initiation 0.040-inch diameter thoriated tungsten electrode programmed weld cycle with up-and-down slope.

Electrode Diameter (inch)	Seed	Blanket
	0.040	0.040
Arc Gap (inch)	0.030 - 0.050	0.030 - 0.050
Amperes	12	25
Rotational Speed (rpm)	4	4

7. Dimensional check and identify with vibratory scriber.
8. Leak test on Manifold.
9. Radiograph for penetration and other weld defects.
10. Seal in plastic bag and pack fuel tube assemblies for shipment. Maintain lot identity.
11. Ship fuel tube assemblies to fuel pellet manufacturer.
12. Receive loaded fuel element assemblies from fuel pellet manufacturer.
13. Receiving inspect visually for damage, gamma scan, weigh, and measure end clearance.
14. Clean ID of tube wall at open end with both alcohol-moistened and dry swabs.
15. Insert second endplug flush with tube end.
16. Weld per 6, above.
17. Reweigh and record.
18. Leak test to standard 1×10^{-7} cc/sec. helium.
19. Radiograph second end-closures.

TABLE 11 (Cont)

20. Dimensional and visual inspect.
21. Check for fixed and removable contamination.
22. Alcohol wipe.
23. Pack and ship completed fuel rods to Bettis.

TABLE 12. EXPOSURE OF PERSONNEL DURING FUEL ROD MANUFACTURE

A. Dose (mr) Received by NUMEC Personnel during Manufacture of Fuel Rods*

<u>Man No.</u>	<u>Total Exposure (mr)</u>
1	540
2	570
3	130
4	430
5	420
6	350
7	130

B. Gamma Activity Levels, Fuel Rods

<u>Location</u>	<u>Unloading Single Rods (Approx. 14 g U-233)</u>	<u>54 Rods in Welding Fixture (Approx. 2240 g U-233)</u>
1. <u>Seed</u>		
(a) At Contact	80 mr/hr	2000 mr/hr
(b) At One Foot	15 mr/hr	300 mr/hr
2. <u>Blanket</u>		
(a) At Contact	30 mr/hr	850 mr/hr
(b) At One Foot	1 mr/hr	25 mr/hr

*The seven personnel listed were rotated in duties connected with receiving inspection, preparation for welding, and handling. Inspection of the finished fuel rods was performed on restricted rod groupings by pre-planned moves which limited close-range exposure to the rods and held cumulative exposures to less than 100 mr during the period of manufacture.

TABLE 13. FUEL ROD YIELDS*

<u>First Ends</u>	<u>Blanket</u>	<u>Seed</u>
Start	1412	414
Accept	<u>1409</u>	<u>413</u>
Reject	3 (for inclusions)	1 (for inclusions)
<u>Second Ends</u>		
Start	1329	395
Accept	<u>1319</u>	<u>395</u>
Reject	10 (for inclusions)	0

*The fuel in the rejected rods was successfully recovered as acceptable pellets.

TABLE 14. ACTIVITY LEVELS OF FUEL ROD SHIPPING CONTAINERS

1. Average Activity of Containers Received from NFS at NUMEC
 - (a) On contact - 17 mr/hr
 - (b) At one meter - 2 mr/hr
2. Average Activity of Containers Received from NUMEC at Bettis
 - (a) On contact - Seed - 45 mr/hr
 Blanket - 12 mr/hr
 - (b) At one meter - No data (See Note)

NOTE: Bettis regulations require readings of less than 10 mr/hr at one meter. Acceptance of the shipments at Bettis indicates that this requirement was met.

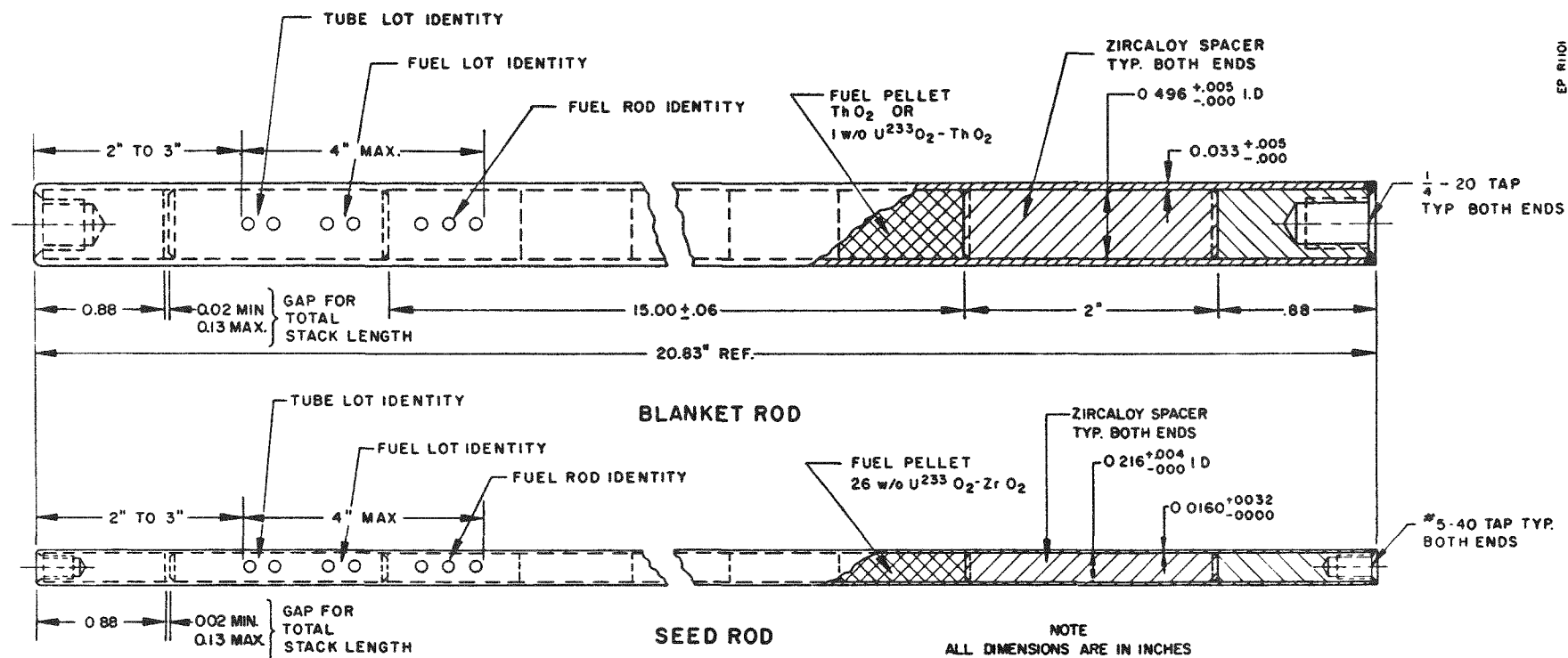


Figure 1. Sketches of Blanket- and Seed-Type Fuel Rods for U-233 Program.

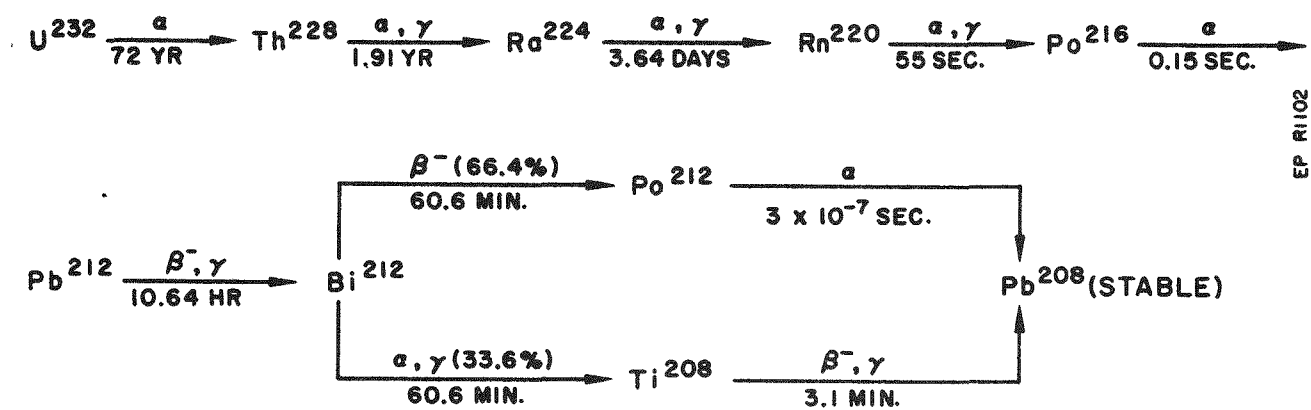


Figure 2. Natural Decay of U-232 in U-233 Fuel.

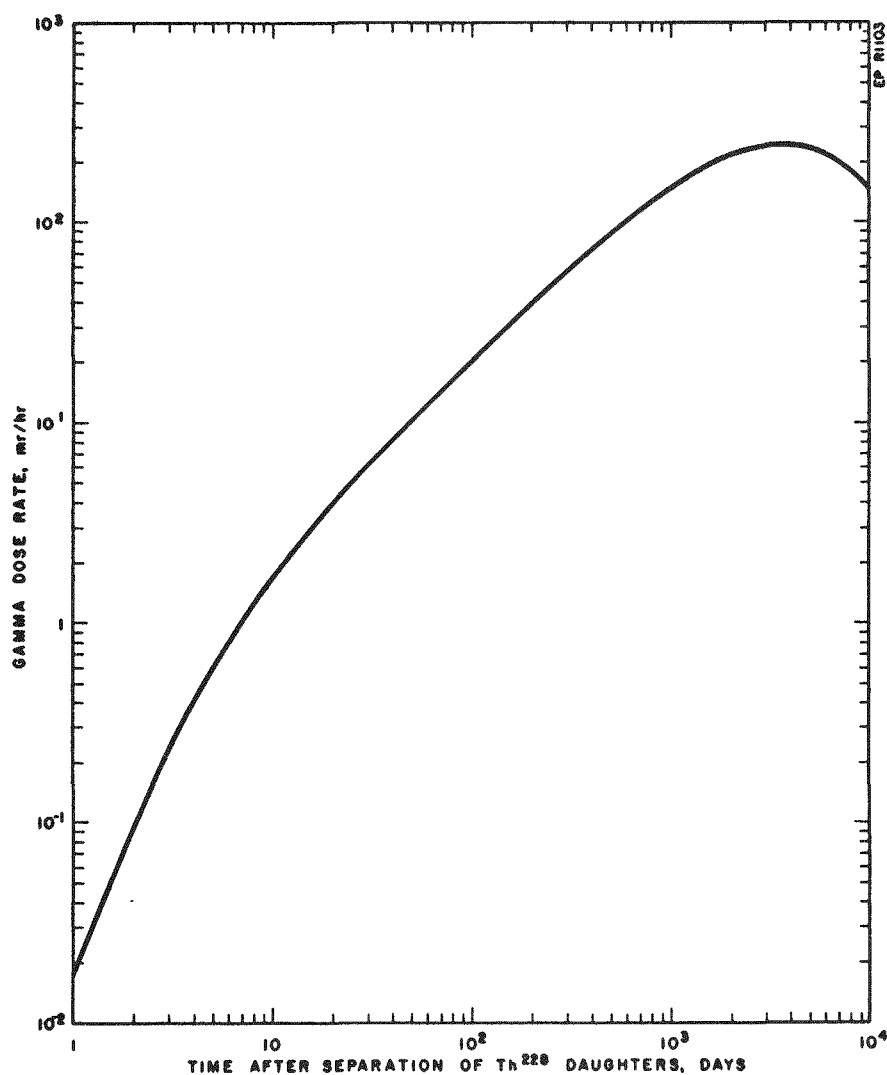
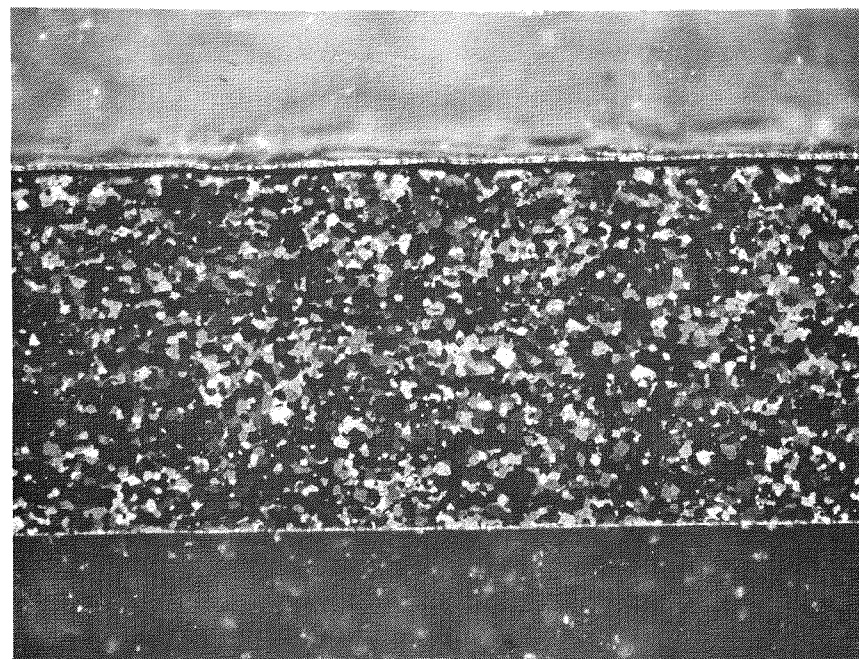


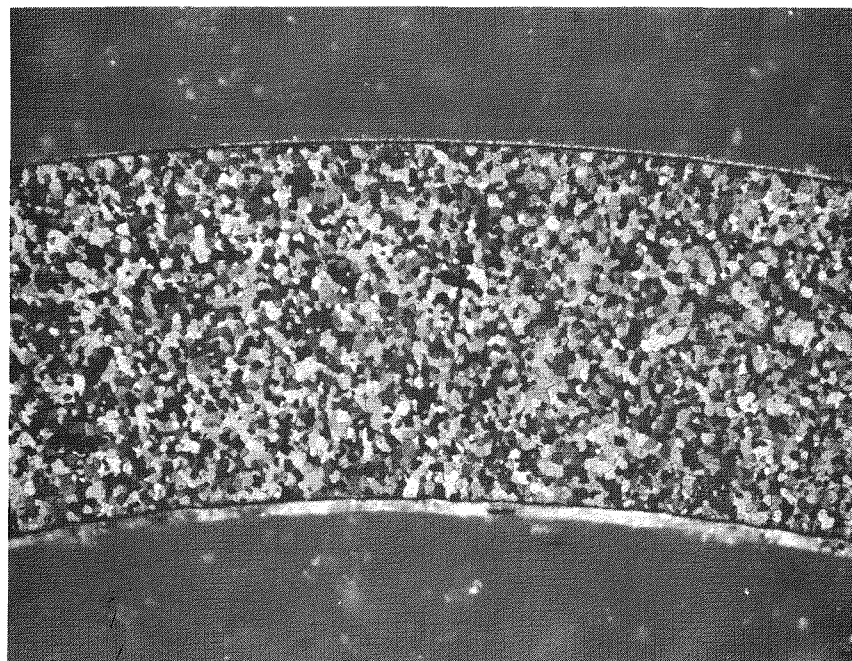
Figure 3. Buildup of Radioactivity in U-233 Fuel.

EP RI104



POLARIZED LIGHT

LONGITUDINAL
100X



POLARIZED LIGHT

TRANSVERSE
100X

Figure 4. Typical Microstructure of Seed-Size Tubing.

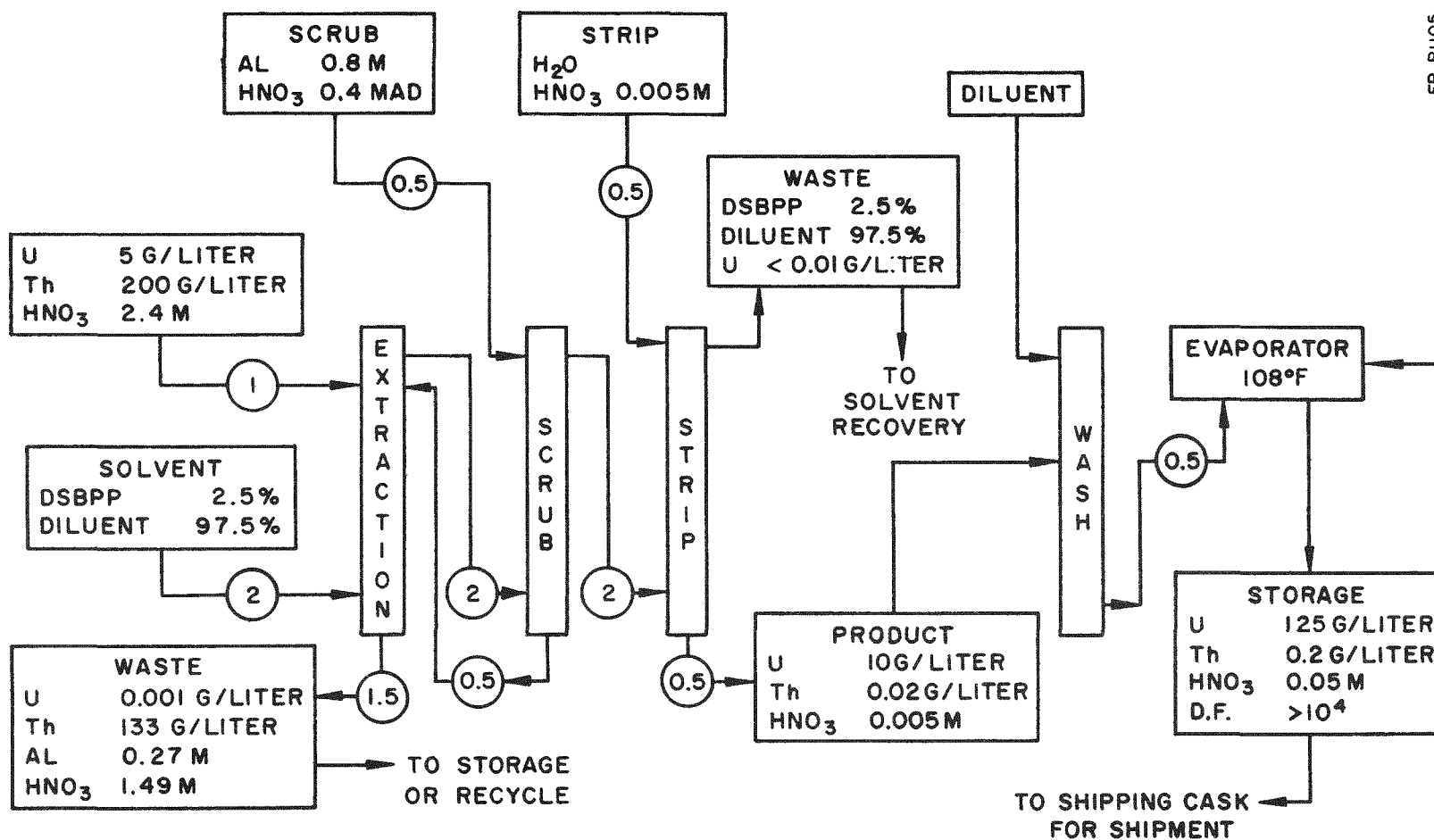


Figure 5. Compositions of Solutions Introduced and Removed during Solvent Extraction. (Courtesy of ORNL)

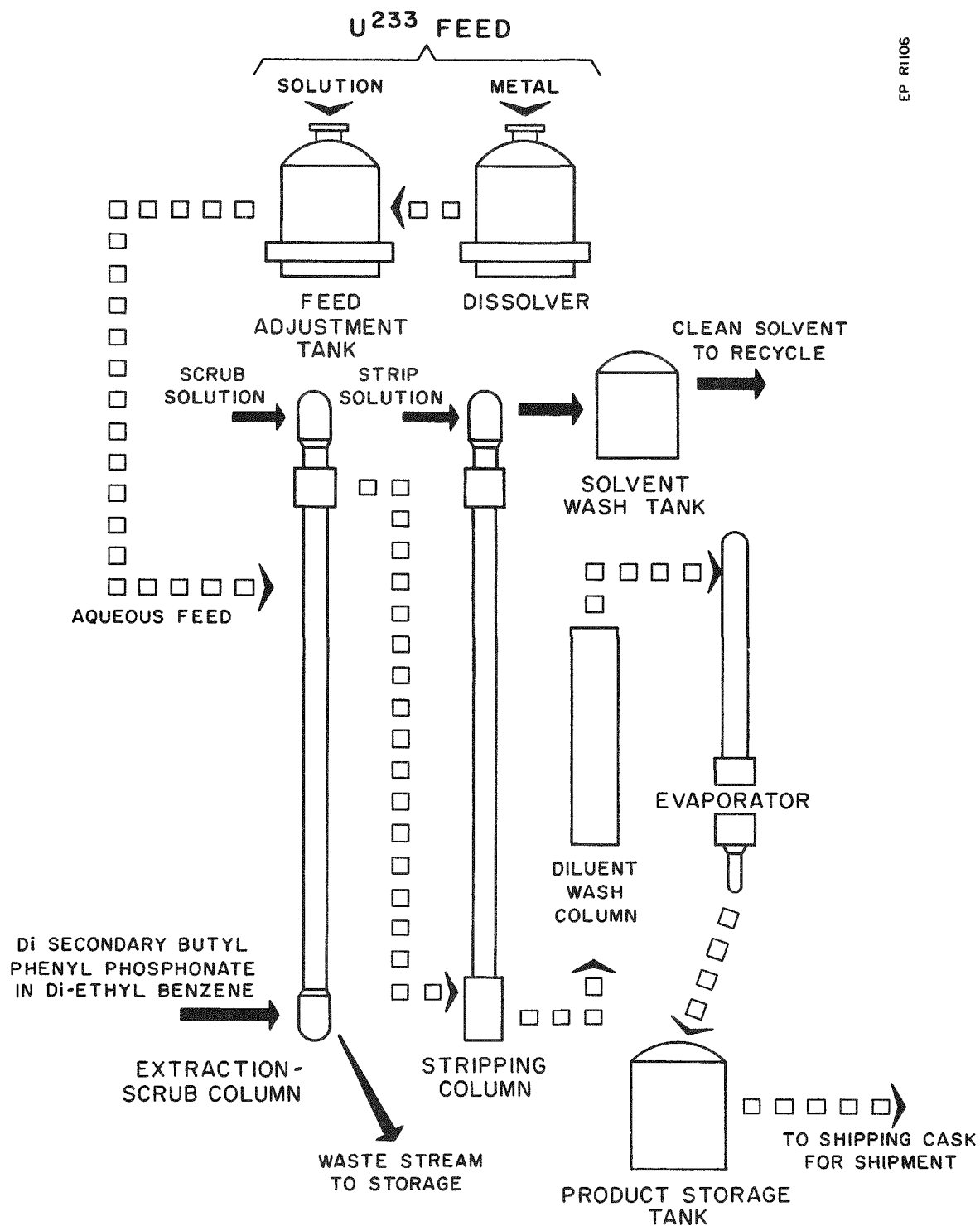


Figure 6. Flow Sheet for Solvent Extraction of Uranyl Nitrate.
(Courtesy of ORNL)

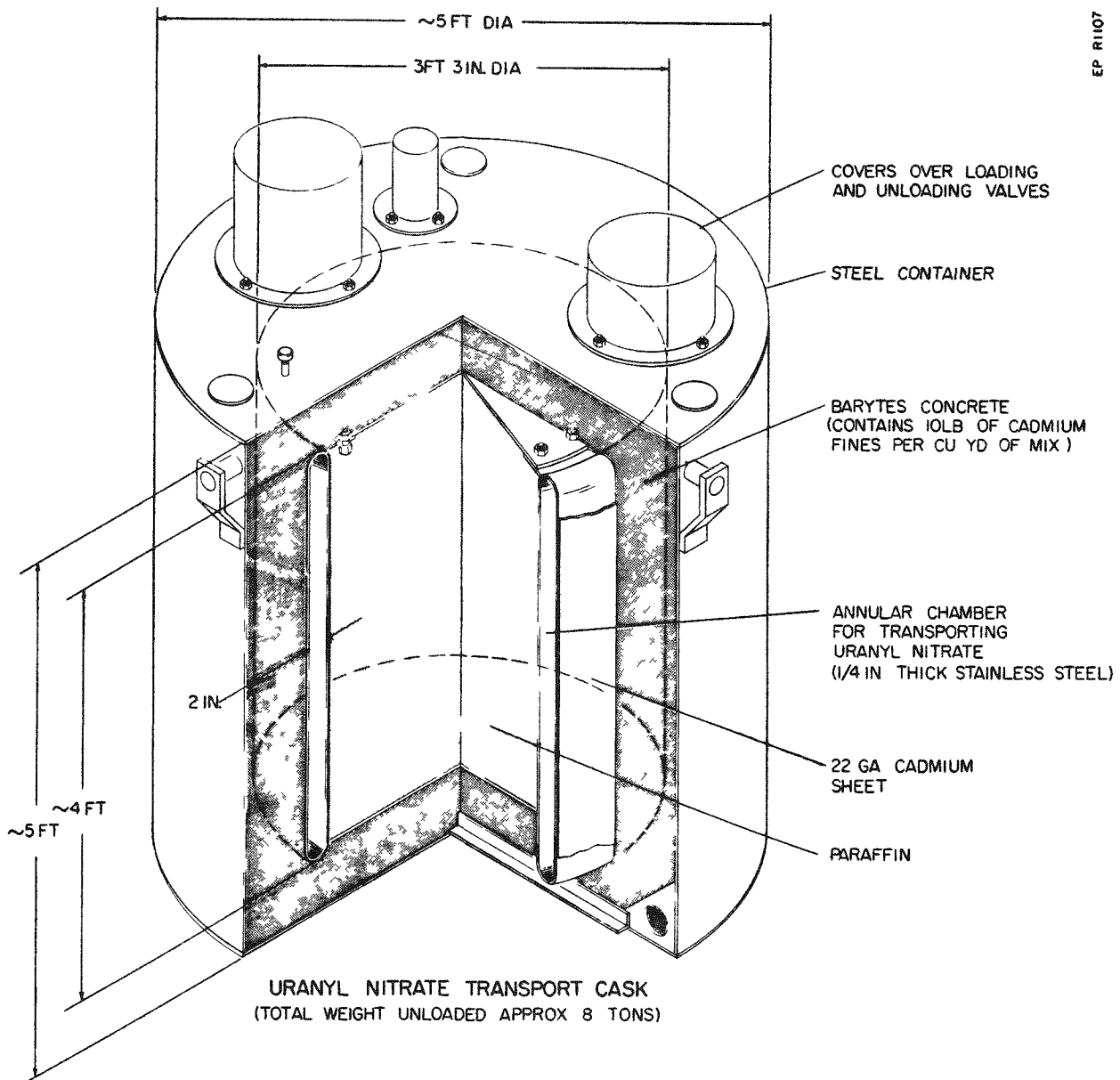
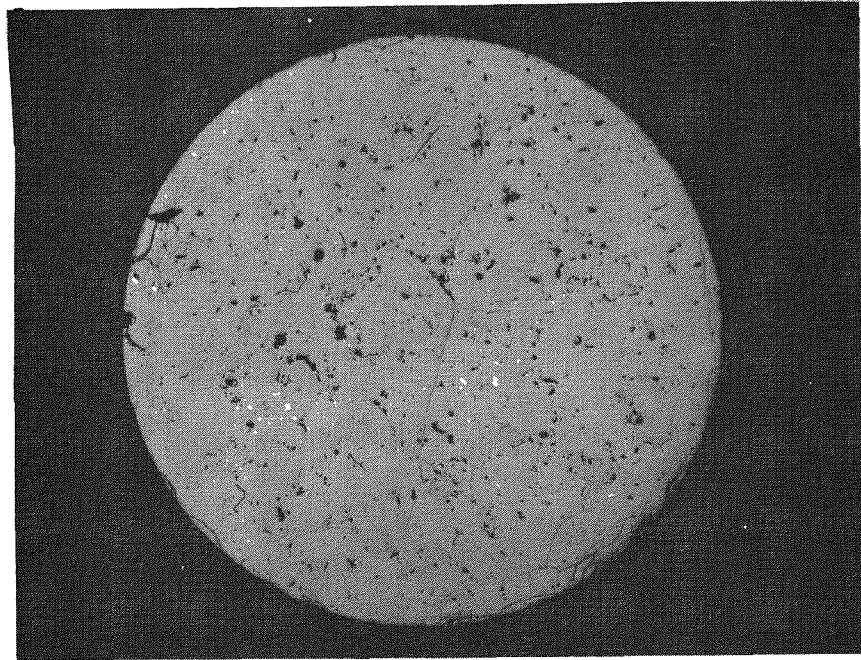
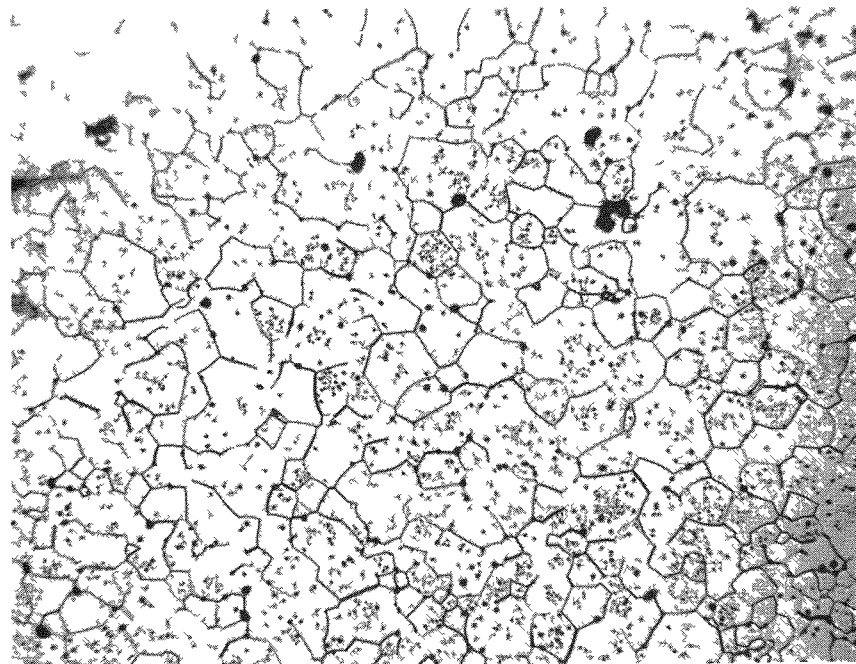


Figure 7. Shipping Container Used to Transport U-233 Nitrate to NFS. (Courtesy of ORNL)



EP R1108

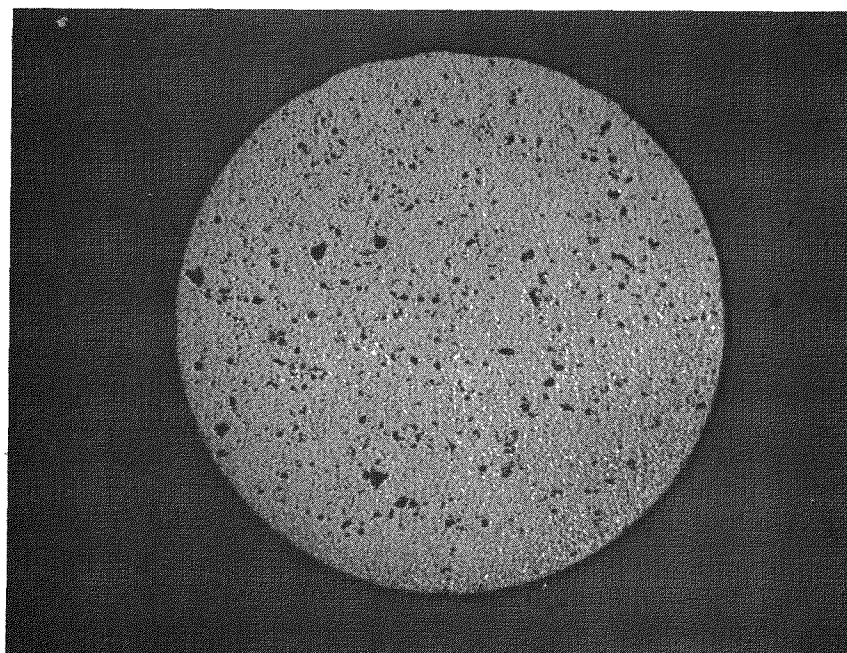
8x



ETCHED

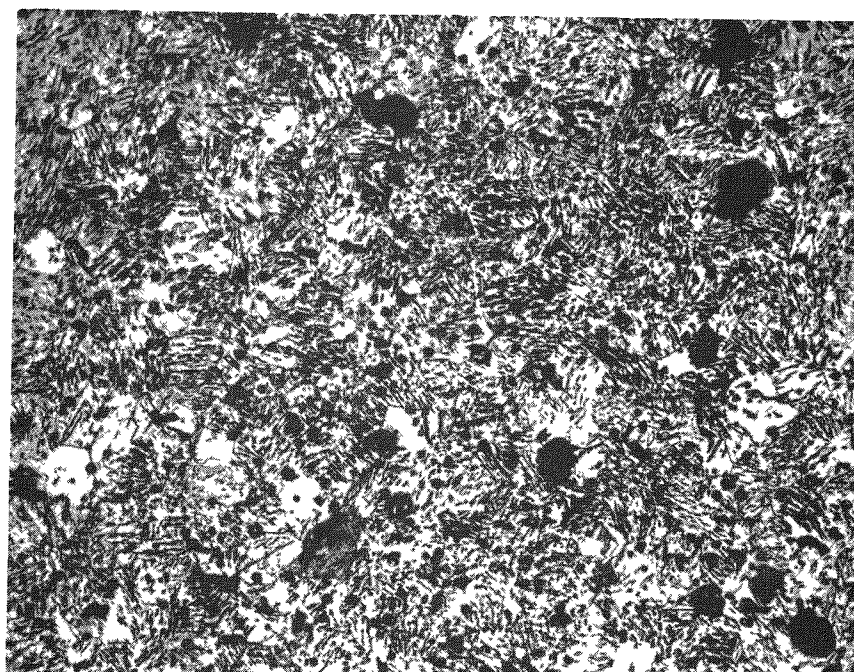
250X

Figure 8. $\text{UO}_2\text{-ThO}_2$ Microstructure.



EP R1109

15X



ETCHED

250X

Figure 9. UO₂-ZrO₂ Microstructure.

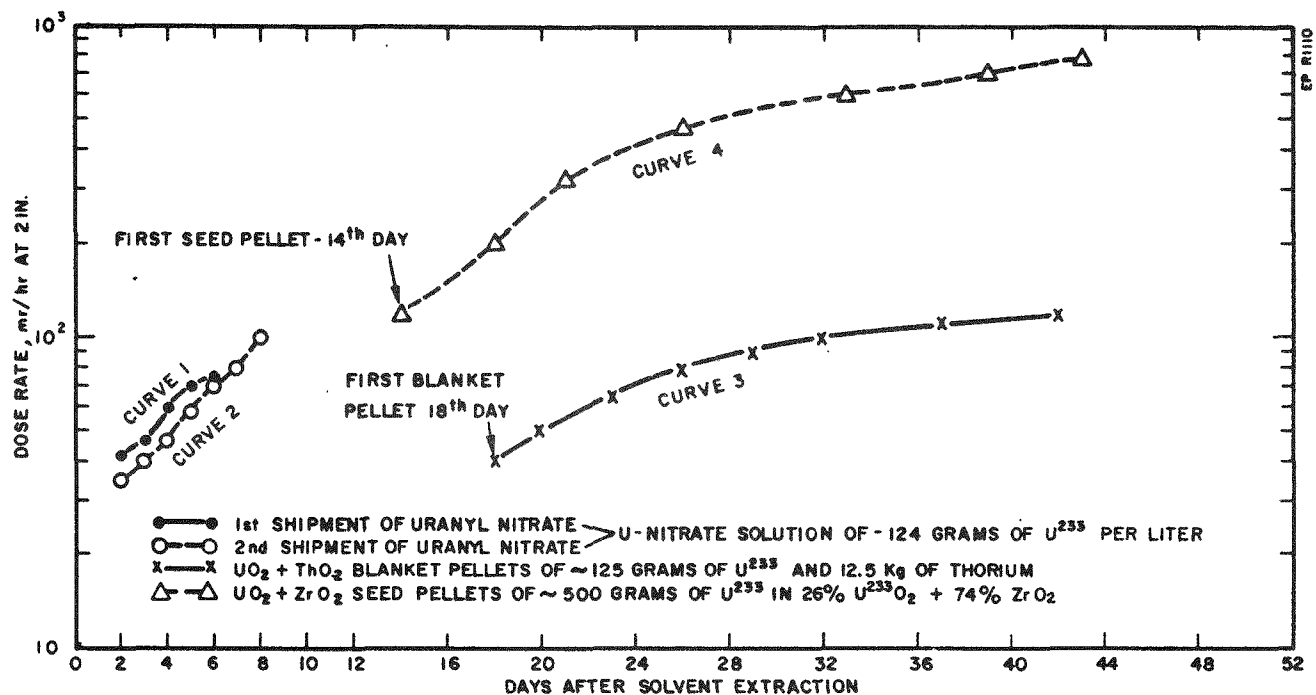


Figure 10. Radioactive Buildup of U-233 Fuels.

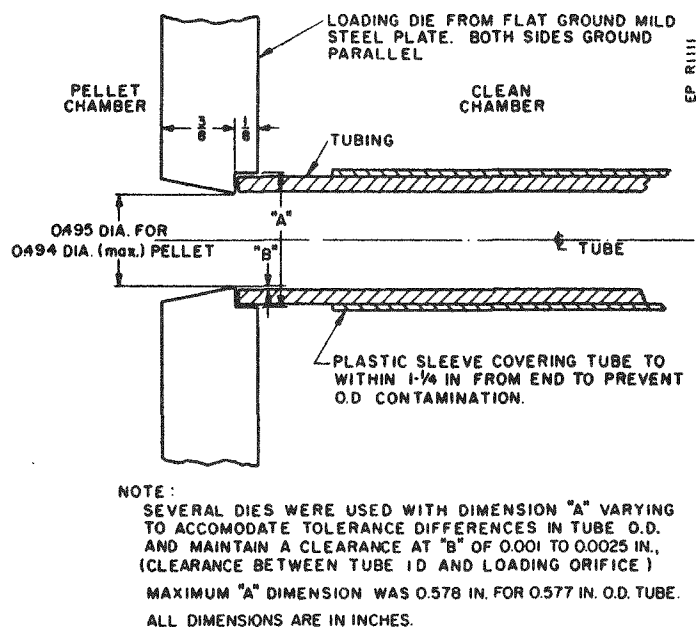


Figure 11. Tube Loading Fixture.

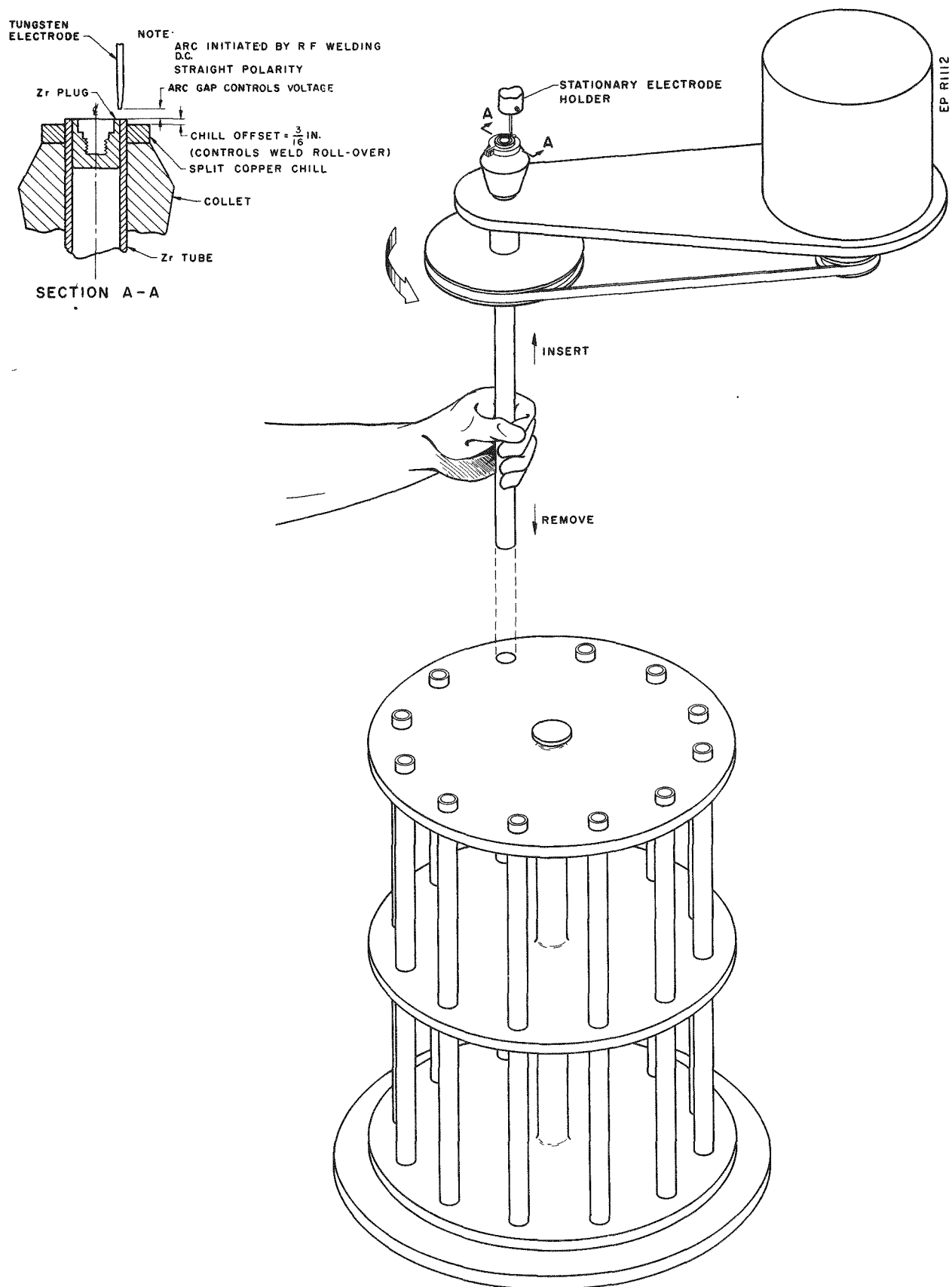


Figure 12. TIG Welding Setup for U-233 Rods.

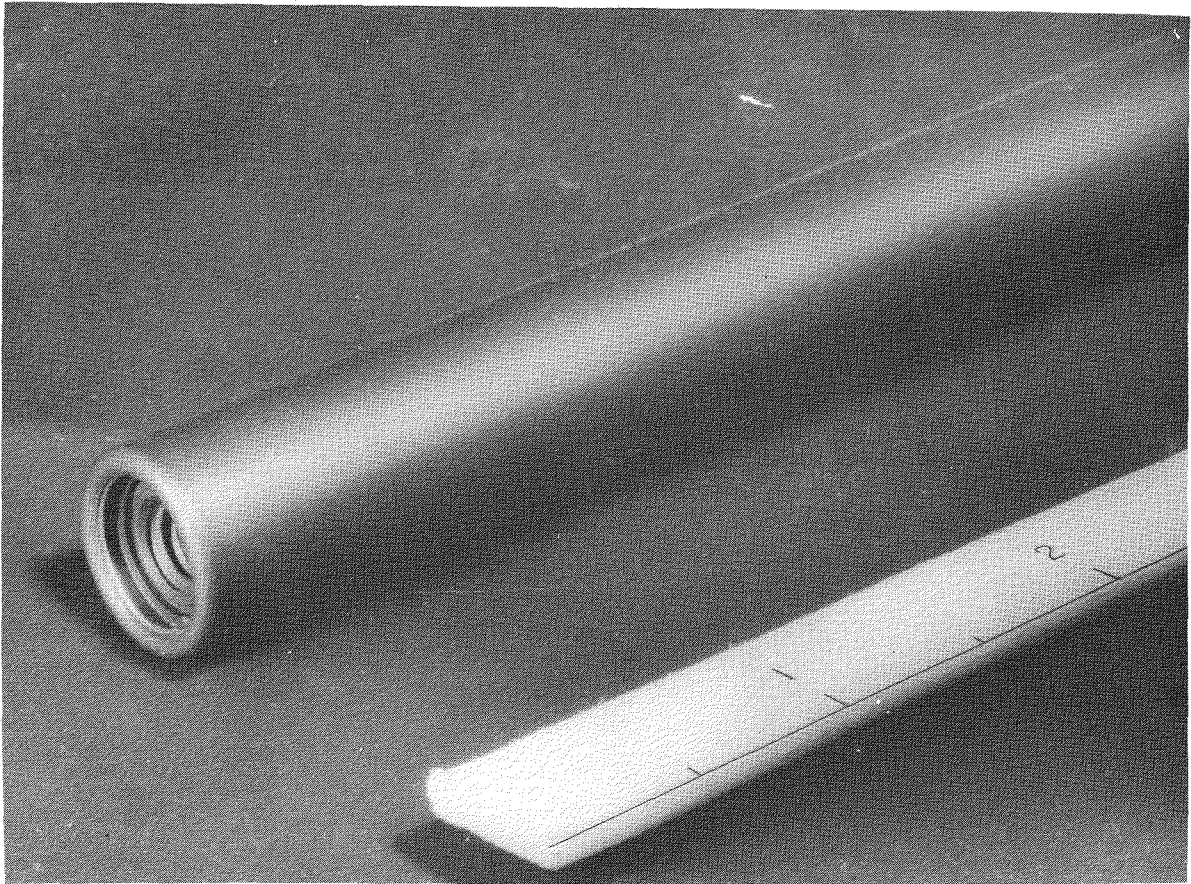
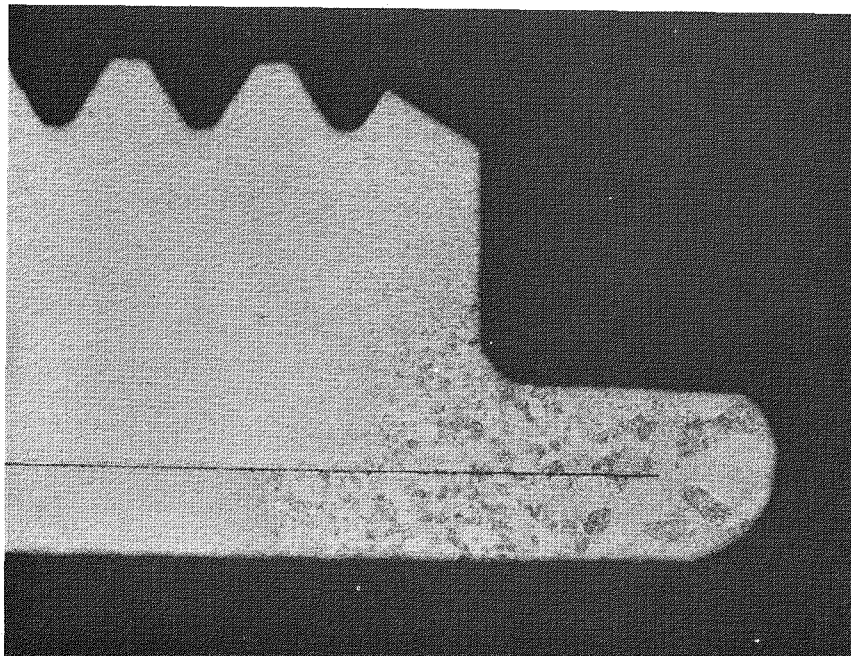


Figure 13. Typical Blanket End-Closure Weld.



15X

Figure 14. Section through Typical Blanket End-Closure Weld.

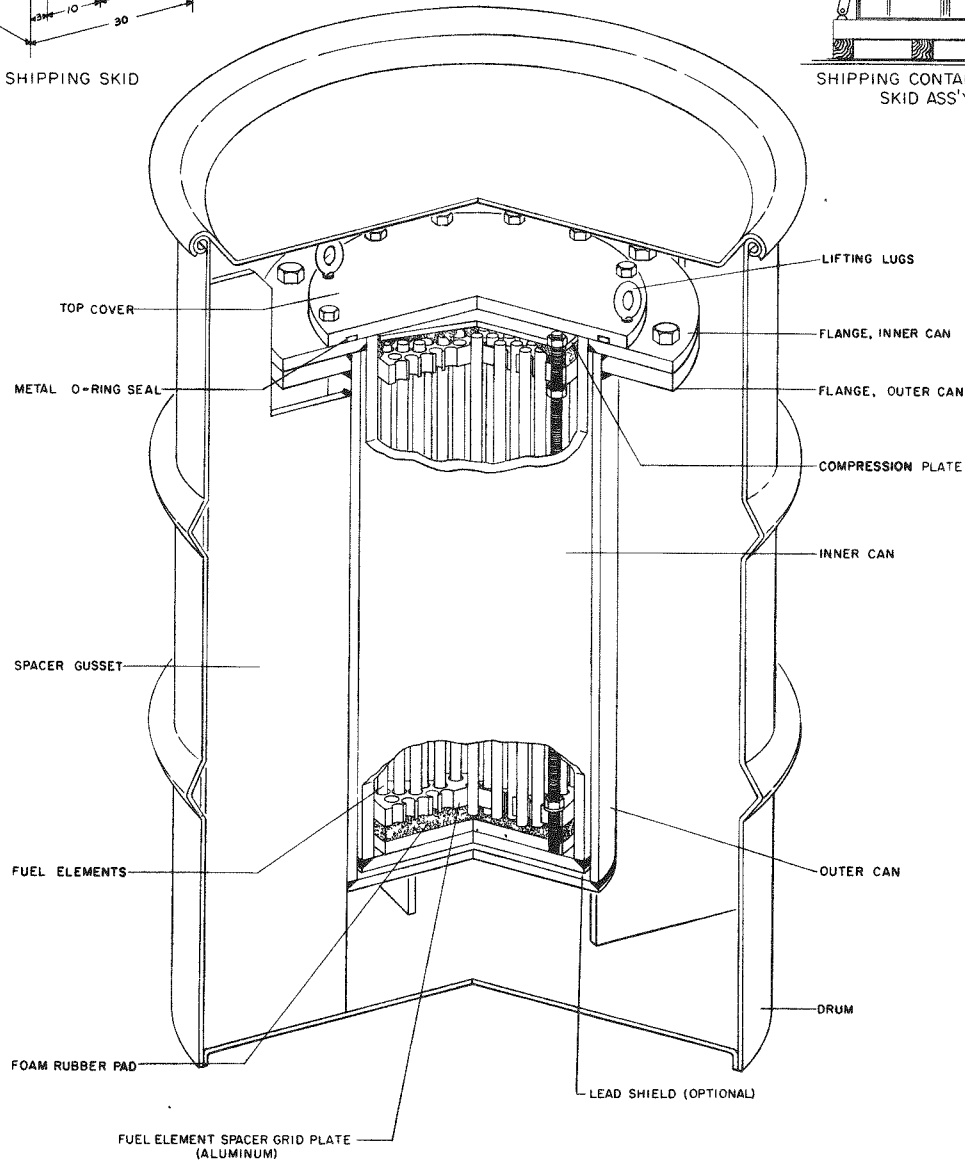
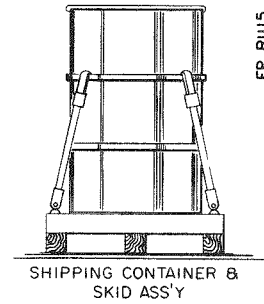
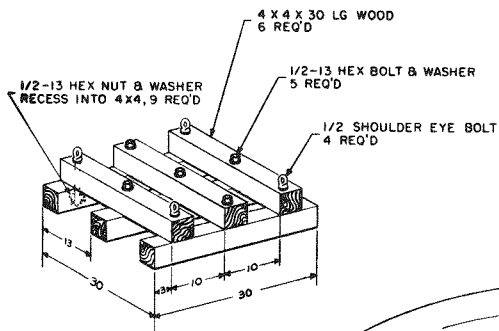


Figure 15. Fuel Element Shipping Container.