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THE DEVELOPMENT OF A DESIGN AND FABRICATION METHOD FOR PLUTONIUM-BEARING ZERO-POWER REACTOR FUEL ELEMENTS

by

A. B. Shuck, H. F. Jelinek, A. G. Hins,
N. J. Carson, Jr., A. A. Denst,
and T. A. Steele

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9700 South Cass Avenue
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ABSTRACT

Approximately 20,000 plutonium-containing fuel elements are required for physics experiments and mockup of large-scale fast reactors. These elements are in the form of stainless steel-jacketed, rectangular plates, $1/4$ in. thick by 2 in. wide, and of varying lengths from 1 to 9 in. The gross weight of the cores will be approximately 14,000 kg, and the plutonium content will be approximately 4000 kg. Of this, 2900 kg of plutonium will be produced as U-28.3 w/o Pu-2.5 w/o Mo alloy core plates. The plutonium in these core plates will be approximately 87% fissile plutonium and 13% nonfissile plutonium. The fissile-plutonium content will be adjusted to 25 w/o of the core-plate weight. Other plutonium isotopic compositions, alloys, and oxides will make up the core plates of the remaining fuel elements.

The choice of the U-Pu-Mo alloy was made to avoid the rapid oxidation and pyrophoric properties of the binary U-Pu alloys in air. At temperatures below 600°C , the alloy is not plastic,* and extremely good protective atmospheres are required if it is to be worked in the plastic range above 600°C . The method selected for development was casting the alloy into refractory-coated carbon molds to form semiprecision bars from which the core plates were machined. Although hard and brittle, the alloy is machinable at room temperature with tungsten carbide-tipped tools.

Jackets were fabricated from precisely formed, Type 304L stainless steel, rectangular sleeves and end plugs that were welded into the sleeves. Techniques that do not radioactively contaminate the outer surfaces were developed to load the core plates into the jackets. The final weld was made with the end plugs placed against the ends of the cores. This provided a finished element that expands immediately when heated by a reactor power excursion. The closing weld was also made in a reduced pressure (approximately $1/3$ atm) of argon-helium gas mixture.

*Plastic--capable of deformation without rupture.

The proposed method of fuel-element manufacture was tested by the pilot fabrication of fuel elements at Argonne National Laboratory, and the investigation was extended to a study of fabrication costs for manufacture at ANL of a production lot of fuel elements. The work culminated in a specification to be used for the commercial and AEC plant fabrication of U-Pu-Mo alloy fuel elements for the Zero Power Plutonium Reactor and for ZPR-6 and -9 experiments.

INTRODUCTION

Plutonium criticality and physics experiments in zero-power reactors will require a large number of fuel elements. These elements will consist of uranium-plutonium alloy core plates jacketed in stainless steel. Approximately 11,000 kg of the alloy fuel, containing 2900 kg of plutonium, will be fabricated for critical experiments in the Zero Power Plutonium Reactor (ZPPR). An additional 3500 kg of the alloy fuel elements are required for physics experiments in ZPR-6 and -9. This fuel will contain approximately 1000 kg of plutonium. It is desirable to use the same fuel-element design in all three reactors. Fabrication is scheduled to start during Fiscal Year 1967 and to be completed in Fiscal Year 1970.

The inventory of previously fabricated zero-power reactor fuel elements containing plutonium includes slightly more than 200 kg of 93% ^{239}Pu -1.1 w/o Al alloy plates fabricated in 1960 and 1961. Nearly 1000 kg of U-20 w/o Pu-2.5 w/o Mo alloy plate elements were fabricated from 90% ^{239}Pu in 1964 and 1965. About 23 kg of Pu-1.2 w/o Al elements, containing 22 w/o ^{240}Pu , were fabricated in 1966. Experience from the fabrication and use of these fuel elements formed the basis for improvement of the element design and fabrication methods.

A. Requirements

The zero-power reactors require fuel elements with the following characteristics:

1. Each fuel element of a given size must be of known and uniform weight and composition.
2. The fuel elements must have a prompt, positive expansion in the longitudinal direction when heated by a power excursion.
3. The core plates must not burn if exposed to air.
4. The outside of the jacket must remain free of plutonium contamination.
5. The elements must withstand shipment, normal wear, and repeated service without distortion or jacket leakage.
6. The fuel elements must be practical to manufacture.

To meet these requirements, the core alloy should have a high coefficient of thermal expansion, sufficient strength to expand the jacket, and resistance to oxidation and ignition. The jacket must fit the core tightly in the longitudinal direction, and have flat ends to transmit thermal expansion to the adjacent elements in the reactor assembly. If the jacket is gas-bonded to the core, the pressure of the gas should be sufficiently low so that the jacket does not inflate at reduced barometric pressure or at increased operating temperature.

The binary U-28 w/o Pu alloy oxidizes rapidly in air. Under certain conditions, the temperature at which ignition and sustained burning can take place in air is near room temperature. The oxide is powdery, and the powder adds to the problem of radioactive contamination. The metallic plutonium alloys for fast critical experiments have been investigated,¹ and a U-20 w/o Pu-2.5 w/o Mo alloy was recommended for the SEFOR Critical Experiment. A similar U-28 w/o Pu-2.5 w/o Mo alloy has been developed for the ZPPR fuel. These alloys have the requisite physical properties of thermal expansion and strength. The alloys oxidize slowly under air exposure at temperatures below 400°C. Ignition tests by Fischer and Schnizlein^{2,3} revealed that sustained reactions take place in a rather limited temperature range between 600 and 700°C. Below 600°C, there was insufficient heat to sustain combustion. Above 700°C, the combustion tends to be extinguished by the accumulation of adherent oxides.

The alloys have very little room-temperature ductility, although some increase in malleability and ductility does occur above 550°C. Above 450°C the alloy rapidly getters oxygen from an inert atmosphere containing less than 0.01 v/o oxygen and 50 ppm moisture. Very high-purity atmospheres will be required if the metal is to be worked in the 550-650°C plastic range.

The plutonium alloys must be handled with adequate knowledge of, and respect for, their hazardous properties. Plutonium is an intense source of alpha radioactivity and a mild source of gamma rays and neutrons. An analysis of personnel exposure to radiation from plutonium showed that the ZPPR compositions can be fabricated by normal glovebox techniques with local shielding of melting and machining operations.^{4,5}

B. Previously Used Fabrication Methods

Two plutonium-containing loadings have been made for experiments in ZPR-3. The first of these loadings was made by the Dow Chemical Company at the Rocky Flats Plant from plutonium, δ -stabilized with 1.2 w/o of aluminum. This malleable material was rolled from cast slabs to the required thickness of 0.085 to 0.086 in. (2.1 to 2.20 mm), and strips were sheared and machined to a width of 1.763 to 1.767 in. (44.78 to 44.88 mm).

Plates were cut to lengths of 0.930, 1.930, and 2.930 in. (23.6, 49.02, and 74.42 mm). The edges were radiused, and weights were adjusted by tumbling the plates with an abrasive compound for controlled periods of time.

The jackets were fabricated by folding 12-mil, Type 304 stainless steel strips around a rectangular mandrel and welding one edge. A flat plug was welded into the bottom. The jackets were made to a comparatively loose fit to facilitate core loading. A flat spring was placed in one end of the jacket to hold the core plate against the opposite end of the jacket. Originally, each plate was coated with nickel deposited from carbonyl to allow loading and welding without jacket contamination and to form a secondary oxidation barrier. The carbonyl-nickel coating was found to be impractical to apply and prohibitively costly. Dow Chemical Company personnel developed a method of loading that protected the jacket from contamination by means of a disposable, shim-stock loading funnel and sheath. This method was used for a large portion of the loading.

The side of the element through which the jacket was loaded was closed by a stainless steel plug strip. The welds were made at subatmospheric pressure of helium-argon gas mixture by means of a TIG welder developed at Argonne National Laboratory. The reduced-pressure welds were required to prevent inflation of the jackets at lower atmospheric pressure and by thermally expanded gas. The external atmospheric pressure forced the sides of the jacket against the core plate, and the springs were not sufficiently strong to hold the core against one end of the jacket during thermal expansion and contractions. The objections to this design are: (1) The element does not expand on a temperature excursion, and (2) the ratio of stainless steel volume to core volume was objectionably high.

The second ZPR-3 plutonium loading was manufactured by the Nuclear Materials and Equipment Company (NUMEC). The fabrication process was a slight modification of a procedure developed at ANL and described in ANL-6955.⁶ A U-3.10 w/o Mo master alloy was prepared in yttria-coated graphite crucibles. This was remelted with 19.55 w/o plutonium in yttria-washed carbon crucibles. The metal was bottom-poured into precision molds of refractory-washed carbon. The resulting castings were inspected, the sprue was notched and broken away, and core plates were machined to length. The as-cast widths and thicknesses were generally within tolerance, and the resulting core plates were brushed clean and adjusted to weight by milling one edge.

The jackets were made from 0.012-in. (0.30-mm)-thick, Type 304 stainless steel panels. They were drawn with a cavity depth one-half the thickness of the plate to be jacketed. Cavity-length, -width, and one -corner configuration was designed to produce a tight fit on the core plate. A turned-out rim was provided for welding. Strips of stainless steel were tack-welded

upon the end rims as filler metal. The core plate was placed between two jacket panels and the assembly clamped between copper welding chills that prevented burnthrough into the core plate. Two longitudinal seam welds were made. Runout bars were clipped over the ends of the transverse joints, and a weld was made to produce a cylindrical weld bead approximately 0.100 in. in diameter. The last weld was made at $1/3$ to $1/2$ atm of argon-helium pressure so that the element internal pressure was below that of the Idaho altitude pressure. The heavy end welds were milled parallel to each other to produce bumper shoes to transmit the fuel-element expansion to adjacent elements.

The principal advantage of the SEFOR Critical Fuel design was the ease of assembly of core plates into the close-fitting jackets with minimum contamination risk. The disadvantages of the design were (a) high rejection rates of castings, (b) exposure of edge welds to handling damage, and (c) volumetric inefficiency.

PART I

FUEL-ELEMENT DEVELOPMENT

The purposes of the work described in this report were as follows:

1. To design an improved fuel element.
2. To develop and pilot-test methods for manufacturing the fuel elements.
3. To write specifications for commercial procurement of the fuel.
4. To develop means for technical control of the commercially fabricated elements.

A. Fuel-element Design

The reference design for the fuel element was developed in cooperation with the Idaho Division, the ZPPR Project Organization, and the Reactor Physics Division. The alloy was U-28 w/o Pu-2.5 w/o Mo. The fuel element is shown in Fig. 1, the core plate in Fig. 2, and the jacket components in Figs. 3 and 4. The nine different lengths of fuel elements each have the same cross section and the same end closures. This design allows the cores to be cut from lengths of cast or fabricated U-28 w/o Pu-2.5 w/o Mo alloy bar. Core length, end parallelism, and end perpendicularity to the sides are the most critical dimensions. The core-plate thickness can vary between 0.200 and 0.205 in. The width tolerance of 1.900 to 1.960 in. was provided so that the core weight could be adjusted to within the limits tabulated on Fig. 2.

The fuel elements will be repeatedly shipped between Idaho and Illinois and will eventually be handled by mechanical loading devices. Very strong jackets with comparatively thick welds and end plugs were indicated, even at the expense of some additional stainless steel thickness over the previously designed element. The core-plate-to-stainless-steel ratio was reduced by the use of a thicker core plate and by elimination of the radiused corners. Accordingly, the jacket thickness was increased from 0.12 in. (0.30 mm) to 0.015 in. (0.38 mm). The jacket sleeves were made from precision-formed rectangular tubing. The end plugs were cut from rectangular wire or strip, and finish-machined. This design substantially reduced the tooling cost when compared to methods that require separate molds or dies for each fuel-element size.

Cross-sectional clearance is provided so that the plutonium alloy core plates can be loaded into the jackets by contamination-preventing techniques. The 0.100-in. (2.5-mm)-thick end plugs allowed heavy welds to be made. These welds possessed good strength and were plutonium- and helium-tight. Well-designed chills are required to prevent heating of the plutonium alloy cores. The welds can be made at relatively high

NOMINAL LENGTH	1	2	3	4	5	6	7	8	9
A Gage Maximum	$\frac{1.010}{1.000}$	$\frac{2.010}{2.000}$	$\frac{3.010}{3.000}$	$\frac{4.010}{4.000}$	$\frac{5.010}{5.000}$	$\frac{6.010}{6.000}$	$\frac{7.010}{7.000}$	$\frac{8.010}{8.000}$	$\frac{9.010}{9.000}$
Gage Minimum	1.000	2.000	3.000	4.000	5.000	6.000	7.000	8.000	9.000

NOTES:

1. Fuel element must be evacuated or filled with dry Argon-Helium gas at less than 1/2 standard atmosphere pressure.
2. Fuel element must pass by its own weight between parallel planes larger than the projected area of the elements and spaced no more than 0.250" apart.
3. Assemble and weld with end plugs in contact with core plate.
4. The maximum length of the fuel element shall not exceed the maximum lengths of the core plus the thickness of the end plugs plus .003".
5. End plugs must be parallel and square with jacket sleeve sides within .003".

350-921

Fig. 1. Drawing of ZPPR Fuel-element Assembly

NOMINAL LENGTH	1	2	3	4	5	6	7	8	9
Core Length Dimension A	$\frac{0.804}{0.800}$	$\frac{1.804}{1.800}$	$\frac{2.804}{2.800}$	$\frac{3.804}{3.800}$	$\frac{4.805}{4.800}$	$\frac{5.805}{5.800}$	$\frac{6.806}{6.800}$	$\frac{7.806}{7.800}$	$\frac{8.806}{8.800}$
Core Weight Grams	$\frac{91.56}{90.64}$	$\frac{207.7}{205.6}$	$\frac{323.8}{320.5}$	$\frac{439.8}{435.4}$	$\frac{556.0}{550.3}$	$\frac{671.9}{665.4}$	$\frac{786.2}{778.4}$	$\frac{904.1}{895.1}$	$\frac{1020.2}{1010.0}$

NOTES:

1. Surfaces marked ∇ shall be parallel and perpendicular to adjoining surfaces within 0.003".
2. Local shrinkage depressions on faces will be allowed that are not more than 0.040" deep and cover no more than 10% of the projected core surface.
3. Core plates shall not show porosity or fissures.

350-922

Fig. 2. Drawing of ZPPR Fuel Core Plate

NOMINAL LENGTH	1	2	3	4	5	6	7	8	9
"A"	$\frac{0.940}{0.935}$	$\frac{1.940}{1.935}$	$\frac{2.940}{2.935}$	$\frac{3.940}{3.935}$	$\frac{4.940}{4.935}$	$\frac{5.940}{5.935}$	$\frac{6.940}{6.935}$	$\frac{7.940}{7.930}$	$\frac{8.940}{8.930}$

Thickness in corners not to be less than .012

NOTES:

1. Surfaces marked with ∇_3 must be parallel within .003" and perpendicular with surfaces marked ∇_2 within 0.003".
2. Surfaces marked with ∇_2 must be a maximum of 0.213 apart with -5 PSIG pressure inside jacket.
3. Surfaces marked with ∇_1 must be parallel and flat within .003".
4. Edges to be free of burrs and nicks

350-923

Fig. 3. Jacket Sleeve, ZPPR Element

350-924

Fig 4 End Plug, ZPPR Element

currents, and they are not sensitive to minor variations in welding current and voltage. Prompt expansion of the fuel element, when the core is heated, required tight contact between the end plugs and the core. The end plugs are designed so that they may be pressed against the core by the welding fixture. The welds are designed so that the shrinkage and thermal contraction will draw the end plugs firmly against the cores.

The fuel element designed for ZPPR and the two previously fabricated designs for ZPR-3 plutonium fuel loadings are shown in Fig. 5.

350-90

Fig 5. Plutonium-loaded Elements for Zero Power Reactor (A) ZPR-3 Pu-1.1 w/o Al Element, (B) ZPR-3-SEFOR Critical U-20 w/o Pu-2 5 w/o Mo Element, and (C) ZPPR Element

B. Fabrication Methods

There are three potential methods for fabricating the core plates: (1) precision casting, (2) casting and rolling, and (3) casting and extrusion. Time limitation dictated the early selection and development of the most promising of these methods. The reference alloy is hard and nonductile at room temperature. Very little plasticity develops until the alloy is heated to above 500°C. The zero-power reactor groups have asked for a flexible design, so that plate-type elements can be made in lengths of from 1 to 9 in. by 1-in. intervals. The maximum acceptable thickness is 0.250 in., and the width is governed by the 2-in. drawer depth of all three reactors.

An objection to the individual-plate casting technique, used for the previous production, is that a separate mold and die are required for each core-plate size. A small defect or a crack in one end of a large plate caused the rejection and remelting of the entire plate. The production of precision-sectioned bar, which could be readily subdivided into any length of core plate, appeared to be the answer to the objections to precision castings. The molds, or bar-forming tools, could all be of the same size, and small defects in the bars could be cut out with a minimum production of return scrap.

The alloy is somewhat difficult to machine. It ranges in hardness from 35 to 42 Rockwell-C and is brittle. Chips tend to break out from the machined edges. Machining techniques were needed for parting off and finishing core plates. Machine cuttings are not efficiently remelted, and this is an important factor in plutonium losses and recovery costs. Therefore the production of machine chips should be minimized.

Jacket components were developed in cooperation with Fabrication Procurement Services and Central Shops Division. Emphasis was placed on the development of techniques that yield good precision and freedom from defects. The philosophy used for making the jacket components was the same as that used for fabricating the cores. The jacket components were fabricated as continuous lengths of formed tube from which the jacket sleeves and end plugs were parted.

The control of contamination during fuel-element assembly is a potentially costly problem. An alpha contamination of 100 dpm on the surface of the fuel element represents 1.4×10^{-9} g of plutonium. Techniques are required to minimize the risk of contamination of the jackets during assembly. Weld contamination has been a serious problem because welds with fused-in contamination are almost impossible to decontaminate, and because the welding arc tends to spread the contamination over the equipment.

Welding and weld inspection has been a source of difficulty in the procurement of commercial fuel elements. Few vendors realize the precision of the welding fixtures and the degree of arc control required to satisfactorily weld thin stainless steel jackets. The existing code standards and inspection requirements that form the basis for most commercial welding practice do not cover the conditions for fuel-element jacket welding. Objectives of this program were to develop welds that were relatively insensitive to variations in arc current and to provide weld-inspection standards for zero-power reactor fuel elements.

C. Alloy Preparation and Casting

The U-28 w/o Pu-2.5 w/o Mo alloy was prepared in two steps: (1) by alloying uranium with molybdenum, and (2) by adding plutonium to the binary alloy. A U-3.54 w/o Mo binary alloy was first melted and cast into ingots. The ingots were broken up and samples taken for analysis. The U-28 w/o Pu-2.5 w/o Mo was made by remelting the binary alloy with

plutonium. A small quantity of uranium was added to compensate for the deliberate overcharging of molybdenum in the binary alloy. The ternary alloy was cast into multiple-bar molds. Generally, the alloy and casting procedures were similar to those developed previously for the ZPR-3-SEFOR critical experimental fuel. Several experimental melts were made before the pilot production to determine the alloying and casting parameters required for the new composition and longer molds. These melts provided bars for core plate machining, jacketing, and rolling experiments.

350-929

Fig 6 Furnace Glovebox Line

One of the furnace glovebox lines used for the development of ZPPR casting methods is shown in Fig. 6. The furnaces were of the induction-heated, bell-jar type. A schematic section through one of these furnaces is shown in Fig. 7.

350-919

Fig. 7. Furnace Schematic Drawing

the alloy, but the reaction is difficult to control at high temperatures, and the magnesium tends to deposit on the furnace parts and within the molds.

Thoria, magnesium-zirconate, zirconia overlain by thoria, and yttria have been used to coat graphite and magnesia crucibles. Thin coatings of yttria and thoria were the most successful. Yttria was preferred in the Fuel Fabrication Facility because it does not present an additional source of alpha-radioactive contamination. Coated crucibles and a stopper rod are shown in Fig. 8.

The molds were machined from a fine-grained carbon, such as National Carbon Company, Type ATJ Graphite. The machined crucibles and molds were thoroughly dedusted and a thin yttria coating applied. Two types of molds were used. One type consisted of alternate separator plates and U-shaped cavity plates. With the two end plates, these formed the multiple-cavity mold. The

The standard AEC Type V-2 crucible design was used. This crucible has a paraboloid cavity of about $2\frac{1}{2}$ -in. diameter by 9 in. deep with a $1\frac{1}{2}$ -in.-diam pour hole in the bottom of the crucible. The pour hole is plugged by a $3\frac{3}{4}$ -in.-diam conically tipped stopper rod. Graphite and magnesia crucibles were tested for melting the binary and ternary alloy. Without protection, both of these materials react with uranium and with plutonium at the melting and casting temperatures. Uranium and plutonium form carbides with the carbon crucible. Magnesia is reduced to metal in contact with uranium-plutonium. The magnesium released to the molten metal causes boiling in the melt. The bubbling action has been used to promote stirring

350-853

~ 0.125X

Fig. 8. Yttria-coated Magnesia Crucibles and Stopper Rod

molds were held together by eight steel tie rods. Spring washers were used under nuts on the tie rods to compensate for the greater thermal expansion of the steel. The U-shaped cavity plates were excessively fragile, and the assembly did not give good dimensional control. The second type of mold tested gave better results. In this design, the cavities were milled into one side of 7/16-in.-thick plates. The plates were stacked together to provide a multiple-cavity mold. The existing furnace space limits the length of cast bars to approximately 10 in. long. This mold is shown in Fig. 9. The mold cavity dimensions are shown in Fig. 10.

Fig. 9. Yttria-coated, Multiple-cavity Mold

Heating was done by 10,000-Hz induction power supplied to an uncooled coil. The preferred design is a solid molybdenum or tungsten coil situated within concentric molybdenum heat shields. The interchangeable coil assemblies were installed within a stainless steel vacuum bell. The bell-induction-coil assembly was lifted clear of the crucible and mold by means of two electrically driven screws.

The lower portion of the furnace is a fixed, short cylinder. The lift bell and the furnace bottom are externally cooled by chilled water, which is pumped at a subatmospheric pressure. No coolants are used within the furnace shell. The loads are supported on a zirconia stool, which rests on the cooled furnace bottom. Porous-alumina tiles are placed around the molds to provide radial insulation. The top of the mold extends into the furnace coil, and the bottom is cooled conductively through the zirconia

350-920

Fig. 10. Mold-plate Drawing

stool. A thermal gradient of about 600°C from the bottom of the top of the molds promotes directional solidification of the casting. This directional solidification was found necessary to cast solid bars.

The furnace is evacuated by a 6-in. oil-diffusion pumping system consisting of a fractionating diffusion pump, a booster diffusion pump, and a 32-cfm rotary roughing pump. The 10,000-Hz power is supplied to the resonant induction circuit from a 0- to 240-volt motor generator. Approximately 80 μ F of capacitance was required in the furnace coil-capacitor tank circuit.

D. Preparation and Application of Crucible and Mold Coating Material

The yttria used for mold and crucible coating was supplied as minus six-micron powder. About 100 g of oxide was mixed with 100 ml of absolute ethyl alcohol. The oxide settles out immediately after mixing but, if the mixture stands for several hours, it can be shaken up into a smooth suspension. The recommended practice is to prepare the yttria at least 15 hr before use. The coating suspension can be further diluted with ethyl alcohol if necessary. Before each use, the crucibles and molds were recoated by the following procedure: The pour hole of the crucible was stoppered, and a small amount of the mixed suspension was poured into the cavity. The crucible was tilted and rotated to spread the suspension on the crucible wall, and the excess was poured back into the container. This caused a thin deposit of the oxide to slip-cast against the dry, porous crucible wall. A small brush was used to paint the wash in the pour hole and on the crucible bottom. Any areas not properly coated were touched up. The desired crucible coating was quite thin. A heavy coating separated from the crucible and spalled off during heating. The crucibles were dried and stored in the dry, inert atmosphere of the glovebox line, or dried in air at 100 to 150°C. The stopper rod was coated by dipping in the suspension in an upright cylinder, or the coating was applied by a brush.

The same yttria and alcohol suspension was used for coating the carbon molds. Care was taken to provide a very smooth, uniform-thickness coating, which would not degrade the precision of the mold cavity. The mold cavities were coated by pouring a small amount of suspension onto the surface of the mold cavity and quickly spreading it by brushing and by tilting the mold plates. Any excess was drained away, and the coating was allowed to dry with the mold plates supported horizontally. After the flat surfaces of the mold had dried, the edges and verticle surfaces were carefully painted with the suspension and the molds again dried. For best adherence and mold replication, the mold coating should be less than 0.005 in. thick. The thickness can be judged by the shade of the coating. Dead-white coatings were too thick. A light-gray coating adhered well and seemed to give adequate protection to the mold surface.

E. Charge Materials

The materials used in preparing the alloy were 0.22% ^{235}U , depleted uranium, hydrogen-reduced molybdenum, and calcium-reduced plutonium. The uranium was cut from 5/8-in.-diam excess EBR-II blanket rods, sheared to lengths of $1\frac{1}{2}$ to 4 in. The sheared rods were vapor-degreased, pickled in nitric acid, washed, and dried prior to use in the furnace charge. The molybdenum was cold-pressed into 5/8-in.-diam x 1/4-in. tablets to reduce melting losses.

The plutonium was in the form of 2-kg reduction buttons of about 8.5% ^{240}Pu content. Some of the reduction buttons were badly oxidized. These were cleaned by brushing and scraping to remove as much of the loose oxide as possible. The buttons were broken by means of a wedge cutter and die on a 50-ton hydraulic press. Pieces narrower than 1 in. were required for charging the crucible. The melt components were weighed to an accuracy of 0.01 g.

1. Preparation of the Binary Alloy

A uranium-molybdenum alloy was prepared first by dissolving molybdenum in molten uranium. Care was required to keep the molybdenum from floating into the dross. This occurred when we attempted to melt the fine hydrogen-reduced molybdenum powder and recovery was then low. Much better results were achieved with the tableted molybdenum, which was sufficiently porous to dissolve readily. Although the crucible capacity for these tests was about 10 kg, it should be possible to prepare large amounts of the uranium-molybdenum alloy. About 3.85% of molybdenum in the charge yielded a 3.54 w/o molybdenum alloy. This alloy contained slightly more than the molybdenum needed to produce the U-28.4 w/o Pu-2.5 w/o Mo alloy, and a small amount of uranium was added to the charge for the ternary alloy.

Good results were achieved by placing a few fairly large pieces of uranium at the bottom on the crucible, placing the molybdenum on these, and adding the rest of the uranium at the top of the crucible. The larger unmelted pieces of uranium held the molybdenum down as the smaller pieces of uranium melted and trickled down through the molybdenum. Cleaned shearings of 1/16-in.-thick molybdenum plate were also tried as melting stock, but these dissolved much more slowly and with poorer recovery than the pressed tablets.

A temperature of 1450 to 1475°C for 20 min was needed to dissolve the molybdenum; to be sure of dissolving it, the melt was usually held at temperature for about 40 min. The alloy was cast into yttria-coated-carbon, bar molds of the same type as was used to cast the ternary alloy.

2. Preparing and Casting the Ternary Alloy Bars

Twelve melts were made to establish procedures for making the precision U-28 w/o Pu-2.5 w/o Mo bars. The following variables were investigated: (1) the alloy melting cycle, (2) alloying and casting temperature, (3) mold temperature, (4) crucible materials and wash, and (5) mold dimensions. The procedures required to make the alloy were modified slightly from those established for the U-20 w/o Pu-2.5 w/o Mo alloy.

Graphite and magnesia crucibles were tried for the ternary melts. The melts made in Type ATJ* graphite crucibles, coated with yttria, showed an average carbon pickup of less than 200 ppm per melt. Precautions had to be observed in using carbon crucibles. If induction power was coupled directly to the crucible, the power had to be applied gradually before the metal melted. When full power was applied at the start of the melt, the crucible wall was heated to above 1450°C where the coating was reduced by the graphite. The melting uranium and plutonium, trickling down the walls of the superheated crucible, rapidly attacked the exposed graphite. Alloy losses and carbon pickup were excessive under these conditions. When power was applied slowly, the metal was melted while the crucible wall was below 1300°C. At this temperature, the yttria remained on the crucible wall and the carbon pickup by the melt was not excessive.

Because of difficulties with carbon crucibles reported by the commercial fabricator, several melts were tried in magnesia crucibles. The binary and ternary alloys each reduced magnesia at the temperatures used for the alloying cycle. An yttria coating was applied in the same manner as to the graphite crucible. This greatly reduced the evolution of magnesium. Magnesium stirring was thought, at first, to be beneficial in obtaining a homogeneous melt. Experience showed, however, that losses resulting from splatter and excessive drossing were higher with the bare magnesia crucibles than with coated ones. The castings resulting from the coated crucible melts were of satisfactory composition and homogeneity.

The following methods of packing the crucibles gave satisfactory results: The stopper was seated in the pour hole with a gentle twisting action. Several larger pieces of binary alloys were used to block the stopper rod in position, and the other pieces of uranium-molybdenum alloy were distributed about the stopper. These pieces were placed loosely so that the crucible would not be broken by the thermal expansion of the metal charge.

The plutonium was placed on top of the binary alloy. It melted first, and contact was achieved as it trickled down through the packed pieces of binary alloy. Once the binary alloy started to melt, forming a pool of

*National Carbon Company designation.

molten metal in the crucible, the power could be turned up to melt the remainder of the charge quite rapidly. The time that the metal was kept molten and its temperature were a compromise between what would produce the most homogeneous alloy and what would minimize plutonium losses through crucible absorption, evaporation, splatter, and drossing. Vacuum melting between 10^{-2} to 10^{-5} Torr was used for all melts. Inert-atmosphere melting was not explored because evaporative losses were thought to be rather insignificant and we wanted to thoroughly out-gas the molds before casting the alloy into them. Table I lists the casting variables explored and the conditions selected for pilot testing.

TABLE I. Casting Parameters

Melt Variable	Binary Alloy		Ternary Alloy	
	Range Tested	Selected Datum	Range Tested	Selected Datum
Heating cycle, min				
To Melt	40-75	60	40-100	50
Above 1200°C	-	-	8-40	30
Above 1275°C	8-45	40	-	-
Central mold temp, °C*	150-300	200	200-600	450
Max melt temp, °C	1350-1500	1470	1300-1480	1380
Casting temp, °C	1335-1445	1400	1270-1390	1280

*The mold temperature was a function of the heating-cycle time and maximum melt temperature.

One of the core-plate castings, before removal from the mold cavity, is shown in Fig. 11. Pouring temperature and mold temperature affected the shrinkage that occurred when the castings were made. When

the pouring temperature was too high and the molds were too hot, the faces of the castings tended to dimple. This was the result of liquid contraction, which occurred as the superheated metal cooled within the plastic casting skin. If the pouring temperature was too high and the mold too cold, internal shrinkage took place. The best surface and casting soundness occurred with the mold quite hot and the pouring temperature as low as would allow a good flow. The bars were 3-5 mils thicker at the top end of the mold than at the bottom. For large-scale production it may be advantageous to taper-grind the molds at the parting line to compensate for this effect.

F. Rolling Tests

Secondary metal-working methods such as rolling, extrusion, and forging are the traditional methods of fabricating sheets from metal billets. Exploratory tests were made of billet casting and

350-855

Fig. 11. Core-plate Casting in Mold

rolling as an alternate fabrication method to precision casting. These tests included the rolling of 2 x 2 x 0.200-in. cast plates to determine rolling temperatures, and the rolling of 5/8-in.-thick slabs to determine size effects.

Rolling was done on a 5-in.-diam x 8-in., two-high laboratory rolling mill. The mill was housed in the inert-atmosphere glovebox system. The mill was equipped with a hot table and roll heater that could maintain the rolls at about 350°C. The specimens were heated in a resistance furnace. Compression tests had indicated that essentially no compressive yield occurs in the U-28 w/o Pu-2.5 w/o Mo alloy at temperatures below 550°C. Accordingly, the rolling tests were done with the billets heated to 575, 600, 625, and 650°C. The temperature of the billet was determined by means of a thermocouple in the work piece

All the pieces oxidized rapidly. The alloy was an excellent getter of oxygen from the helium glovebox atmosphere, which contained about 1500 ppm O₂ and 50 ppm H₂O. The oxide was not adherent but tended to spall off as the pieces were handled. Because of the brittle nature of the alloy, 5% reduction passes were made. The first three pieces broke. The last piece, which was rolled at 650°C between rolls at 260°C, was given several 5% passes before light edge cracking took place. It was obvious that a much better atmosphere or another method of protecting the surface would be required if loss of the plutonium were to be kept satisfactorily low.

The chilling effect of the 260°C rolls on the thin test coupons may have affected plasticity. To test this possibility, 5/8-in.-thick pieces of the alloy were cast and rolled. The first specimen was heated to 550°C and reduced 5% between rolls heated to 300°C. Figure 12 shows this unacceptable, cracked strip. The second billet was reduced 50% in five passes at 650°C with the rolls at 325°C. Figure 13 shows the rolled strip. There was little edge cracking and no evidence that further reduction could not be made, but the billet had a heavy coating of powdery oxide.

350-826

~1.0X

350-849

Fig. 12. Cracked Billet Rolled at 550°C, 5% Reduction

Fig. 13. Billet Rolled at 650°C, 50% Reduction

We concluded from these tests that the U-28 w/o Pu-2.5 w/o Mo alloy must be rolled at temperatures of 650°C or higher. However, this alloy must be protected by a much better atmosphere than was available, or it must be clad during rolling. Selection of a satisfactory cladding for 650°C rolling is not easy. Only a few metals are compatible with plutonium alloys at temperatures above 600°C. The commonly used iron and nickel alloys form low-temperature eutectics with plutonium.

G Core-plate Machining

The equipment used for machining core plates was a horizontal milling machine and a vertical milling machine. Inspection was done by standard gaging equipment, a surface plate, a balance, and a parallel-plane gage. This equipment was located in a 4 x 6 x 18-ft helium-atmosphere glovebox.

Finishing of core plates from the cast bars required (1) milling one side to produce a reference surface for parting, (2) parting the bar into core plates, (3) milling the ends, (4) milling the remaining side to a predetermined core weight, and (5) chamfering all edges. Parting was done on the milling machine with slitting saws on the arbor. Side and end chamfering was done on the vertical mill with end mills and angle cutters. Figure 14 shows the machining operations.

350-925

Fig. 14. Steps in Machining Core Plates (A) Parting, (B) Edging, (C) Chamfering, and (D) Finished Core

1. Parting of Cores

The initial attempt to machine the alloy indicated that machining at room temperature would be slow and tool life extremely poor. There was also an indication that the alloy became more machinable at elevated temperatures. An electrically heated fixture, which held four bars, was made for the horizontal milling machine to test the hot-machining characteristics of the alloy. This is shown in Fig. 14A.

Cuts were made through core plates heated to 100, 150, 175, and 200°C. The core plates machined a little better at 100°C than at room temperature. There was marked chipping at the edges of the cut. At 150°C, there was some improvement in the machined surface, and chipping was reduced, but not eliminated. Some oxidation occurred on the cast and cut surfaces. The machining characteristics of the alloy markedly improved at 175 and 200°C. Chipping was greatly reduced. The cuttings had a characteristic milling curl, and it appeared that tool life would be improved. Oxidation of the chips and core-plate surfaces was quite rapid at these temperatures, but because of the improvement in machining a number of core plates were hot-machined.

The surfaces produced were good, but the specified length and the end parallelism were difficult to obtain. For dimensional control it was necessary to stabilize the temperatures of the work pieces, milling machine, and arbor before parting. If the temperatures were not stabilized, the pieces and the machine changed dimensions as the cuts were being made. An indicator gage showed that the arbor could elongate as much as 0.010 in. during a cut. Because of the problems encountered in hot milling, additional work was done on the tools, fixtures, and procedures for room-temperature machining of the alloy. The castings had a tendency to crack when clamped to the parting fixture, but all pieces had to be firmly clamped or they would break ahead of the cutters. Any dulling of the sawteeth produced chipping along the edges of the surfaces generated by parting. The fixture described for hot parting was also used for room-temperature parting. A 0.025-in.-thick pad of aluminum was placed between the fixture and the casting to prevent fracture during clamping. Solid carbide and carbide-tipped milling saws were tested. The most satisfactory milling saws were 3 in. in diameter by 0.060 in. thick with 12 carbide-tipped teeth. They operated satisfactorily at 100 rpm with a table feed rate of 0.0075 in. per revolution. At these conditions, expansion caused by heat generation in the workpiece was kept to a minimum. The "side-cutting" type saw was preferable to the "hollow-ground" type because it was less likely to bind in the workpiece.

Experience showed that hot parting was slow and difficult to control. The cores produced were coated with a powdery oxide that was difficult to remove. There was also an element of danger when working on hot materials with rubber gloves. The single advantage of hot parting

was that it permitted the use of the more available and inexpensive high-speed-steel slitting saws. Room-temperature parting was more exacting, and the saws are more expensive, but there was no waiting for thermal equilibrium to be reached, and the surfaces produced were flat, parallel, and relatively free of oxide. These factors led to the decision to use room-temperature parting in the pilot-lot run. The useful life of the carbide cutters as originally ground was only about eight cuts. This was increased to more than 35 cuts by regrinding the cutter and chamfering the sides of the teeth. Chamfering removed that portion of each tooth that is first to dull and the most easily broken. Chamfering also reduced chipping along the edges of the cut surfaces. In some instances, the surfaces appeared polished.

The problem of edge chipping during parting was not solved until late in the run. Because the chipping could be sufficiently severe to cause a plate to be rejected, cores were parted 0.030 in. oversize. Near the end of the run, after chamfering of the sawteeth eliminated the chipping, this allowance was no longer required.

2. Facing Operations and Weight Adjustment

Cores were finished in three steps: (1) The core was milled to length, (2) the side was milled to produce cores of a specified weight range, and (3) all edges were chamfered. All three operations were carried out at room temperature with carbide tools in the small milling machine as shown in Fig. 14B. The sides and the ends of the cores were milled with 9/16-in.-diam, carbide-tipped, end-milling cutters. Two fluted cutters with a zero rake angle were easier to sharpen in the glovebox than spiral cutters. The mill was operated at 1060 rpm, and the work was fed at about 2 in./min. Roughing cuts of 0.015-in. depth could be made. Finishing cuts were less than 0.005 in. in order to produce flat, smooth surfaces.

The edges of the side and end surfaces were chamfered with 45° angle cutters made from a 1/8-in.-thick carbide slitting saw. Two cutters and a spacer were "ganged" so that both edges of a side or end surface could be chamfered simultaneously. The spacer was made from a 1/8-in.-thick slitting saw by grinding the teeth to a point. Thus the spacer "scribed" a very fine line on the workpiece that aided in positioning the cutters and made the weight of metal removed in chamfering more uniform from core to core.

The fact that a relatively uniform weight per unit length was removed in chamfering allowed the core weight to be adjusted before chamfering. The side of each core was shaved with the end mill until a specified weight range was entered. This operation required that the core be taken from the milling machine, weighed, and returned to the machine for adjustment to final weight.

H. Analytical Sampling

The samples for chemical analyses were prepared from the excess pieces after parting. Samples were taken from the top, middle, and bottom of the bars. Approximately 1/4-in.-wide pieces were placed in the vertical mill and their thickness reduced to less than 1/16 in. Milling was done on both sides to produce a thin section that was relatively free of oxide. These sections were broken into a number of samples weighing between 0.3 and 0.6 g. No sample was taken less than 1 in. from the top or 1/4 in. from the bottom of a casting. The pieces were weighed to four decimal places and sealed in individual vials for shipment to the analytical laboratories. Nine samples were prepared from each melt. A top, center, and bottom sample were required for major-element analyses. A bottom sample was required for carbon and hydrogen analysis, one for oxygen and nitrogen analysis, and another for minor-element determinations. The remaining three samples were stored for reanalysis.

I. Core-plate Inspection and Accountability

The castings were measured for thickness. These measurements indicated if any portions of the casting were unusable and also served as core-plate thickness measurements. Thickness was measured with a deep-throated micrometer at points on a 3/4-in. grid pattern and in shrinkage areas.

Core-plate length was measured with a micrometer or a dial comparator. Parallelism and perpendicularity of the core-plate surfaces were measured by means of a dial indicator and an angle plate. The cores were weighed to 0.01 g on a direct-reading laboratory balance. An additional weighing was made just before the core plates were loaded into the jackets.

Each core plate was passed through a parallel-plane gage (Fig. 15A) to determine whether the plates were flat. The planes were 1 x 2 x 12-in. machinist parallels, spaced 0.208 in. apart. The parallels were spaced by gage blocks, and the assembly was not clamped. The core plates were passed through the gage with their larger faces horizontal and their longest side parallel to the length of the gage. They were completely contained within the gage at one point in the test. Any interference sufficient to move the upper parallel was considered cause for rejection.

The length tolerance of ± 0.002 in. was not difficult to attain after the parting and facing parameters were fixed. We found that gage blocks could not be wrung together in a dry inert atmosphere. The error increases as the number of blocks in the assembly increases. We recommend that one-piece gage standards be made for use in production. A simple go/no-go gage was made for measuring length and width in the glovebox line. This gage is shown in Fig. 15B.

Fig. 15

Core-plate Inspection Equipment (A) Parallel Plane Gage, (B) Go/No-go Length and Width Gage, and (C) Edge Perpendicularity Gage

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The gage used to measure parallelism and perpendicularity of the ends and sides of the core plates is shown in Fig. 15C. The 0.003-in. specified maximum deviation from parallelism and perpendicularity was shown to be achievable. The core-plate weight range given in Specification PF-1600, Revision, was readily achieved. The thickness given in Specification PF-1600 of 0.200 to 0.205 in. with a minimum in shrinkage areas of 0.160 in. can be met. In some cases, high spots (mold marks) had to be removed. This was done by an end milling. All cores of 3 in. or more in length passed between parallel planes spaced 0.028 in. apart. There was little evidence of warp or bow in the core plates or in the castings.

A complete weight balance was carried out on each operation in which the casting or core changed weight. The weights of the products produced were added and compared to the weight of raw materials entering. All items were weighed to 0.01 g; nothing was found by difference. Table II is the weight-balance sheet for the machining of castings from melt R-307. The overall balance is a loss of 0.83 g of alloy. This is about the maximum discrepancy tolerated. For most batches, and for the total of all batches, the balance was a gain and not a loss. This may have resulted from oxidation of core plates and machine cuttings.

TABLE II. Core-plate Machining Weight Balance^a

						MELT NO. <u>R-307</u>
						BATCH NO. _____
						DATE <u>5-2-66</u>
INPUT						TOTALS
1. Casting No.	<u>1</u>	<u>2</u>	<u>3</u>	<u>5</u>		
Casting Wt.	<u>1144.31</u>	<u>1130.71</u>	<u>1132.82</u>	<u>1163.22</u>		<u>4571.06 g</u>
2. Casting Scrap Remelttable	<u>0</u>	Reclaimable <u>0</u>				<u>0</u>
3. Total						<u>4571.06 g</u>
PRODUCTS						
1. Analytical Samples						
Vial No.	Designation	Weight	Vial No.	Designation	Weight	
117	R-307-1T	0.4503	125	R-307-2B	0.5053	
118	R-307-1T	0.5198				
119	R-307-3C	0.3064				
120	R-307-3C	0.5804				
121	R-307-2B	0.5677				
122	R-307-2B	0.6010				
123	R-307-2B	0.5810				
124	R-307-2B	0.4669				
					4.5788	<u>4.58 g</u>
2. Core Plates						
Core No.	Rough Weight	Finished Weight				
100	441.66	437.37				
101	442.16	437.21				
102	443.78	437.72				
103	443.63	437.73				
106	451.60	437.70				
107	450.01	437.47				
108	453.13	437.78				
109	456.86	437.70				
						<u>3500.68 g</u>
3. Scrap Generated in Machining						
Remelttable	<u>704.75</u>	Reclaimable <u>360.22</u>				<u>1064.97 g</u>
4. Balance [Gain or (Loss)]						<u>(0.83 g)</u>
Percent Balance						
76.6 = percent to core plates						
15.4 = percent to recycle						
7.9 = percent to recovery						
0.1 = percent to samples						
<u>100.0%</u>						

^aThis table does not represent the yield expected from a production batch in which castings would be sized to maximize the yield.

J. Jacket Development

Each jacket consisted of a sleeve (Fig. 3) and two end plugs (Fig. 4). The end plugs were identical, and the sleeves differed in length only for the elements of different size. The end plugs were designed to be made from shaped wire, and the sleeves from formed tubing. A vendor was found for the shaped wire. The vendors felt that the dimensional tolerances and tubing specifications could not be met without substantial development costs. A process was therefore developed in ANL shops and demonstrated on the production of 8000 jackets to meet the initial requirements for 148,000 jackets for fuel and sodium elements.

The fabrication method developed was to flatten a welded, Type 304L, stainless steel tube over a steel mandrel. The rough-shaped tube was then

precision-die-formed to the final dimension. The initial work was done on 0.015- to 0.016-in.-thick stainless steel sheet stock sheared to 9.5- by 4.212-in. rectangles. These were rolled into cylinders by means of a three-roll cylinder former. The edges of the cylinder were clamped in a chill fixture and TIG-welded by means of a mechanically traversed torch. The welding current and torch traverse speed were carefully controlled so that the weld thickness was not less than 0.014 in. or greater than 0.018 in.

The resulting cylinders were flattened over a hardened-steel bar, 0.206 in. long by 1.843 in. wide. Next, the four 90° bends were formed by means of an edging die. The rough-shaped sleeve was flattened and then annealed for 45 min at 785°C in a vacuum furnace.

The precision-shaping operation required more development than was expected. First attempts were to expand a tube into a box die by drawing a plug through the inside of the tube. Nearly all the deformation occurred near the four corners of the tube, which were excessively thinned or broken. Several designs of plugs and box dies were used, and the rough tube dimensions and heat treatments were varied, but the results were not satisfactory. Highly localized strain was developed at the corner bends of the tube as the plugs were drawn through, causing corner thinning to between 0.006 and 0.011 in.

A technique was needed for distributing the deformation over the flat sides of the tube without causing corner thinning. This was accomplished by expanding the rough-shaped tube into a stationary box die by means of a three-piece expanding mandrel. This method stretched the 2-in. face of the sleeve, but produced very little deformation or thinning of the corners. The corner preforming operation was found to be unnecessary. Figures 16 and 17 show the steps of flattening and stretch-forming the jacket sleeves. The two sets of tools for tube flattening and stretch forming were installed on a press brake, as shown in Fig. 16A. The tooling is $12\frac{1}{2}$ in. long and allows two sleeves to be formed at a time. Figure 16B shows the round sleeves installed on the mandrel ready for flattening. These are cut approximately $1/16$ in. longer than the finished jacket-sleeve length. The sleeves are flattened by actuating the press, as shown in Fig. 16C. The press is then opened, as shown in Fig. 16D, and the flattened sleeves are removed from the mandrel. The stretch-forming operation was done by means of a three-piece mandrel, the center piece of which consisted of a wedge operated by an auxiliary hydraulic cylinder. The expansion of the sleeve was controlled by means of a split box die, which was mounted in the press brake. The cylinder and box-die assembly are as shown in Fig. 17A. The box die was held closed by the press brake during the forming operation as shown in Fig. 17B. After each forming operation, the wedge was retracted and the box die opened for removal of the jacket sleeves, as shown in Fig. 17C. The sleeves are stretched approximately 6% in circumference and reduced approximately 0.045 in. in length. The length reduction was independent of the length of the sleeves.

350-926

Fig. 16. Jacket-sleeve Forming I, Tube Flattening (A) Flattening and Stretching Equipment in Press Brake, (B) Tube Sections on Mandrel, (C) Flattening Operation, and (D) Flattened Tubes on Mandrel

350-852

Fig. 17. Jacket-sleeve Forming II, Stretch Forming (A) Flattened Sleeves in Forming Die, (B) Forming Operation, (C) Formed Sleeve and Wedge-expanded Mandrel, and (D) Sleeve-forming Sections

Initially, abrasive cutoff saws were used to part the tubes to rough length, and the formed sleeves were milled to final length. These operations resulted in an internal burr, which was removed by hand. Shimming dies were developed in which all cutting edges were on two male tools that are slipped into the sleeve. The sleeve was held in two sets of close-fitting female tools that firmly supported the tube wall at each side of the cut. The tube and salvage end were sheared by imparting a wringing displacement between the two sets of dies. This shearing technique caused the burr to be formed on the outside edges of the sleeve, where it was easily removed. This prevented the turned-in edge, which caused difficulty in fitting the end plugs.

The fit of the end plugs into the jacket sleeves is critical for prevention of jacket contamination. The end plugs should be a light press fit with minimum corner and end gaps. The following method was used to make the end plugs: Wire was formed to the cross section shown in Fig. 4 by Turk's-head and roller-die drawing. Lengths of the straightened rectangular wire were cut 0.030 in. longer than specified by means of an abrasive cutoff wheel. The cut wires were annealed at 1060°C. The end plugs were held in a fixture and gang-milled to finished length, and the radii were ground by means of a contoured grinding wheel. This method was too costly, and shaving dies were developed to produce the corner radii. The bottom edge chamfers were ground on to the ends of the plugs. The jacket components were degreased, gaged, and visually inspected. The end plugs were checked for fit into the jacket sleeves.

K. Fuel-element Assembly

The assembled and welded fuel elements must meet the following requirements:

- a. Fixed plutonium contamination from any surface of the fuel element shall be less than 200 dpm alpha and shall total less than 500 dpm from all surfaces. Wipe counts shall show less than 10 dpm alpha.
- b. The end plugs shall be in contact with the core.
- c. The space between the jacket and the core shall be filled with inert gas at a pressure of $1/2$ atm or less.
- d. The welds shall have the general contour shown by Fig. 1 and shall be leak-tight.
- e. The assembled elements shall meet the dimensional requirements shown by Fig. 1.

1. Core-plate Cleaning

Alpha contamination was a major problem of fuel-element assembly. It was complicated by the state of oxidation of many of the core plates. Some of the cores were cast two months before jackets were available. Other core plates were hot-machined. These plates were coated with a loose oxide that tended to contaminate everything it contacted.

The oxide was removed before the core plates were taken to the low-contamination area for loading and welding. Cleaning was done in a nitrogen-filled glovebox. The core plates were scrubbed by means of a steel brush to loosen the oxide. The loose particles were removed by ethyl alcohol washing. The cores were wiped with lint-free cloth. If dark streaks appeared on the wiping cloths, the cores were again scrubbed, rinsed, and rewiped. This procedure was repeated until all loose oxide was removed. The cleaned cores were vacuum-dried for 2 hr and weighed to the nearest 0.01 g. The core plates were placed in a slotted, aluminum carrier and were sealed from the cleaning glovebox in a vinyl pouch. The cleaned cores were stored in a dry, inert atmosphere and were loaded within 3 days after cleaning.

2. Component and Funnel Preparation

The jacket components were scrubbed in detergent solution and rinsed in distilled water, acetone, and ethyl alcohol. They were placed in a 100°C oven for 1 hr. The bottom end plugs were welded in the jacket by the welding machine and chills used to weld the assembled element. A precision-ground steel block was used within the jacket sleeve to align the bottom plug while welding. Bottom perpendicularity with the sides of the sleeve was held within 0.003 in. total indicator runout. The ends of the plug were manually welded in the sleeve, and the chill was transferred to the welding machine where the sides of the sleeve were welded to the end plug. The finished welds were inspected for conformity to the drawings and for defects. A weld buildup of less than 0.001 in. was removed by stoning.

The fuel-element identification number was applied by means of a typed stencil and an electrolytic etching and oxidizing process. (An Electroetch Corporation Type SC-44 electrolyte was used with alternating current.) A neutralizing solution and a polarized oil were applied to stabilize the mark, which was an easily read black.

The top of the jacket sleeve had to be protected from contact with the core. This was done by loading the cores into the jacket through a thin funnel. The funnels had to be precisely made because the minimum clearance between the cores and jacket is only 0.003 in. Disposable funnels were desired to avoid a funnel-decontamination operation. Vacuum-formed polyethylene and acetate funnels were previously tried. The thin polyethylene lacked the rigidity required for handling and tended to tear as the cores

were inserted. The acetate funnels were more rigid, but they were difficult to form with sufficient precision. Funnels were made from 0.001-to 0.002-in. stainless steel foil, but these required hand fitting. The sharp, rigid, stainless steel edges presented a risk of cut gloves during handling. Satisfactory funnels were made from 0.003- and 0.005-in. aluminum foils by the steps shown in Fig. 18. A 3/8-in.-wide blank was punched from the foil with the die shown in Fig. 18A. This was wrapped over a plug gage, and the ends were spot-welded together, as shown in Fig. 18B. The loop was manually shaped over a forming plug and then precision-formed to a steel mandrel by means of a urethane "rubber" die shown by Fig. 18C. The funnel was held in a clamping tool shown in Fig. 18D, and the flash was trimmed with a sharp knife. The product of each of these operations is shown in Fig. 19. The 0.005-in. aluminum foil does not cut the gloves. The critical dimensions were held within 0.0015 in. The "spout" of the funnels were made 0.001 to 0.002 in. narrower than the core-plate thickness. This provided a slight braking action so that the core plate could be pushed slowly to the bottom of the jacket.

350-906

Fig. 18. Funnel Fabrication (A) Blanking Dies, (B) Spot Welding, (C) Rubber-die Forming, and (D) Trimming Fixture

350-889

Fig. 19. Funnel-making Sequence

Difficulty was experienced in loading some of the cores because of interference with the jacket and funnel. A 7° flaring tool was developed that widened the mouth of the jacket about 0.005 in. This made loading much easier. The jacket was returned to its original dimensions when it was clamped in the welding fixture.

3. Core Loading

The cores must be loaded into jackets in a glovebox that is essentially free of plutonium contamination. Contamination-preventing techniques that prevent radioactive contamination of the welds and outer jacket surfaces must be practiced. These included the practice of allowing the jacket and "clean" fixtures to touch clean surfaces only. Special tools were reserved as contaminated tools for handling the plutonium. These were placed in special holders, which prevent the contaminated surfaces of the tools from touching clean surfaces. Where possible, clean surfaces exposed to contamination were protected by adhesive tape, and large areas of the glovebox were protected by heavy aluminum foil. The plutonium cores were handled by deliberate movements that avoid the transfer of microscopic dust particles from core surfaces to clean surfaces. For example, the core plate was inserted slowly into the jacket to prevent the displaced gas from sweeping plutonium-contaminated particles from the core plates.

Figure 20 shows four steps of fuel-element assembly. Two or four jackets were held in the welding fixture, which is shown partly assembled. The top surface of the welding fixture was protected by strips of pressure-sensitive tape while the cores were loaded. The funnels were inserted into the jackets, and the fixture was placed at the loading station on a clean sheet of aluminum foil, which protected the floor of the glovebox from plutonium contamination.

350-815

Fig. 20. Fuel-element Assembly Sequence

The core plates and contaminated funnels were handled with modified laboratory forceps. The tools used to handle core plates were separately racked in a contaminated-tool holder. The sealed pouch containing the core holder was placed in a clean tray. The pouch was slit open, and a core was taken from the core holder by means of the forceps. The core plate was positioned over the funnel and inserted into the jacket. Insertion rates of 1-3 in./sec did not cause contaminating particles to be swept out of the jacket by the displacement of the helium. The core was pushed firmly against the bottom of the jacket by means of a "contaminated" push rod. After the core was loaded, the loading tools were wiped with tissue and returned to the contaminated-tool holder. The funnel was removed from the jacket and discarded to a dry-active waste container. Care was used to prevent spilling contaminated particles from the funnel onto the jacket and chills.

The plugs were handled by modified forceps that gripped the ends of the plugs by the top edges. These forceps were kept separate from contaminated tools in an uncontaminated-tool holder. The top-end plugs were machined to produce a light push fit in the jacket and were pressed firmly against the ends of the core. After the end plugs were installed, the tape was stripped from the top surfaces of the welding fixture. The tops of the jackets were wipe-surveyed by means of the ethanol-moistened swab. The swabs were dried and counted by means of a scintillation counter. Contamination in the form of loose dust could often be wiped away by swabbing, but careful techniques were required to prevent spreading

the contamination. Too much contamination required returning the elements to the plate-cleaning station for recleaning, and re-jacketing the cores in new components. When a low alpha count from the cotton swabs showed that the elements were clean, the welding fixture was clamped to fix the element components and was transferred to the welding station.

L. Fuel-element Welding

The requirements for subatmospheric pressure of inert gas within the fuel elements, positive contact between the end plugs and cores, and relatively heavy welds between the 0.015-in.-thick sleeve and end plug influenced the choice of welding equipment and procedures. Welding was done in a chamber that could be evacuated and back-filled with an argon-helium welding-gas mixture. The chamber was equipped with a torch carriage, torch with gas supply, and a vise with adjustments for positioning and aligning the welding fixture beneath the torch. The torch carriage was moved in the X direction by means of a lead screw driven by a variable-speed gearmotor. The torch holder was provided with fine screw adjustments in the Y and Z directions. The welding chamber was evacuated by means of a 140-liter/min vacuum pump through a throttling valve. A regulated flow of argon-helium mixture was supplied to the welding torch. A dynamic subatmospheric pressure was maintained on the welding chamber by controlling the gas supply and vacuum valves.

The welding fixtures (shown in Figs. 21-23), consist of machined-steel frames to which massive copper-chill blocks are brazed. The copper chills were designed to contact all four sides of each jacket

350-918

Fig. 21. Welding Fixture, Open to Show Components

350-930

Fig. 22. Short-weld Operation

350-928

Fig. 23. Long-weld Operation

sleeve within 0.030 in. of the fusion joint. The end plugs extend above the jacket sleeve 0.030 to 0.035 in. The end chill, which contacts the end plug within 0.030 in. of the edge of the plug, exposes the ends of the plugs so that the short weld can be made. Two styles of end chills were used, one of which is shown in Fig. 22. The second style of end chill clamps four elements simultaneously and exposes one of the long seams in each element. This chill is shown in Fig. 23. The chill is interchangeable between the two sides of the fixture and may be used to alternately weld each side of the jacket sleeve to the end plug.

The requirement that the end plugs be forced against the core was met by clamping the elements against individual spring-loaded plungers that exerted a 40-lb upward force against the bottom of the fuel element. This is shown in Fig. 21. This arrangement also compensated for slight variations in the length of the elements.

Welding power was supplied from a welding rectifier that was controlled by means with a magnetic amplifier and a remote programmer. A high-frequency starter initiated the arc. The weld programmer timed and controlled (a) the high-frequency starting current, (b) the torch traverse, (c) gas flow, and (d) welding current. The weld cutoff provision of the programmer was not used because weld runout blocks were provided at the ends of the fuel elements.

The following sequence of welding was developed: Transverse chills were clamped on each element in the fixture. The end welds were made by means of a manually held TIG torch. The current was controlled between 25 and 35 amperes by a foot-operated potentiometer. The shieldings gas flow was 25 cfh of 60 v/o helium and 40 v/o argon. Care was taken to prevent burning into the plutonium core. Figure 24 is a photomicrograph of welds made under satisfactory conditions.

Section through End-play Sleeve Welds Showing Desired Weld Contour

25X

Section through Sleeve Seam Weld

43X

Fig. 24. Specification Photomicrographs Showing Desirable Weld Contours of Sections through End Plug and Sleeve Weld

When the transverse welds were completed, the chill bars were removed and the fuel element inspected. Any weld buildup was removed by light stoning. The longitudinal chill bars were attached and fuel elements forced down against the spring plungers. The loaded fixture was placed in the welding chamber, and one side weld was made at glovebox pressure and with a helium-argon gas mixture flowing through the torch. The weld chill fixture was reversed and moved to the opposite side. The chamber was closed and evacuated by means of a rotary vacuum pump to under 0.2 Torr for 5 min. The chamber was purged with 60 v/o helium-34 v/o argon gas mixture. The flow was regulated to approximately 15-cfh helium, and 10-cfh argon to the electrode shroud. The vacuum valve was adjusted to maintain a dynamic chamber pressure of 6 psia. The weld was made along sides of the four fuel elements with the following settings:

Welding Parameters

Voltage	12 ± 1.0
Amperes	36 ± 2.0
Gas Flow	10-cfh argon, 15-cfh helium
Electrode	0.060-in.-diam thoriated tungsten
Electrode Gap	0.025 ± 0.003 in.
Traverse Rate	3.2 in./min

The same welder settings could be used for both atmospheric-pressure and reduced-pressure welding. A section through an acceptable weld made at reduced atmospheric pressure is shown on the right-hand side of Fig. 24.

Tests were made of weld quality versus weld current. Figure 25 shows a section through a weld made at 22 to 25 amperes. Weld penetration

350-933

Fig. 25. Section through Weld Made at 22 to 25 amperes
Showing Insufficient Weld Penetration

is insufficient and incomplete. Figure 26 shows welds made at 40 to 50 amperes. The increased weld current caused the excessive fixture heating and weld penetration into the jacket cavity.

350-934

Fig. 26. Burnthrough at Corner Caused by Slow Torch Traverse and Excessive Current

Care was required to prevent contamination during the welding operation. Contamination can be caused by high rates of evacuation, excessive welding temperature, and weld burnthrough. The incidence of contamination was reduced by evacuating the weld chamber slowly to prevent contaminating particles from being flushed out of the elements. Massive chills and current control were required to prevent overheating or weld burnthrough.

Loose and surface materials can often be decontaminated by wiping or polishing. Contamination that was deposited in the welds or dispersed by the arc was not easily removed. A rule was made that, if the contamination could not be reduced to the specified level within 30 min, the core plate was rejacketed.

M. Final Inspection

The fuel-element attributes examined by final inspection are (a) removable and fixed plutonium contamination of the exterior surfaces, (b) neutron emission rate, (c) dimensional conformity to the drawings, (d) leak tightness, (e) jacket fit as determined from measurements, X-rays, and fill, and (f) gross weight. Weight of the stainless steel jacket was determined as the difference between gross and core weights. A careful visual inspection was made for jacket defects and the welds were inspected by means of a 15X and 30X stereomicroscope. Some of the elements were submitted to the Quality Assurance Section of the Special Materials Division for gamma-spectrometric estimation of the plutonium content.

Removable plutonium contamination was estimated by wiping all surfaces of the fuel element with an ethanol-moistened $4\frac{1}{4}$ x 9-in. counter filter paper. The ethanol was evaporated from the paper, and the alpha emission of the filter paper was counted by means of a gas-proportional counter. The maximum allowable wipe count is 10 dpm.

The fixed count was determined by the direct monitoring of each flat surface of the fuel element by means of a gas-proportional counter. Count-rate meters such as the Eberline PAC gas-proportional counter were used for preliminary monitoring. If the rate meter showed an alpha count rate below 200 cpm from any surface, the fuel element was placed between two 4 x 10-in. gas-proportional probes spaced $1\frac{1}{2}$ in. apart. An integrated count was made by means of a scalar controlled by an electronic timer. The specified maximum alpha count rate with this instrument was 500 cpm.

1. Decontamination

Several methods are available to decontaminate fuel elements. Surface contamination may often be reduced by wiping with ethanol-moistened tissues or scrubbing with a chelating detergent. If these measures did not reduce surface counts to the desired level, mechanical or electrical polishing methods were used which removed a thin layer of stainless steel and, it was hoped, the contaminating material with it. Locally contaminated spots were treated by stroking with polishing cloth. Care was required to keep from spreading the contaminated area. In some cases, electropolishing in standard sulfuric-phosphoric acid electrolyte was effective. Generally, if contamination could not be reduced to an acceptable level in about 30 min, it was futile to spend more time on the elements. Contamination on the stainless steel surface could generally be removed. Plutonium in a weld could not be removed by the above methods, and the elements were rejacketed.

To reduce counting time, the integrating alpha counter was combined with a neutron counter. This instrument is described in greater detail in Part III of this report. The two alpha-proportional probes were incorporated into a paraffin block with four $^{10}\text{BF}_3$ neutron-counter tubes. Each element was counted twice--once with an aluminum shield to absorb alpha particles, and once without a shield. This allowed compensation for the proton-recoil effects on the proportional counters.

2. Visual and Microscopic Inspection

If counting indicated that the fuel elements were sufficiently contamination-free for handling, they were removed from the glovebox system for visual inspection, gaging, and leak detection. Welds were inspected for contour, cracks, porosity, or skips. The jackets were

inspected for scratches, nicks, or gouges. Fifteen- and thirty-magnification stereomicroscopes were used for this inspection.

The gages used to measure the fuel elements were similar to those shown in Fig. 15 for core-plate measurement. The element length only must be measured and recorded. This was done by means of a dial comparator. Thickness was gaged by a push-through gage consisting of two 10 x 2 x 1-in. ground parallels spaced 0.2500 in. apart by means of gage blocks. The element was required to pass this gage with its long dimension parallel to that of the gage. Any displacement of the upper parallel was an indication that the element was too thick.

A dial-indicator perpendicularity gage was used for measurement of the accuracy of end-plug positioning. Pin-type go/no-go gages were used to gage width and length. A direct-reading (Mettler P-12) balance was used to weigh each element.

The data recorded for each element were core weight, element weight, core length, and element length. Calculated values were recorded for the ^{239}Pu , ^{241}Pu , and ^{240}Pu . The weight of stainless steel was calculated as the difference between the element and core weights.

3. Leak Detection

Leaks in the jackets of fuel elements may be shown by the effect upon jacket fit, mass-spectrograph leak detection, and bubble testing. The atmospheric pressure holds the jacket firmly against the core if the pressure within the jacket is at 6 to 7 psia. If the jacket leaks, the jacket becomes loose and springy. Very small leaks were detected by pressurizing the element in a chamber with 6 atm of helium. The pressurizing chamber was then blown off, and the plates were transferred to a leak-detector vacuum-sampling chamber. A continuing flow of helium was detected by a mass-spectrometer leak detector attached to the sampling chamber. Often the pressurizing operation resulted in jacket inflation, which also indicated leakage.

A large leak tends to bleed off the impregnated helium during transfer and pump-down. Therefore, the large leaks may not be indicated by the mass-spectrometer leak detection described above. Most of these leaks may be detected by impregnation with helium and, immediately after impregnation, transferring the plates to a fluid and watching for bubbles. The pressure over the fluid may be reduced, or the fluid warmed to increase the sensitivity of the method to leaks. The following technique was used for bubble-testing experimental fuel-element production: The plates were impregnated with 6 atm of helium. The pressure chamber was opened and the plates transferred to an ethanol-filled glass desiccator. The desiccator was closed and pumped to 4 and 5 psia. The plates were inspected for bubble evolution.

PART II

PROCESS FOR THE PILOT-LOT MANUFACTURE OF FUEL ELEMENTS
FOR THE ZERO-POWER PLUTONIUM REACTOR

This describes the process details developed at Argonne National Laboratory to manufacture ZPPR fuel elements. It was used for fabrication of a pilot lot of fuel elements manufactured at ANL from April to June 1966. Short-term tooling and existing equipment were used to manufacture the pilot lot of fuel elements, but process steps were selected that appear to be adaptable to quantity production with production tooling.

Batch sizes were limited to approved, unmoderated, plutonium mass limits for general operations in the Building 350 glovebox lines. These mass limits restrict the quantities of plutonium in any of the glovebox lines to less than 2.6 kg (or 9.2 kg of U-28.2 w/o Pu-2.5 w/o Mo alloy). In quantity production, the nuclear-hazard aspects of the operation must be carefully analyzed and consideration given to alloy-dilution factors, which may allow more latitude in batch limits for some of the operations.

The environmental conditions maintained during the fabrication of the pilot lot of fuel elements were as follows: (1) complete containment in gas-tight gloveboxes of all operations that involved exposed plutonium, (2) a helium atmosphere in the gloveboxes, which contained less than 0.1 v/o of oxygen and 30 ppm of H₂O, (3) restriction of water and hydrogenous material from the gloveboxes for atmosphere purity and criticality-control reasons, and (4) melting, alloying, and casting operations under a vacuum of 10^{-3} to 10^{-5} Torr.

The fuel elements were fabricated in accordance with the requirements of Specification PF-1600, Draft II, and Drawings PF-1601A, PF-1601B, and PF-1601C. The fuel elements are used as "building blocks" for fast critical assemblies and zero-power reactor experiments. They are required in a variety of sizes, but each size of fuel element must contain a known quantity of fissile plutonium and fertile uranium. Within a given size, these quantities should agree within about 1%.

Metallic fuel containing 25 w/o fissile plutonium and approximately 69 w/o uranium-238 is required for projected physics experiments. The binary alloys of uranium and plutonium have extremely poor properties--they tend to be self-fragmenting, rapidly oxidizing, and pyrophoric. A ternary alloy, containing 2.5 w/o molybdenum, was developed to bring these characteristics within controllable limits. The plutonium available for FY 1967 manufacture of ZPPR fuel elements contains 10 to 14% of nonfissile $^{240}\text{Pu} + ^{242}\text{Pu}$. A constant isotopic content is required. This will be accomplished by preblending the plutonium to a ^{240}Pu composition of $11.5 \pm 0.5\%$. The isotopic $^{239}\text{Pu} + ^{241}\text{Pu}$ content of the ternary alloy will

then be adjusted to 25 w/o. With this isotopic composition of plutonium, the nominal chemical composition of the ZPPR fuel alloy will be U-28.2 w/o Pu-2.5 w/o Mo.

The U-28.2 w/o Pu-2.5 w/o Mo alloy blackens when exposed to the air at room temperature, forming an adherent oxide. It maintains one-piece integrity for several months of air exposure. Under furnace oxidation tests, the air ignition temperature of massive pieces was near that of uranium, approximately 600°C. The room-temperature compressive strength of as-cast alloy was over 140,000 psi, and hardness was between 35 and 42 Rockwell C. The linear coefficient of expansion of the as-cast alloy was $14.7 \times 10^{-6}/^{\circ}\text{C}$ over the temperature range of 20 to 200°C. The density of cast plate specimens was from 18.1 to 18.4 g/cm³.

The alloy displayed no plasticity between 20 and 400°C. Test ingots of the alloy fragmented when rolled at 5% reductions at temperatures below 550°C. At 650°C, the alloy rolled easily, but problems encountered at this temperature appear to make mechanical fabrication impractical. Oxidation rates were excessive, even in the helium atmosphere which contained <0.1% O₂ and <30 ppm H₂O. The oxide formed under these conditions was not adherent, and oxidation losses were high. Eutectic alloys are formed with ferrous and nickel alloys at much lower temperatures. Rolling of jacketed ingots did not appear to be practical, and there was a serious problem of sticking to tools and dies in extrusion experiments.

The alloy casts readily into oxide-coated carbon molds. Mold replication was good. Precision of the as-cast dimensions require careful control of the casting parameters. When the mold dimensions, mold temperature, melt-superheat temperature, and casting temperature were brought under control, the casting and machining operations described in the following sections of this report met the requirements for core-plate fabrication.

Because of the extreme toxicity and α radioactivity of plutonium, absolute containment of the plutonium within stainless steel jackets is required. The jackets must be gas-tight and filled with inert argon-helium gas mixture to prevent the oxidation of the core plate. The pressure of the filling gas must be less than 1/2 atm so that the jacket is not inflated away from the core at an 840-millibar, Idaho-altitude pressure and at 150°C. The fuel elements will be repeatedly loaded and unloaded both by mechanical means and manually in zero-power reactor assemblies and in storage containers. The fuel elements can have no sharp or protruding edges or weak corners. Each element can occupy no more than 0.250 in. of width, including bow, in the assembly. The fuel element must expand promptly in its longitudinal direction when the core is heated by a nuclear power excursion. This is achieved by the use of a core alloy that has a high strength and thermal expansion, and by assuring that the jacket end plugs are in preloaded contact with the ends of the core plate.

A. Synopsis of Process

Figure 27 is a flow diagram for ZPPR fuel-element manufacture. The major steps of core-plate fabrications were (1) preparation of uranium-molybdenum binary alloy, (2) melting the ternary alloy and casting it into bars, (3) parting core slugs from the bars, (4) milling the cores to precise length, (5) weight adjustment of the core by milling to a variable width, and (6) cleaning the cores to prevent contamination in the jacketing operation. Accurate materials accountability was practiced for all operations on the cores. Precise charge calculation and accurate weighing of all materials and products were required.

Fig. 27. Flow Diagram for Manufacture of ZPPR Fuel Elements

The steps of jacket-sleeve fabrication were (1) roll-forming and welding stainless steel strip into welded tube, (2) die-flattening the tube, (3) die-forming the longitudinal edges of the sleeves, (4) stretch-forming the tube into a precision box die by means of an expanding mandrel, and (5) cutting the jacket sleeves to length. End plugs were cut from formed Type 304L stainless steel wire and were finished to precision tolerance by grinding and milling. Thorough cleaning of all jacket parts was required.

Contamination-preventing methods were practiced in assembling and welding the fuel elements. After being thoroughly brushed and wiped to remove loose metal or oxide dust, the core plates were transferred to a contamination-free assembly area. Clean jackets were moved to the area, and the bottom plug was installed and welded in place. The jacket sleeves were installed in the welding chills, and aluminum-foil funnels were inserted in the jacket opening. During loading, these funnels prevent contact between the jacket lip and core plate and subsequent contamination of the weld area. The core plates were handled by special tongs and fixtures so that the jackets and contamination-free areas were not contaminated.

Welding was done in a chamber evacuated and back-filled with an argon-helium gas mixture at $1/3$ to $1/2$ atm. Copper chills contacted all sides of the element during welding to prevent the expansion of gas within the element and possible weld contamination or blowout. A spring-plunger column loaded the bottom end plug, core plate, top end plug, and top chill with about 40 lb of force as the top end plug was being welded in place. Electrode movement was mechanically controlled and programmed during welding. The weld was made by fusing the edge of the jacket sleeve and end plug by means of a dc arc drawn from a tungsten electrode. With properly designed chills and welding parameters, a radiused bead was made without buildup.

Dimensional measurement was carried out at each step of the process for quality control. Go/no-go gages will be used for final inspection of production core plates and jackets. Weighing to the precision shown by the specification is required, and measurements must be recorded for core-plate length and end-plug length. The assembled and welded element length was measured. Both bubble testing and helium mass-spectrometer leak detection was done. Whole-plate alpha counting and scanning are required by the specification. Neutron counting can be done on a sampling basis. Both alpha and neutron counting was done on all core plates of the pilot-production operation.

Precise weighing of all feed materials and products, including turnings and residues, was required for accountability as well as for composition control. Sampling and analysis were considered to be verification operations. The principal burden of accountability was placed upon weight accounting and process records.

B. Process Material

1. Depleted Uranium

The uranium used for pilot-lot production was excess material from the EBR-II blanket production. The ^{235}U content of this uranium was specified as 0.22 ± 0.02 w/o. Typical analysis of the depleted uranium is shown on the following page. Selected low-carbon derbies must be used for the ZPPR production. The uranium was saw-cut from the derbies (or for part of the pilot production, cut from scrap rolled rods). Pieces with entrained dross or slags were rejected.

2. Molybdenum

Molybdenum was in the form of 99.85 w/o purity hydrogen-reduced powder pressed into 3/4-in.-diam by 1/4-in.-thick tablets. The molybdenum tablets were used in the form supplied, or they could be crushed or broken for charge weight adjustment. Note: Powdered molybdenum caused difficulty in alloy preparation. Scrap molybdenum plate shearings were satisfactory.

3. Plutonium

Plutonium for ZPPR pilot-lot production was withdrawn from the ANL research allocation. The total impurities in this material were less than 0.10 w/o. The ^{239}Pu range was from 90.60 to 90.98 w/o; the ^{240}Pu range was 8.15 to 8.50 w/o; and ^{241}Pu was from 0.83 to 0.88 w/o.

Although 11.5 w/o ^{240}Pu is not available for pilot-lot production, the composition of the alloy charges were made up for U-28.30 w/o Pu-2.50 w/o Mo. The plutonium buttons were inspected for oxides and dross. Those that were wrapped in plastic were severely oxidized and corroded. Scraping, wire brushing, and liquid-nitrogen immersion was used for cleaning. Nitrogen-immersion cleaning was effective only for heavy slags or oxides, causing them to spall from the surface of the plutonium button. The buttons may frost after liquid-nitrogen treatment unless the glovebox atmosphere is very dry. Note: DO NOT DEGREASE OR CLEAN PLUTONIUM IN HALIDE SOLVENTS--IT MAY REACT DANGEROUSLY.

Plutonium buttons were broken or crushed at room temperature or sheared at about 200°C. The equipment used for breaking and cutting the plutonium buttons was a 50-ton hydraulic press equipped with a wedge cutter and die in a telescoping (pillbox) fragment shield.

UNION CARBIDE NUCLEAR COMPANY
POST OFFICE BOX 1223
PADUCAH, KENTUCKY

ANALYSIS OF URANIUM METAL INGOTS

					Date <u>May 27, 1959</u>
Ingot Identification	3950	3951	3952	3953	3954
W/o ^{235}U	0.2115	0.2131	0.2122	0.2177	0.2118
Density, g/cc	18.97	18.94	18.96	18.98	18.96
Spectrochemically Determined Impurities (ppm U basis)					
B	<0.4	0.8	<0.4	<0.4	<0.4
Cd	<0.7	<0.7	<0.7	<0.7	<0.7
Cr	<20	<20	<20	<20	<20
Cu	20	7	10	10	10
Mg	<7	<7	<7	<7	<7
Mn	90	35	50	35	50
Si	90	120	70	70	140
Fe plus Ni	210	170	210	125	155
All others*	100	100	35	35	100
Chemically Determined Impurities (ppm U basis)					
Nitrogen	10	21	20	17	13
Carbon	255	375	310	210	380

*All others include only Al, Be, Co, P, Pb, Sn, V, and Zn.

Original Signed By

V. G. Katzel
Chemical Laboratory Department

C. Pilot-production Procedures

1. Accountability and Materials Weighing

The intent of these procedures was to control accountability and compositions by accurately weighing feed materials, products, and residues, and to maintain compositional records on the basis of charge calculations. Chemical, spectrographic, and mass-spectrometric analyses were used to confirm calculated compositions or to locate errors in charge compositions. All feed materials and products were weighed where Fig. 27 indicates WEIGH.

a. Equipment. Plutonium is about 30 times as valuable as gold. Its worth justifies the use of high-grade analytical-type balances or bullion balances, not "process scales." The following equipment was used in the ANL pilot production:

- 1 Glovebox PF-7, helium-filled, 4 x 4 x 18 ft.
- 1 10-kg SECO Model 140 Balance, sensitivity 10 mg/division, accuracy about 0.01 g in 10 kg.
- 1 5-kg SECO Model 140 Balance, sensitivity 2 mg/division, accuracy about 0.002 g at 1 kg.
- 1 Mettler Model K5 Direct Reading Balance, capacity 2 kg, sensitivity and accuracy 0.05 g at 2 kg.
- 1 Mettler 200-g Direct-reading Analytic Balance, accuracy 0.0001 g at 200 g.

b. Operation of SECO Balances

- (1) Always load and unload the balance with beam raised and pans arrested. Lower the beam gently; then release the pan arrests.
- (2) The best accuracy can be obtained with the magnetic dampers retracted by the method of equal swings. Take three or five swing readings. The balance point is shown as the midpoint between the average swings.
- (3) The magnetic dampers can be used to speed weighing with only a slight loss in accuracy. Adjust the dampers with a half-capacity load on each pan so that they bring the balance to rest in about three swings. Establish the no-load rest point before weighing, and weigh to that point rather than to the center of the pointer scale.
- (4) When finished with the balance, leave it in good condition for the next man.

c. Mettler Balance Operation. The Mettler Balances are direct-reading instruments. The zero point should be adjusted before each weighing, and the balance should be "spanned" at least daily. Spanning was accomplished by placing a full-capacity, known-value weight on the balance pan and observing the deviation from the known value. When weighings are made at midbalance capacity, calibration weighings should be made at that value.

2. Alloy Preparation and Casting

Molybdenum is soluble in uranium, but has limited solubility in plutonium. For this reason, and to minimize plutonium losses, which would occur at alloying temperatures, a U-Mo binary alloy was prepared which was cast and analyzed before remelting. The ternary alloy was then made by remelting the U-Mo alloy (which was deliberately made slightly rich in molybdenum) with additional uranium and the plutonium. The ternary-alloy melt was brought to the desired casting temperature and poured into a reusable, multiple-cavity, oxide-coated, carbon mold. Fuel-alloy bars, 2 x 0.202 x 10 in., were cast in the pilot production. The production casting dimensions should be of a length that will minimize return scrap. The castings were cleaned, inspected, and transferred to the core-plate machining operation. Return scrap was remelted and cast into usable core bars.

a. Melting and Casting Equipment. The binary and ternary alloys were each prepared in gloveboxed melting furnaces. Although located in different lines, the furnaces are essentially similar and have the same capacities: 9 to 12 kg of alloy. In large-scale production, the binary alloy should be prepared in large batches to reduce the number of melts and the amount of analytical work. The furnace for binary-alloy melting need not be gloveboxed. The ANL equipment used for binary-alloy casting is housed in Glovebox PF-9 and consists of the following items:

(1) Glovebox PF-9, 4 x 6 x 21 ft, helium atmosphere.

(2) Vacuum-induction furnaces, lift-bell type with induction-coil assembly in bell. The coils were solid tungsten within an insulator support. Power was coupled to the crucible through a tantalum susceptor around the crucible and to the upper part of the molds.

(a) Crucibles were AEC, V-2-type MgO with 3/4-in.-diam pour hole, a 1-in.-diam MgO stopper rod, and a crucible cover. A MgO pedestal and funnel supported the crucible assembly on the mold. An yttria-wash coating was used on all surfaces that contact the molten alloy.

(b) Book-type molds had four to six $0.205 \times 1.970 \times 10\frac{1}{2}$ -in., yttria-coated cavities. Molds were open-top, and metal dropped directly into them. The upper ends of the molds were inductively heated, the bottoms cooled by the furnace shell, and the sides thermally insulated.

(3) A 6-in. diffusion pumping system (for evacuating each furnace) consisting of diffusion primary and booster pumps and a 48-cfm mechanical backing pump.

(4) Power supply, 30 kW, 10,000 Hz, 0-220 V with tank capacitors outside the glovebox line.

(5) Controls and interlocks for furnace power supply and vacuum systems.

(6) Optical pyrometer, for metal temperature. Thermocouples were read by means of a potentiometer for mold temperatures.

b. Process Materials

(1) Crucibles. Two crucible materials have been used, yttria-coated carbon and magnesia. Both crucibles are of the standard V-2 size with bottom pour hole and tapered stopper rod. The carbon crucible served as its own susceptor. The MgO crucible required a tantalum susceptor to eliminate local heating from the charge and cracking. The capacity of the V-2 crucible was about 9 kg, but 5 or 6 kg is more easily charged. Boron content of crucible materials was <20 ppm.

(2) Molds. Molds were machined from National Carbon Company ATJ graphite or the equivalent fine-grained, high-density carbon. The molds consisted of several 7/16-in.-thick plates, each with a milled cavity and two end plates. Drawings of the cavity plate and end plates are shown in Fig. 10. The number of cavity plates used varied with the size of the melt. The top of each cavity plate is milled to form a runner basin, and two end plates are required to complete the mold and close the runner. Approximately 1200 g of alloy was melted for each plate cast.

c. Crucible and Mold Coating. The molds were thoroughly "dedusted" before coating. This may be done by using dry compressed-air blast or by heating to 300 to 400°C and "quenching" in boiling water. The intense boil at the surface removed loose carbon dust. The molds for U-Mo alloy were similar to those for the binary alloy, but they did not need to be precise. We used older developmental molds that were not of casting precision for binary casting.

ANL has successfully used, and is presently using, $<6\mu$ yttria suspended in ethyl alcohol for coating carbon crucibles and molds. The suspension was made in the ratio of 90 g of yttria per each 100 ml of alcohol. The dry powder was dispersed in the alcohol and allowed to hydrate for at least 15 hr before use. It was resuspended by stirring or shaking just before use. Other coating materials that have been successfully used are ThO_2 and ZrO_2 . Coating materials should not be used that contribute impurities limited in Section 6.5.1 of Specification PF-1600.

The carbon crucible and mold coating is done in the glove-box line. Both crucibles and molds were reused, but had to be recoated each time they were reused. The coating operation added significantly to the moisture content of the inert atmosphere and was done in a separate glovebox from those used for handling the alloy.

The crucible coating procedure is as follows:

(1) Prepare dispersion in the ratio of 90 ± 5 g of $<6\mu$ yttria to 100 ml of reagent-grade ethanol. Shake or stir for 10 min, and set aside for not less than 15 hr in a stoppered flask.

(2) Resuspend the yttria in alcohol by stirring or shaking just before use.

(3) Plug the crucible pour hole by means of a rubber stopper. Pour the yttria suspension into the crucible. Tilt and rotate the crucible so that the wash contacts all areas of the crucible cavity. Pour out the suspension. Remove the stopper, and drain in the vertical position.

(4) Paint the yttria suspension into the pour hole and on the bottom of the crucible by means of a camel's hair brush.

(5) Pour the yttria suspension into a cylinder, and dip the stopper rod. Drain in a vertical position.

(6) Allow the coating to dry for at least 2 hr without heat. The coating should be thin, and of a light-gray appearance, rather than a chalk white. If too thick, it will spall off after drying. Coated crucibles may be stored for a long period in a very dry atmosphere or in an oven at 100 to 150°C.

d. Mold Preparation and Coating Procedure. The molds were disassembled after each use. They were carefully cleaned and recoated with mold wash. The same yttria suspension was used on molds as was used for crucible coating, although similar results were attained with thoria coatings. A thin, uniform coating should be applied by brushing or pouring. Air-brushing can also be used, but the method described below gave better results.

The procedure is as follows:

(1) Lay the flat surface to be coated in a horizontal position. Pour the suspension onto the surface of the mold, and spread quickly with a flat camel-hair brush. At the same time, shake and tilt the mold plate to spread the coating evenly. Set the mold plate aside to dry in a horizontal position. When dry, the coating should have a light-gray appearance due to the underlying carbon. If it is chalk-white, the coating is too thick. If the molds are stored for 24 hr before use in an atmosphere of less than 30 ppm moisture, oven drying has not been necessary. The coating should be very smooth. Every detail in the coating will be replicated in the casting.

(2) Coat cavity sections of the mold by painting a thin, uniform coating.

(3) Coat the runners or tundishes in the same manner as the molds.

(4) Use the carbon crucibles and molds as many times as possible. The first casting made in a new mold is usually markedly inferior to those made subsequently.

e. Charge Calculation and Weighing. To calculate the alloy charges accurately, one must know the compositions of the feed materials. Charge calculations were carried to the number of significant figures justified by the data. If the composition data were available for four-place significance, the calculations were carried to five significant figures and rounded back to four significant figures. The "even-number rounding rule" was used for rounding 5's to the nearest even significant figure. The balance precision was 2 mg in 5.0 kg, and all charge weighing of plutonium was done to at least ± 0.010 g accuracy. ANL accountability records were balanced out to the nearest 0.1 g.

f. Furnace-charging Operations

(1) The stopper rod was carefully seated. Care was taken not to remove the coating from the carbon at the contact between the crucible seat and the stopper rod. With MgO crucibles, the stopper rod was twisted lightly together and the seating surfaces inspected. There must be complete seat contact, or the stopper rod seat will leak.

(2) The stopper rod was held vertically, and three or four fairly large pieces of uranium (or U-Mo alloy) were carefully placed so that they boxed equally against the rod and held it in position.

(3) When a coated crucible was used, care was taken not to scrape the coating while loading the crucible.

(4) The molybdenum pieces were added next for the binary melts, so that the molybdenum was near the bottom of the crucible. The balance of the uranium was added above the molybdenum. The purpose of this loading sequence was to cause the melting uranium to trickle down over the molybdenum, which was held down by the yet unmelted uranium. If the molybdenum floated into the dross, it would not dissolve. Pieces of approximately $1/8$ -in. (3-mm) size appeared best for alloying. Larger pieces dissolved slowly and required prolonged melt contact or high-temperature superheat contact. When molybdenum powder was used, a large portion of it appeared in the dross.

(5) In charging the ternary melts, the plutonium was placed on top of the U-Mo alloy or uranium. Plutonium melted first and achieved the maximum contact in trickling down through the uranium. The pieces of uranium, U-Mo alloy, and plutonium could be any convenient size above $1/8$ in. (3 mm). A certain amount of fines could be melted, but the losses increased as the proportion of fine material increased.

(6) A crucible cover was used to reduce splatter losses and radiation heat losses. The crucible cover also held the stopper rod in alignment after the charge had melted.

(7) The furnace was assembled as shown in Fig. 7. The furnace bell and induction coil were lifted by two screw jacks.

(8) The coated molds were assembled and bolted together and placed upon the mold support which rests upon the water-cooled furnace bottom. Porous Al_2O_3 tiles were placed around the molds for insulation. In ternary melting, the top of the molds extends into the induction field and supports the crucible pedestal and crucible. This establishes a temperature gradient of about 600°C from bottom to top of the $10\frac{1}{2}$ -in.-long mold assembly. This temperature gradient is required to obtain solid castings of the elongated tablet geometry. The midpoint temperature of the molds is near 300°C .

In binary-alloy melting, an insulating spacer was placed below the crucible, so that the mold temperature gradient was reduced to about 300°C , with the midpoint temperature near 240°C .

(9) After construction of the mold-crucible stack, the stopper-rod extension was attached to the stopper rod by means of a TZM alloy pin.

(10) The furnace bell and induction coil were carefully lowered. The stopper-rod gland was loosened so that it would slide smoothly down the stopper-rod extension. If the bell had to be raised, the stopper-rod extension was held down to prevent loss of the stopper-rod seal.

(11) A separate roughing pump was used to evacuate the furnace and to check for leakage. When the furnace pressure as indicated by thermocouple gages had reached near 0.1 Torr, the valve to the diffusion pumps was opened and the furnace rapidly evacuated to 10^{-4} Torr. The heating, melting, and casting cycle could now be started.

g. Binary-alloy Melting Operations

(1) Heating was started very slowly to outgas the molds, crucible, and metal charge. The pressure was not allowed to exceed 0.1 Torr at any time. When the pressure decreased to below 10^{-3} Torr, the power was increased to bring the temperature to 1100°C. This was done quite rapidly with the carbon crucible, but was done slowly with the MgO crucible. (The wall of the coated-carbon crucible should not be allowed to exceed 1500°C, to prevent the coating from being destroyed.)

(2) Melting occurred between 1100 and 1200°C. Outgassing and splatting were violent when too much power was used immediately after melting. The crucible temperature was held below 1200°C until the charge was fully outgassed.

(3) After the melt became quiet, the power was rapidly advanced to bring the temperature to 1450-1475°C, as read by optical pyrometer. The melt was held at this temperature for 20-30 min to allow dissolution of the molybdenum, and lowered below 1400°C for casting.

(4) Casting was accomplished by lifting the stopper-rod extension. The vacuum pumps were left on during pouring and cool-down to below 400°C.

(5) The furnace could be opened in about 2 hr after casting. Overnight cooling was preferred as it is much easier to handle the fully cooled furnace components.

(6) The molds were carefully stripped from the castings to avoid damage to the mold surfaces. Normally they required only a light brushing and recoating.

(7) A weight balance was run on the binary melts. The balance of feed material and product weight was one measure of the success of alloying. Dross and skulls should be less than 10 g per kg melted, and the feed and product balance should agree within 10 g.

h. Ternary-alloy Melting and Casting. The casting conditions, heating rates, time at temperature, superheat temperature, and casting temperature had to be reproduced as precisely as possible from melt to melt if composition control, homogeneity, casting dimensions, and casting quality were to be controlled.

(1) The crucible and mold preparation steps were as previously described for ternary-alloy melting and casting.

(2) The furnace setup differed only in that the top of the mold was positioned in the induction-coil field. The bottom of the mold was cooled. The resulting temperature gradient promoted directional solidification and eliminated the pipe defect that otherwise would occur.

(3) The furnace was evacuated to 10^{-4} Torr, and heating was started slowly. Crucible and mold outgassing occurred below 300°C . The heating rate was controlled to prevent the vacuum-gage readings from exceeding 0.1 Torr (where the diffusion pumps became ineffective).

(4) Outgassing was completed in about 20 min, and the vacuum dropped to below 10^{-3} Torr. The power was rapidly increased to a setting that would heat the crucible to about 1350°C (corrected for optical pyrometer error). The plutonium melts at about 630°C , and the U-Mo alloy at approximately 1160°C .

(5) The power was adjusted to heat the metal to 1380°C just before pouring.

(6) The power was then reduced and, as the metal cooled to 1280°C corrected optical-pyrometer reading, the stopper rod was lifted and the alloy was cast into the molds. Pouring at reduced power prevented the large power-factor change that would otherwise occur as the metal left the induction field.

(7) The power was shut off, and the furnace was allowed to cool under vacuum to below 400°C . Overnight vacuum cooling was preferred for best casting surface quality, but this was undesirable when more than one shift used the same furnaces. The cooling rate could be materially increased by admitting helium to the furnace as soon as the mold temperature fell below 600°C . The furnace could then be opened about 2 hr after casting.

(8) The furnace was purged with helium and brought to 1 atm pressure before opening. The bell and induction-coil assembly was raised, and the molds and crucible were removed from the furnace and placed in trays. (Care was taken to recover all of the furnace products, including any splattered alloy in the furnace base.)

(9) The crucible skull and dross could usually be loosened and removed by means of tongs. Beads of metal that stuck to the crucible walls and cover were scraped free. (The gamma surface dose rates go to quite high levels with reuse of the crucibles, and this frequently limits the number of times that they can be reused. The crucibles were surveyed by health physics personnel after each use and were handled according to their direction.)

(10) The molds were opened carefully to avoid damage to the mold surfaces or to the brittle castings. A casting sprue could connect several castings, and the castings had to be broken from the sprue. All the metal removed from the molds was placed in the cleanup tray, and the molds were carefully brushed out. Brushings from the mold were placed in a recoverable residue container even though they appeared to contain no metal. After being cleaned, the molds were set aside for recoating.

(11) The castings were brushed or wiped to remove surface-mold wash. The casting surfaces should have a matte-finished metallic appearance. All surface brushings were placed in recoverable residue containers.

(12) A preliminary inspection was made of the castings for conformity to specifications and drawings for casting quality-control purposes. Areas having gross defects were noted and, if small, these were cut out in the machining operation.

(13) All furnace products were transferred to Glovebox PF-7 for weight balance. Castings and metal residues were weighted to the nearest 5 mg. Normally the feed material-furnace weight balance should agree within 0.1%.

(14) Acceptable castings were transferred to the machining operation in Glovebox PF-5. Recoverable scrap was remelted using the following procedure:

i. Recovery Melting

(1) Sprues and solid-metal return scrap were fed back with virgin metal into the regular melts. These were charged into the melts at their calculated compositional values.

(2) Fine material from crucible scrapings, furnace cleaning, and other residues of questionable composition were accumulated and remelted in a batch that was sampled and analyzed. If the composition of this material was within specification, it was remelted and cast into fuel elements.

(3) A large percentage of the metal was recovered from skulls and drosses by crushing them down to a compact mass and melting them in a perforated-bottom crucible. The metallic portion drained away from the dross and was collected in a second crucible. This metal was sampled and analyzed before casting.

3. Core Plate Machining and Inspection

Core plates were made from the precision castings by machining. The castings were parted to form core slugs that were milled to width and chamfered to form core plates. The two larger surfaces of the slugs were left in the as-cast condition; however, all other surfaces and edges were machined.

a. Equipment. The equipment available for machining plates was as follows:

- 1 Glovebox PF-5, helium-filled, 4 x 4 x 18 ft.
- 1 Heald Machine Works heavy-duty, horizontal, back-gearred milling machine.
- 1 Arboga-Maskiner vertical milling machine, Model EM 825.
- 1 Mettler Model K7T direct-reading balance; capacity 800 g; sensitivity and weighing accuracy, 0.03 g.
- 1 Herman Stone surface plate, 2 x 2 ft.

Glovebox PF-5 also contains micrometers, angle plates, dial indicators, and an assortment of machinist's tools.

b. Inspection of Castings. The castings were inspected to determine their suitability as core-plate material. A 3/4-in. grid was drawn on one of the larger casting faces with a soft pencil so that one of the longer lines was centered on the casting face, and the shorter lines began 1/4 in. from the bottom of the casting. With the grid drawn, the following measurements were made:

(1) The thickness of the casting was measured by means of a deep-throat micrometer at every grid intersection.

(2) The width of the casting was measured across every lateral line on the grid.

(3) The usable length of the casting was measured by means of a scale.

c. Machining Techniques. Two techniques were available for machining ZPPR core plates from alloy castings: (1) room-temperature machining with carbide or carbide-tipped cutters and (2) hot machining with high-speed steel cutters. Combinations of these two techniques could also be used.

Room-temperature machining was performed as follows:

(1) The casting was clamped in a horizontal position in the small Arboga mill.

(2) One side of the casting was faced with end mill. The purpose of this step was to produce a locating side for parting.

(3) Four castings were fixtured in the large mill, each with the trimmed side against the top. A 0.025-in.-thick strip of aluminum was used as a pad between the casting and the fixture.

(4) Two 1/16-in.-thick x 3-in.-diam slitting saws were used on the arbor. The cutters were spaced approximately 0.030 in. farther apart than the core length desired. This excess length reduced rejection due to edge chipping.

(5) The castings were parted with a cutter speed of 100 rpm and a table travel of about 0.0075 in. per revolution. Since the cutter has 12 teeth, the depth of cut was slightly less than 0.0006 in. per tooth.

(6) The plates were returned to the vertical milling machine for finishing. The ends were milled by a carbide-edged, side-cutting end cutter. The plates were weighed, and the amount to be removed from the second side was calculated to achieve the upper limit of the weight specification after chamfering.

(7) All edges of the core plates were chamfered by means of two angle cutters and a spacer mounted on a single arbor. Carbide-edged cutters were used for room-temperature machining. High-speed-steel cutters were used for hot machining.

(a) Hot machining involved the same steps as room-temperature machining, but with the following differences of procedure:

1) The aluminum pads were not needed.

2) The core plates were heated to above 175°C before machining was started.

- 3) It was necessary that expansion of the core plate be considered and that all machining be done at the same temperature.
- 4) The core plates were clamped loosely until an equilibrium temperature was reached, but were clamped tightly for machining.
- 5) Slitting saws used for the cutoff operation were of the side-cutting type to prevent binding.

(b) Hot machining had the following disadvantages:

- 1) Twenty to thirty minutes were required after setup to bring the castings, milling fixture, and arbor to steady-state temperatures so that the dimensions could be reproduced.
- 2) The precision of hot machining was not as good as that obtained by milling with carbide cutters at room temperature.
- 3) There was an added hazard of burns to rubber gloves and the machinists hands.
- 4) The overall machining time cycle was impractically long.

d. Weight Adjustment. The experimenters requested that the variability of fissile plutonium content of the cores in a given size element be not greater than 1.4 w/o. The compositional and core weight specifications were set to produce this value.

(1) The core plate weight was adjusted to within $\pm 0.3\%$ by milling a variable amount from the width of the core plate.

(2) The core plate was weighed after the ends and one side were milled. The amount to be removed was calculated.

(3) The second edge was milled by the amount required to adjust the plate to a specified weight.

e. Dimensional Inspection. The following procedures were used on pilot-lot production to characterize the product. (Note: ANL will accept go/no-go gage procedures for all core dimensional inspection except core length in ZPPR production.)

(1) Casting thickness was measured by micrometer at 3/4-in. grid-intersection intervals.

(2) Length was measured by micrometer at the centerline and at $3/4$ in. on either side of the centerline.

(3) End and side squareness and parallelism were measured by means of a dial-indicator perpendicularity gage.

(4) Templates were used to gage chamfer.

(5) Core-plate twist and bow were inspected for by passing the core plates through a parallel-plane gage. The parallel planes were separated by 0.206 in., plus the plate length divided by 2000. The planes were sufficiently large that the entire core plate was contained between the planes at one position.

f. Sampling for Chemical Analysis. The castings are sampled at $3/4$ in. below the top, at the center, and at the bottom corner as shown by Drawing PF-1602 of the specification. Samples were submitted for plutonium-isotopic, minor-element, carbon, and oxygen analysis as well as for uranium, plutonium, and molybdenum assay. Solid samples gave better results than drillings. The following method was used to prepare samples from each melt:

(1) Thin slices were cut from the casting by means of the slitting saws. The slices were cleaned up by end milling.

(2) Clean pieces weighing 0.3-0.6 g were broken from the slice by means of pliers.

(3) The pieces were weighed quickly to four significant figures and placed in a glass vial. Plastic was not used; this weight had to be accurate.

(4) The vial was marked.

(5) The samples to be analyzed were sent to the chemists.

(6) Duplicate samples were vacuum-sealed in glass ampules.

g. Machining Accountability. All the alloy entering the machining operation was accounted for. This was done as follows:

(1) The castings were weighed to the nearest 0.01 g before they were machined.

(2) The products of machining were separated and weighed. The good cores, chemical samples, remelttable material, and the fines produced were each weighed to the nearest 0.01 g.

(3) The weight of products was compared to the weight of raw materials. If an unexplainable loss was observed it had to be found before the next batch of castings could be machined. When a balance was obtained, the products were sent on for further processing.

4. Jacket Manufacture

The jackets for ZPPR fuel elements were made from Type 304L stainless steel sheet and wire stock. The materials and the means of inspecting them are described in detail in Specification PF-1600. The process described here includes the making of cylindrical and rectangular tubing.

A jacket consists of three pieces, a sleeve and two end plugs. The sleeves were made from sheet metal by (1) rolling and welding of sheet to form cylindrical tubing, (2) die-flattening of the cylindrical tubing, (3) forming of the narrow sides in a press brake, (4) annealing, and (5) final sizing by stretching.

The following operations were used in pilot-lot jacket-sleeve manufacture:*

a. The 0.015-in. stainless steel was sheared into rectangular pieces 9.5 x 4.212 in.

b. The long ends were preformed for rolling, and the sheet was rolled into a 1.355-in.-diam x 9.5-in.-long cylinder.

c. The rolled piece was placed over a mandrel in a seam-weld fixture. The piece was clamped over its entire length. The copper chills were placed as close to the weld area as possible to obtain the required chilling effect.

d. The tube was seam-welded with a power-traversing, controlled-arc, TIG welding machine. The weld was made at 12 V and 10 Amps. Head speed was 30 in./min, and the gas flow was 30 cfh helium plus 5 cfh argon. No filler rod was used.

e. The cylindrical tubing was die-flattened in a press. The tube was placed in a shaped die with the weld up. A flat strip, 0.206 x 1.843 x 12 in., was placed inside the tube to hold size.

f. The 90° bends were formed in a press brake by two forming dies and a flat strip.**

*Production jacket sleeves are being manufactured from commercially drawn, welded tubing made from vacuum-melted Type 304L stainless steel.

**This step was eliminated from the ZPPR jacket-sleeve production procedure (see Section I-J, page 34).

g. The tubes were stress-relieved at 1450°F for 45 min in vacuum. The annealed tube was relatively soft and was free of oxide scale.

h. The tubes were placed in a die and stretched to final dimensions. The stretching was done by means of three wedges that move the narrow sides of the tube out against a boxdie. The die cavity measured 0.243 x 1.996 x 9.5 in.

i. The sleeves were cleaned by vapor degreasing, followed by detergent washing and air drying.

j. The jacket sleeves were cut to length with an abrasive cut-off wheel, and the ends were finished by milling and grinding. The cut ends were deburred.

The end plugs were made from drawn rectangular wire by the following steps:

a. The wire was cut to length plus 0.030 in. with an abrasive cutoff saw.

b. Thirty wires were clamped in a fixture, and their ends were finished by milling.

c. The end radii were formed by grinding the 30 wires in the fixture.

5. Fuel-element Assembly and Welding

The components supplied to the fuel-element assembly were core plates, jacket sleeves, and end plugs. The steps of fuel-element assembly were (1) welding of bottom plug in sleeve, (2) testing weld, (3) inserting core plate in sleeve, (4) evacuating and back-filling assembly with inert gas at 1/2 atm, (5) welding second end plug, (6) contamination survey, and (7) decontamination. Disposable loading funnels were made from 0.004-in. aluminum foil. These were used to protect the lip of the jacket from plutonium contamination during loading.

a. Equipment. The following equipment was used for fuel-element assembly and welding:

(1) Glovebox PF-17, helium-atmosphere-filled, size 4 x 6 x 18 ft.

(2) Vacuum welding chamber, mechanical roughing pump, inert-gas purge, and pressure regulator. Access to the welder was by a removable front panel.

(3) Motor-driven, weld-torch traversing mechanism and work positioner within the vacuum chamber.

(4) Welding-chill assembly.

(5) 200-Amp dc welding machine, Vickers Incorporated, with magnetic-amplifier current control and high-frequency starter.

(6) ANL-constructed programmer for weld-torch traversing mechanism, weld machine, and inert gas supply.

(7) Electrolytic marking machine, stencils, Formula SC-44 electrolyte, and neutralizer manufactured by Electromark Corporation, 2093 East 19th Street, Cleveland, Ohio.

The vacuum welding chamber was a 24 x 16 x 12-in. stainless steel box containing the weld-torch traversing mechanism and an adjustable position fixture for holding the welding chill. The chamber was evacuated through a high-efficiency filter by means of a laboratory-type (Welch) vacuum pump. Argon and helium gas are supplied through a mixer-flow regulator and introduced to the chamber through the electrode holder. A regulated dynamic partial pressure of inert gas was maintained on the box by pumping with a controlled flow of gas supplied to the welding torch.

The weld programmer controlled the following functions: (1) evacuation of the chamber, (2) chamber purge and preflow of weld gas to torch, (3) high-frequency ionization, (4) arc start, (5) torch drive, (6) weld amperage, (7) arc cutoff, and (8) postweld gas flow.

The same equipment was used for welding both ends into the jacket sleeves. In production of the ZPPR fuel elements, separate and less sophisticated equipment can be used to weld the bottom plugs into the jackets. Bottom end plugs were welded into jacket sleeves with a steel dummy in the place of the core, and the weld was made at atmospheric pressure instead of reduced pressure. (A bubble test or helium test can be given this weld and the seam weld at the manufacturer's option.)

Loading funnel blanks were stamped from 0.004-in. aluminum foil. The blanks are wrapped on a form, spot-welded, and die-formed. The funnels had to be accurately formed because of the close clearance between the core and jacket.

b. Contamination-preventing Technique. Prevention of contamination is the main problem of assembly. Each contact that metallic surface makes with plutonium transfers sufficient material to the clean

surface to be detected in a contamination survey. Because one-millionth of a gram of plutonium yields 143,000 counts per minute, any transfer of material usually exceeds the acceptable limits of contamination. Plutonium may also be transferred by dust, by tools, and by evaporation and redeposition from the arc. Dust contamination was easily removed. Contamination by rubbing or abrasion was usually difficult to remove and required the removal of a surface layer. We have not been successful in removing contamination deposited in the welds. Plutonium in the welding arc was particularly objectionable because a complete decontamination of chills, welding machines, and adjacent equipment was required. Such an incident would require many man-hours.

Contamination-preventing techniques required the following:

- (1) Thorough cleaning of the fuel plates, with removal of as much oxide and particulate material from the surfaces as possible.
- (2) Handling fuel plates in foils and sealed plastic envelopes after cleaning. (Note: The core plates should not be stored for longer periods in contact with plastic, which breaks down under alpha radiation.)
- (3) Assembly of the fuel elements in an area of low contamination. Foil or plastic should be used on work areas for ease of decontamination.
- (4) Understanding of the problem by the operator, and development of a deliberate technique for contamination prevention.

c. Assembly and Welding Operation

- (1) The inspected, cleaned, weighed, and identified core plates were wrapped in aluminum foil and sealed from the contaminated glovebox system in individual vinyl envelopes. (Note: Batch-limits for vinyl-wrapped plutonium may be reduced.)
- (2) The inspected jacket sleeves with one end plug welded in place were electrolytically etched with an identification number. This operation consisted of cleaning the surface of the jacket with acetone, application of a numbering stencil and electrolytic marker, application of neutralizer, and wiping with polarized oil.
- (3) Four jacket sleeves were placed in the welding chill. The aluminum funnel was placed in each jacket sleeve, and the exposed chill parts were covered with aluminum foil or plastic.
- (4) The tray of core plates was brought to the loading station, a core plate was selected, and the wrapper was carefully removed. The wrapper was consigned to dry active waste.

(5) The core plate was picked up by means of a "contaminated" loading tong and slowly inserted into the jacket. If insertion was too rapid, contamination was swept out of the jacket by the displacement of the gas within the jacket. The tongs were placed in a "contaminated-tool tray."

(6) The funnel was removed, and the core was pressed down against the bottom plug by a "contaminated" push rod. The push rod was placed in the "contaminated-tool tray."

(7) The top end plug was picked up by an "uncontaminated" tong and inserted into the top of the jacket sleeve. The end plug had to fit tightly. It was not forced against the core at this time, but was pressed into place by the clamping action of the chills.

(8) Each jacket was loaded as described above. The tack weld chills were placed, and the weld-fixture lock bolts were locked. The end tack welds were made.

(9) The longitudinal-weld chill bar was installed.

(10) The welding-chill assembly was placed in the weld chamber, and the positioning fixture was clamped. Alignment was visually checked.

(11) The first longitudinal welds were made at atmospheric pressure. The chamber was closed and evacuated to <0.2 Torr for 5 min. It was then purged with 60 w/o He-40 w/o Ar gas mixture at 6 psia.

(12) The weld was made at the sleeve-end plug interface by the following program:

(a) High-frequency arc start on chill starting block.

(b) Weld current on.

Volts:	12.0 ± 0.2
Amperes:	38 ± 2
Gas flow:	15 cfh helium, 10 cfh Argon
Electrode:	0.040-in.-diam thoriated tungsten
Electrode gap:	0.025 ± 0.003 in.
Traverse rate:	3.5 in./min

(c) Weld current cut off on chill insert block.

(13) After welding, the chamber was brought to glovebox pressure and opened. Filter-paper wipes were made on each weld. These were transferred from the glovebox and monitored.

(14) If the wipes did not show contamination, the fixture was removed from the welding machine and the elements were removed, visually inspected, and counted by means of a scintillation counter. If no contamination was detected, the fuel elements were removed from the glovebox and inspection was continued on open benches.

6. Fuel-element Inspection

Fuel-element inspection consisted of:

- a. Helium-leak detection and bubble testing for large and small leaks.
- b. Dimensional gaging, parallel-plate gaging, and length gaging.
- c. Weighing.
- d. Alpha counting.
- e. Neutron counting on a batch sample basis.

The steps of final inspection are fully described in Specification PF-1600, Sections 13, 14, 15, 16, and 17.

PART III

MARK-I PLUTONIUM FUEL ELEMENT ALPHA AND NEUTRON MONITOR

The Mark-I Plutonium Fuel Element Alpha and Neutron Monitor was designed for the simultaneous determination of the level of fixed-alpha contamination and neutron emission from a plate-type fuel element. The instrument was developed out of a need for a standard counting geometry for zero-power reactor fuel elements which had just been fabricated. Portable instrumentation can be used for these determinations, but the accuracy, sensitivity, and reproducibility of the determinations are not adequate.

The detector system consists of an alpha portion and a neutron portion. The system is positioned in a 30.5-cm cubicle aluminum shell, filled with paraffin moderator.

The alpha-detection system consists of two 32 x 14.5 x 1-cm, rectangular, gas-proportional chambers, sandwiched 1.25 cm apart. The alpha-detector window material is 0.00635-mm aluminized mylar, with a density of 0.85 mg/cm². The anode is a 1-mm-diam tungsten wire, strung on Teflon insulators in a common plane, and arranged so that the detector has effectively 10 central wires.

The counting gas used is 90% argon and 10% methane. The gas, at a positive pressure, flows through the detector, regulated by a flow-metering valve and observed by an oil-filled flow indicator.

The alpha detectors are operated at a voltage on the plateau, which maximizes the alpha-counting efficiency, while minimizing the sensitivity to background neutron and gamma radiation. The output signal of the alpha detectors is summed, preamplified, and fed into an ANL nine-decade scaler. A preset timer regulates the counting duration.

The neutron-detection system consists of four ¹⁰BF₃ tubes (Nancy Wood type NAG-10-2). These detectors are placed in the paraffin moderator, two on each side of the alpha detectors. They are placed 17.5 cm apart, with 3.8 cm of paraffin moderator between the fuel element and the tubes. The signal pulses are preamplified, summed, and fed into a nine-decade scaler.

The ¹⁰BF₃ tubes are operated at a voltage on the plateau below the gamma threshold level.

A block diagram of the alpha-particle and neutron-detection system is shown in Fig. 28.

Fig. 28. Block Diagram of Alpha-particle and Neutron-detection System

The fuel elements are carefully positioned into the fuel-element holder and inserted for counting into the frame between the alpha detectors, as shown in Fig. 29.

Discrimination between the alpha particles and the pulses from the neutron-proton reaction in the gas-proportional alpha detectors is accomplished with a thin, aluminum, movable alpha shield, which is inserted between the alpha detectors and the fuel element. Two counting orientations are then produced, one with and one without the alpha shield. The difference between the two determinations is due to the fixed alpha contamination on the fuel element. Applying appropriate yield factors gives the total surface alpha contamination in disintegrations per minute (dpm).

350-966

Fig. 29. Alpha-particle and Neutron-detection System

Each element is counted for a long enough period to yield a 3% standard error for the alpha determination and a 0.5% standard error for the neutron determination.

The alpha-detection efficiency is determined by carefully positioning a ^{239}Pu standard in the fuel-element holder. Considering the geometry of the system, the plutonium alpha particle range, and an equally distributed isotopic source on the loading end of the fuel element, the alpha counting efficiency ranges from 26 to 29%.

Neutron-detection efficiency is determined for each size fuel element by calculating the neutron intensity (in neutrons per second) emitted from the elements using the plutonium weight, isotopic, impurity, and neutron-yield data. The efficiency of the neutron-detection system ranges from 0.40 to 0.45% and is a function of the fuel-element size.

The Mark-I fuel-element monitor has been a reliable quality-control instrument. It can determine the fixed alpha contamination on the surface of reactivity-coefficient fuel elements while simultaneously determining the neutron-emission rate. Experimental data have proved that the alpha-contamination level can be determined ± 20 dpm with a lower detection limit of 20 dpm. Neutron intensities from the fuel elements can be determined $\pm 3\%$ experimental error, which from a typical 4-in. ZPPR element would be ± 360 neutrons/sec.

PART IV

ANALYSIS OF MANUFACTURE COSTS

The following feasibility and cost analysis was made, based upon the manufacture at Argonne National Laboratory of ZPPR fuel elements containing 1100 kg of plutonium for delivery by April 1, 1968.*

A. Analysis of the Requirement

To ensure 1100 kg of plutonium, fabricated into 3900 kg of U-28.4 w/o Pu-2.5 w/o Mo alloy ZPPR fuel elements by April 1968, part of the production must be done in AEC facilities. Table III shows the distribution of elements by size and weight of alloy that must be fabricated in 12 months' production to meet all, 50, and 25% of the March 1968 fuel-element requirement.

TABLE III. Yearly, Monthly, and Daily Output Rates for
100, 50, and 25% of ZPPR Fuel to March 1968

Nominal Size, in.	Core Weight, g	Total Requirements		50% of Requirements		25% of Requirements	
		No. of Elements	Alloy Weight, kg	No. of Elements	Alloy Weight, kg	No. of Elements	Alloy Weight, kg
4	438	1080	473.0	540	236.5	270	118.3
5	553	1620	895.9	810	447.9	405	224.0
6	668	1080	721.4	540	360.7	270	180.4
7	782	1080	844.6	540	422.3	270	211.1
8	900	1080	972.0	540	486.0	270	243.0
Totals		5940	3906.9	2970	1953.4	1485	976.8
Pu @ 28.4 w/o			1109.5		554.3		277.4
Avg Monthly Output for 12 months		495	325.6	248	162.8	124	81.4
Pu @ 28.4 w/o			92.4		46.3		23.2
Daily Output, 16 days/month		33	21.8	17	11.0	9	5.4
Pu @ 28.4 w/o			6.2		3.1		1.5

Assuming a criticality limit of 2.9 kg fissile plutonium (3.3 kg of 11.5% ^{240}Pu plutonium) and a conservative yield of 65% per casting batch, the total requirement necessitates four melts per day, 50% of the requirement necessitates two melts, and 25% of the requirement necessitates one melt per day. This is based upon four casting days per week, with one-day slack for equipment maintenance or scheduled difficulties.

B. ANL Capability

This study and cost estimate are based upon the assumption that 50% of the March 1968 fuel is fabricated in a single shift in Building 350 at ANL, and that it is fabricated concurrently with the development and

* Although the fuel-element production was not carried out at ANL, these plans and cost estimates were made on the assumption that such an effort might be required.

fabrication of LMFBR irradiation test specimens and other requirements for the Laboratory's experimental programs. Furthermore, plutonium allocated for the experimental program must be kept separate from that for ZPPR. This is accomplished by assigning separate glovebox lines to ZPPR fabrication, and by keeping all fuel materials, products, and residues completely separate.

Additionally, staff, technician, and supporting responsibilities for the ZPPR fuel fabrication should be separated from other Building 350 programs, except for supervisory authority necessary for safe operation of the facility. This requires an increase in personnel to the extent that experimental fuel, irradiation test specimens, and zero-power reactor fuel can be fabricated simultaneously. The additional personnel required to fabricate ZPPR fuel elements would be two staff people, fourteen mechanical technicians, three machinists, two inspectors, and one clerk-punch card machine operator. A Special Materials clerk, a Special Materials technician, and a Health Physics technician will also be required full time for zero-power fuel fabrication. Argonne National Laboratory will not undertake fabrication of zero-power reactor fuels unless these additional people can be made available for the work. In the past, it has been necessary to obtain machinists and technicians by assignment from other divisions as well as from Metallurgy Division.

C. Fabrication Plan

The flow diagram for manufacturing ZPPR fuel elements is shown in Fig. 27. All operations not involving plutonium fabrication or handling should be performed outside of Building 350. These operations include:

1. Machining of graphite molds.
2. Preparation of depleted U- 3.70 ± 0.10 w/o Mo alloy, and cutting the alloy to charge-size pieces.
3. Preblending of plutonium to 11.5 ± 0.5 w/o ^{240}Pu (to be done at Isochem, Incorporated).
4. Manufacturing and inspection of jacket components, including welding of jacket bottom plugs.
5. Making of aluminum-alloy foil funnels.

All operations involving plutonium should be performed in Building 350. These include ternary alloying, casting, machining, assembly, welding, and inspecting the fuel elements.

1. Glovebox Lines

The glovebox lines were selected for minimum interference with other work, for their immediate availability, and for minimum need for modification. These glovebox lines could be made ready for quantity fuel production in two to three months after program authorization.

Figure 30 shows existing glovebox lines that can be made available, with some rearrangement, for ZPPR fuel production. With the exception of PF-17, these lines are not presently used for LMFBR fuel development programs. It is proposed to consolidate assembly, sodium filling, and welding of EBR-II fuel rods in Glovebox PF-25, thus freeing Glovebox PF-17 for ZPPR fuel-element assembly and welding. The lines proposed for ZPPR fuel fabrication are equipped as follows:

a. PF-8 Alloying and Casting. Glovebox PF-8 is 21 ft long x 6 ft high x 4 ft wide. The two existing centrifugal casting furnaces are modified by addition of a 10-in.-deep flanged collar and false furnace bottom. The collars fit between the lift bell and rotor bell and accommodate a 14-in.-deep mold assembly. The rotors and rotor housing are left in place, so that the furnaces may be easily returned to their centrifugal casting functions. New internal molybdenum or tungsten induction-coil assemblies are required to replace the existing induction coils, and spare coil assemblies should be provided. The existing vacuum pumps and induction power supply are used.

The glovebox is also provided with Mettler P-3 and P-10 balances for charge weighing and casting materials balance.

b. PF-10 Machining and Core-plate Inspection. The 21-ft-long x 8-ft-high x 4-ft-wide glovebox annex to the extrusion-press line is cleared out and used for core-plate machining and inspection. This same line is equipped with tools and gages required for the destructive testing and sampling of fuel elements received from a commercial vendor.

The line is equipped with three vertical bench mills for edge milling and chamfering. A 30-in.-diam bag ring on the overhead transition between the PF-10 annex and press line facilitates the installation of the bench milling machines in this contaminated glovebox. Glovebox PF-22 is moved to a location east of PF-10 annex and connected to it by means of a window-section transition. This glovebox houses a Heald Milling Machine similar to one in PF-5 that was previously used for plate cutoff.

c. PF-17 Fuel-element Assembly and Welding. Glovebox PF-17 is set up for assembly of welding fixtures, loading of fuel-alloy cores, preliminary contamination counting, preliminary welding, and reduced-pressure

FUEL FABRICATION FACILITY

(BUILDING 350)

First Floor Plan

Fig. 30. Location of Proposed ZPPR Fuel-element Manufacturing Operations

seal welding. The glovebox, 16 ft long x 6 ft high x 4 ft wide, is presently equipped with a glovebox annex for isolation of high-contamination risk operations. The present reduced-pressure welder was designed for smaller ZPR-3 plates and is not efficient for the larger ZPPR fuel plates.

A new welding chamber is installed at the west end of Glovebox PF-17 and attached by means of an existing 20-in.-diam port. At least eight welding fixtures are provided that can be accommodated to any of the five sizes of core plates. Assembly, loading, welding, vacuum, seal welding, alpha counting, and seal-out are done at six progressive stations in the glovebox line.

d. PF-18 Counting, Decontamination, Leak Detection, and Final Inspection. Glovebox PF-18 is an air-ventilated glovebox in which the welded fuel elements are brought in sealed vinyl envelopes for preliminary counting and decontamination if required (if possible). Once decontaminated, the elements are handled in air where they are leak-detected, gaged, and neutron-counted. New leak-detection chambers are required. The existing neutron counter is used. Gages are required for length measurement, and go/no-go gages for thickness, width, parallelism, and perpendicularity gaging.

e. Radiography. X-ray equipment is available for inspection of core and jacket fit. The estimated costs of equipment are as shown in Table IV.

TABLE IV. Estimated Cost of Equipment to Fabricate 250 ZPPR Fuel Elements per Month

Identification No.	Quantity	Item	Design Time, hr	Cost		
				New Equipment	Installation	Existing Equipment
PF-8	1	Glovebox				\$ 56,412
	2	Centrifugal casters				36,735
	2	Vacuum systems				5,707
	1	Power supply, 10,000 Hz				13,900
	2	Furnace modification	40	\$ 4,800	\$ 400	
	4	Induction-coil assemblies	20	8,800		
	1	Mettler P-10 13-kg balance		1,100 ^a		
	1	Mettler P-3 5.5-kg balance		860 ^a		
	2	Balance enclosures and inertia blocks	10	800		
			70	\$16,360	\$ 400	\$112,854
PF-8 Subtotal						
PF-10	1	Existing glovebox				\$ 50,000
	1	Glovebox PF-23			\$ 2,000	23,000
	3	Arboga vertical milling machine		\$ 3,800 ^a	1,200 ^a	
	3	Special bases		400 ^a		
	3	Motorized tables		1,500 ^a	300 ^a	
	5	Parallel plane gages	20	900		
	5	Go/no-go length and width gages	20	1,000		
	2	Perpendicularity gages	20	800		
	1	Mettler P-1200 1.2-kg balance				
		0.005-g precision		700		
	1	Balance enclosure		250		
	1	Inertia block		81		
	1	Cutoff fixture	40	1,200		
	3	Edge mill fixtures	20	900		
PF-10 Subtotal						
			120	\$11,531	\$ 3,500	\$ 73,000

TABLE IV (Contd.)

Identification No.	Quantity	Item	Design Time, hr	Cost		
				New Equipment	Installation	Existing Equipment
PF-17	1	Glovebox				\$ 51,272
	1	Chamber and vacuum system	80	\$ 5,800 ^a	\$ 2,500 ^a	
	2	X-Y welding machines	160	6,000 ^a		
	8	Welding fixtures	40	10,800		
	1	Scintillation counter		800 ^a		
	6	Positioning stations		1,200		
	1	Welding controls	80	1,800		4,000
	1	Electroetch marker		200		
	1	Vacuum deduster	20	400		
	1	Welch pump		350 ^a		
	1	Power brush and housing with filter collectors	40	400		
PF-17 Subtotal			420	\$27,750	\$ 2,500	\$ 55,272
PF-18	1	Glovebox				\$ 58,673
	1	Alpha, neutron counter				2,800
	1	Ultrasonic cleaner		\$ 450 ^a		
	1	Abrasive cleaner		1,240 ^a		
	6	Nozzles		240	\$ 800 ^a	
	1	Chamber auxiliary		250		
	2	Work stations		150		
	1	Filter chamber	10	450		
PF-18 Subtotal			10	\$ 2,780	\$ 800	\$ 61,473
<u>Other Equipment</u>						
	2	Dielectric sealers		\$ 1,800 ^a		
		Leak-detection equipment				\$ 4,800
	2	Impregnators	20	500		
	2	Vacuum leak test chamber w/pump	60	1,800		
	2	Bubble testers		500		
	1	Mettler P-1200 balance		700 ^a		
Other Equipment Subtotal			80	\$ 5,300		\$ 4,800
Total PF-8			70	\$16,360	\$ 400	\$112,854
Total PF-10			120	11,531	3,500	73,000
Total PF-17			420	27,750	2,500	55,272
Total PF-18			10	2,780	800	61,473
Total other equipment			80	5,300	-	4,800
Total			700	\$63,721	\$ 7,200	\$307,399
Total Contingency and 1959-1967 Equipment Increase				20,000	5,000	66,011
Total Estimate plus Contingency				\$83,721	\$12,200	\$373,410
Total Estimated Equipment and Installation						\$489,331
This job only, Expendable and Tooling Items				\$61,211	\$ 7,400	
^a Capital equipment items				\$24,800	\$ 4,800	

2. Fabrication Process

The process for fabricating ZPPR fuel elements generally follows that outlined in Part II of this report, but with the following modifications in detail: The casting-batch limit is 11 or 3.3 kg of 11.5% ²⁴⁰Pu plutonium. Once the plutonium is alloyed, the U-28.4 w/o Pu-2.5 w/o Mo alloy has a criticality batch limit of 35 kg of the alloy containing 10 kg of plutonium. This increase in batch limit takes credit for the density reduction of fissile plutonium by ²⁴⁰Pu, uranium and molybdenum. The ²⁴⁰Pu and ²³⁸U capture cross section is not included in the criticality-limit increase, but it provides an additional safety factor.

a. Ternary Alloying and Casting. The U-Mo alloy, plutonium, and a small quantity of uranium and return scrap are weighed out in correct proportions to make a 10-kg U-28.5 w/o Pu-2.50 w/o Mo alloy. The uranium is added to adjust for a slight but deliberate overage of molybdenum in the binary alloy. An excess of 0.4 w/o of plutonium is added to compensate for selective dressing. Yttria-coated MgO crucibles are used.

The mold assembly has six 13.0 x 1.980 x 0.206-in. cavities, a turndish, and a runner. Each casting weighs 1.35 to 1.40 kg; the sprue and heel, about 1.5 kg. The 12.5- to 13.0-in. casting length was chosen so that the two core plates can be cut from each casting: two 6-in. plates, a 5-in. plate and a 7-in. plate, or an 8-in. plate and a 4-in. plate. Molds will be National Carbon Company ATJ graphite or Basic Carbon Company Type 18 carbon.

Four technicians are required to make 12 castings per day. Both furnaces in Glovebox PF-8 are required. Table III gives the time analysis for casting.

b. Machining, Inspection, and Cleaning of Core Plates. One long side of each casting is end-milled to provide a straight reference edge. Four castings are clamped in a fixture and parted into eight core-plate sections by means of multiple, carbide milling saws. A slice is taken from the bottom, center, and top of one casting of each melt, for analytical and metallurgical evaluation. This casting is cut into two 5-in. core plates, the size in greatest demand.

After being parted, the core plates are passed to the three vertical mills. The first mill is set up to mill and chamfer the ends, and the second to mill and chamfer the remaining sides. The core plates are then weighed and thickness-gaged. Any face protrusions are removed by the third mill. The core plate is adjusted to specified weight by finish-milling and chamfering the side that was first rough-milled.

Core plates are gaged for length, width, side parallelism, and perpendicularity of adjacent surfaces. The finished core plates are vigorously brushed by means of a fine wire brush and vacuum-cleaned to remove loose particles. Core-plate machining and inspection take 1.3 hr per core plate or 32 hr per batch of 24 core plates. If the yield is reduced to 18 core plates at this time, the time allowance should be 24 hr per day. Assuming 6 hr per day effective, three machinists, and one inspector are required.

3. Fuel-element Assembly

The jackets are prepared outside the glovebox. Each jacket is gaged by inserting a maximum-size dummy core gage. The jacket is prenumbered with the core-plate number and cleaned by washing in solvent acetone. Cleaned jackets are heat- or vacuum-dried.

Control of contamination is the problem of fuel-element assembly. Contamination is controlled by cleaning as much oxide and dust from the surfaces as possible immediately before the core plates are loaded, by handling the core plates with special tools, and by protecting all surfaces of the jackets and fixtures from contact with the plutonium. The jacket opening through which the core plates are loaded is protected by a disposable aluminum-foil funnel.

The jackets are loaded directly into the welding fixtures at the first station in Glovebox PF-17. Exposed surfaces of the fixture are protected by strippable tape, and formed aluminum funnels are inserted in the jacket opening.

The assembled fixture holding four jacket sleeves are passed to the core-loading station. The cores are cleaned by vacuum just before loading to remove any loose particles and individually weighed. A core plate is inserted into each of the four jackets, the loading funnel carefully removed, and the end plug inserted. Each sleeve edge and plug are wiped with a cotton-tipped swab, and the swab is counted by means of a scintillation counter. If the counts are satisfactorily low, the tape is stripped from the welding fixture, which is transferred to the first welding station.

4. Welding

At welding station No. 1, the finger bar chill is installed. The fixture is transferred to welding station No. 2 where the two end welds and one side weld are made. At station No. 3, the finger-clamp chill is removed and the welds are visually inspected. If the welds are satisfactory, a bar chill is installed on the opposite side to that previously welded. The fixture is placed in the chambered welder at station No. 4. The chamber is evacuated and backfilled with welding gas to a pressure of 6-7 psia, and the four fuel elements are seal-welded.

The fixture is disassembled, and the core plates and all fixture surfaces are scintillation-counted at station No. 5. (The more sensitive gas-proportional counter does not function in an atmosphere of helium containing varying amounts of argon gas.) The welds are visually inspected. Satisfactory plates are dielectrically sealed from the glovebox line in vinyl envelopes at station No. 6.

One technician inspects, cleans, and marks jacket components. Two technicians are required for assembly and core-loading operations, and two technicians for the welding operations.

5. Alpha, Gamma, and Neutron Counting

The core plates are transferred to Glovebox PF-18 in sealed vinyl envelopes. The envelopes are removed, and each surface of the core plate is vigorously wiped by a standard counter filter moistened with ethanol. The filter papers are dried and counted in a proportional chamber counter. Each surface of the fuel plate is also counted by means of an Eberline PAC gas-proportional counter.

If the "wipe" count is more than 10 dpm or the count from any surface is greater than 200 dpm, an attempt is made to decontaminate the fuel element. Several methods are available for decontamination including scrubbing in a chelating detergent, polishing with crocus cloth or abrasive, abrasive blast cleaning, and electropolishing. Surface contamination can usually be removed. Contaminated welds usually cannot be decontaminated.

When the element is decontaminated it is transferred to an integrating alpha-particle and neutron counter. This consists of a paraffin "pig" containing a slot for the fuel element and four $^{10}\text{BF}_3$ neutron-counter tubes. Two large gas-proportional counter probes are also located in the slot on either side of the fuel element. Alpha particles and neutrons are counted and recorded simultaneously by means of scalers. The total alpha count must not exceed

500 dpm. The neutron count is recorded for each plate counted. It should not exceed 130% of the neutron count calculated for the plutonium isotopic composition. A high neutron count may indicate an excess of light elements such as beryllium or fluorine.

The fit of the core plate with the jacket ends may be determined by the difference between fuel-element length and the sum of the core-plate length and end-plug thickness. This fit may also be observed by X radiography. Both methods will be used. The total clearance at both ends should not exceed 0.003 in.

Two leak-detection methods are employed: (1) pressure impregnation, with helium and mass-spectrometer leak detection in a vacuum sampling chamber; and (2) impregnation, followed by bubble testing. Method 1 detects small leaks, method 2 larger leaks. Very large leaks are detected by an "oil canning" of the jacket and 15X examination of all welds. All leaking elements must be rejacketed. No leaks are to be repaired.

Gamma-scan assay is a method of estimating the isotopic content of the element by gamma-energy analysis with a multichannel analyzer. The method supplements mass-spectrometer and chemical analysis.

Go/no-go gages and dial comparators are used for inspecting dimensional conformity. The finished fuel element is weighed. Data on each element are core-plate number, core weight, isotopic composition by weight, stainless steel weight, core length, overall length, and casting batch number. To simplify the recording, tabulation, analysis, and eventual use of these data, they are recorded on IBM data-punch cards. Duplicate cards are maintained with the elements, at Building 350, and in Idaho.

Inspection is done jointly by one Metallurgy technician, one Special Materials Quality Assurance Group Technician, and two Fabrication Procurement Services inspectors. To achieve uniformity of inspection at commercial plants, the Special Materials technician and Fabrication Procurement Services inspectors alternate between field inspection and in-house inspection. The Metallurgy technician also evaluates samples submitted from the commercial vendor.

Data recording is the responsibility of a clerk/technician, who also operates the card-punch machine. The data cards are also coordinated with the Special Materials Division data-process system, insofar as possible.

Two utility technicians are employed on transfers between lines, decontamination, waste packaging, and control and general services of technical equipment. Table V shows wages, salaries, and overhead for 3 months of installation work and training and 12 months of fuel-element production to fabricate 554 kg of plutonium.

6. Indirect Materials and Supplies

The costs of crucibles, molds, cutting tools, and other expendable materials and supplies are estimated in Table VI.

TABLE V. Wages and Salaries plus Overhead for
3 months Installation and Training,
12 months Production

Operation	Technicians \$1800/mo ^a	Machinists \$1400/mo ^b	FPS Inspectors \$1500/mo ^b
Alloy preparation, casting, etc.	(4) \$108,000		
Core-plate machining, Sam- pling, inspection, weighing	(1) 27,000	(3) \$63,000	(1) \$ 22,500
Jacket preparation, cleaning, marking, inspection and fuel-element assembly	(2) 54,000		
Welding	(2) 54,000		
Neutron and alpha counting, leak detection	(2) ^c 54,000		
X-raying, gamma-scan assay final inspection	(2) ^d 54,000		(1) 22,500
Transfers, decontamination, waste control, equipment service	(2) 54,000		
Special materials accounting	(1) ^d 27,000		
Data processing, recording, and control	(1) 27,000		
Totals	(17) \$459,000	(3) \$63,000	(2) \$ 45,000
Total hourly wages and overhead			(22) \$567,000
Staff salaries (2) \$2700/mo			(2) 81,000
Total Salaries, Wages, and Overhead			(24) \$648,000

^aIncludes Divisional and Laboratory overhead plus 3% increase.^bIncludes Central Shops overhead plus anticipated increase.^cOne Industrial Hygiene and Safety Division technician included.^dOne Special Materials Division technician in each category.

TABLE VI. Indirect Materials Cost for Fabrication of 2970 Core Plates

Indirect Materials Costs for 300 Melts			
Ceramics			
60 Crucibles	\$ 25	\$ 1,500	
120 Crucible covers	4	480	
120 Stoppers	3	360	
			\$ 2,340
Molds and Mold and Crucible Wash			
60 Molds	\$275 ^a	\$16,500	
20 Pounds of yttria	55	1,100	
20 Pounds of thorium	12	240	
Stock issue items		3,000	
			\$20,840
Tooling			
50 Carbide slitting saws	\$ 16	\$ 800	
20 Carbide end mills	15	300	
25 Carbide form mills	125	3,125	
			\$ 4,225
Welding Supplies			
40 220-ft ³ cylinders--argon	\$ 6.60	\$ 264	
60 190-ft ³ cylinders--helium	11.00	660	
Stock issue supplies		1,000	
			\$ 1,924
			\$29,329
Contingency			
			10,000
Total Indirect Materials			\$39,329

^aMold cost is based upon cost of experimental molds and should be reduced by quantity production.

7. Process Yield and Residue

Table VII shows a material balance based upon the yield of fuel elements from the later melts of the pilot production tests. Although this estimate has been based on 65% yield of core plates per melt cycle, it may be possible to substantially increase this yield and reduce costs. The amount of material to residue recovery should also be reduced over the 7.0% shown by Table VII. The overall process loss of 0.4% is based upon the assumption that only 80% of what is thought to be in the scrap can be recovered.

Process loss cost for the 554.3 kg of plutonium produced can be estimated as follows:

$$\frac{554.3}{92.95\%} \times 0.392\% = 2.34 \text{ kg of plutonium};$$

$$2.34 \times \$43,000 = \$100,620 \text{ for plutonium loss.}$$

The plutonium to be recovered is estimated as follows:

$$\frac{554.3}{92.95\%} \times 7.00\% = 41.74 \text{ kg.}$$

TABLE VII. Representative Material Balance Based upon 10,000 g Charged

	Weight, g			
	Alloy to Cores Samples and Remelt	Alloy to Recovery	Plutonium	Assumed Pu Loss @ 80% Recovery
PLUTONIUM 2821				
BINARY ALLOY 7179				
10,000				
CASTING				
Skulls and Residues 372		372	105	21.0
Loss by Difference 5				0.2
Cast Bars 9623				
MACHINING				
Mill Cuttings 308		308	87	17.4
Analysis Samples 9				
1709				
Remelt				
Core Plates 7597				
CLEANING AND INSPECTION				
Residue 20		20	5.8	0.6
Core Plates 7577				
JACKETING				
7577				
Recovery of usable binary alloy	9295			
Alloy to residue recovery		700		
Plutonium to residue recovery			198	
Assumed plutonium loss				39.2

Plutonium-recovery charges are influenced by the concentration and cleanliness of the residue. They are also affected by whether or not the plutonium has to be separated and returned to the Commission as nitrate meeting the specification, or whether it can be short-cycled back to the fabrication process. For this estimate, we assumed that 75% of the plutonium recovered can be returned to the fabrication process at a recovery cost of \$2,000 per kilogram, and 25% of the residue requires partition and is returned as $\text{Pu}(\text{NO}_3)_4$ at \$8,000 per kilogram. Lower costs could result from melt recycle of skulls and turnings, but the yields from this remain to be established.

8. Estimated Recovery Costs

The recovery costs are estimated as follows:

$$75\% \times \frac{554.3}{92.95} \times 7.00 \times \$2,000 = \$ 62,600$$

$$25\% \times \frac{554.3}{92.95} \times 7.00 \times \$8,000 = \$ 83,480$$

Estimated Recovery Costs \$146,080.

9. Analytical Costs

Approximately 300 casting batches are melted, including re-melts and scrap-consolidation melts. One-fourth of these must be analyzed chemically and spectrographically. Additional analyses are required to establish the homogeneity of core-plate composition and to establish the accuracy of the secondary methods. It is, therefore, assumed that 100 isotopic, chemical, and spectrographic analyses of high precision are required. The costs are estimated as shown in Table VIII.

TABLE VIII. Estimated Costs of Analyzing ZPPR Fuel Elements

Analysis	Cost per Analysis	Cost per 100 Analyses
Plutonium isotopic	\$120	\$12,000
Plutonium assay	20	2,000
Nitrogen and oxygen	80	8,000
Carbon	80	8,000
Silicon	40	4,000
Molybdenum	20	2,000
Spectrographic	80	<u>8,000</u>
Total primary-analysis cost		\$44,000
Molybdenum secondary analysis		3,000
Incidental analysis		<u>3,000</u>
Estimated analysis costs		\$50,000

Each casting batch is analyzed by secondary assay methods for plutonium isotopic and molybdenum. The gamma-scan assay method has been included in the cost of personnel. Molybdenum is analyzed by solution X-ray fluorescence at about \$15 per analysis. Neutron-counting methods are used to monitor light-element α, n effects.

10. Summary of Costs

Table IX summarizes the costs covered in detail by this report. These costs cannot be compared directly with commercial costs unless factors are added for insurance, plant amortization, profit, and general and administrative expense.

TABLE IX. Summary of Costs to Make 2970 ZPPR Fuel Elements

<u>Equipment (gloveboxes and equipment presently installed not included)</u>		
Capital items	\$ 25,500	
This-job-only and tooling items	61,200	
Drafting and design	4,320	
Modification, installation, and rearrangement of facility	12,200	
Total equipment, tooling, and installation		\$ 103,220
<u>Salaries, Wages, and Overhead (including 3 months training and preproduction)</u>		
Staff	\$ 81,000	
Technician	459,000	
Machinist	63,000	
Inspectors	45,000	
Total wages, salaries, and overhead charges		\$ 648,000
<u>Indirect Costs</u>		
U-Mo alloying	\$ 6,400	
Indirect materials	40,000	
Analytical	50,000	
Estimated plutonium recovery	146,000	
Total indirect costs		\$ 242,400
Total equipment; salaries, wages, and overhead; and indirect costs		\$ 993,620
Or approximately		\$1,000,000
Estimated plutonium losses 0.4% of 600 kg \$43,000		\$ 100,000
Estimated recovery costs for 54 kg at \$8,000		\$ 432,000

Table IX shows the estimated costs for manufacturing 2970 ZPPR fuel elements at Argonne National Laboratory with presently existing equipment. These costs do not include many of the factors that would be part of

a commercial fabricator's pricing, such as plant and equipment amortization, general and administrative costs, shipping, residue recovery, profit, etc.

Table X extends the estimate of Table IX to prices for commercially fabricating various quantities of fuel elements by including all equipment costs, shipping, residue recovery, loss of plutonium at \$43 per gram, general and administrative costs, profit, and a contingency on variable costs. From this analysis we estimated that an experienced fabricator could manufacture 550 kg of plutonium into fuel elements for less than \$5,000 per kilogram and that the price should be substantially less for larger quantities.

TABLE X. Summary of Estimated Price for ZPPR Fuel Element Based on Costs Derived in This Report

	Fixed Costs Training and Preproduction	554 kg Pu 2970 Elements	625 kg Pu 3361 Elements	825 kg Pu 4422 Elements	1000 kg Pu 5360 Elements	1350 kg Pu 7237 Elements	1575 kg Pu 8444 Elements	2900 kg Pu 15,547 Elements
<u>Labor, Direct and Divisional Administration</u>								
a Technicians \$10.40/hr	\$ 90,180	\$ 368,820	\$ 417,359	\$ 549,173	\$ 665,740	\$ 898,684	\$1,048,528	\$ 1,930,662
b Machinists \$8.10/hr	12,600	50,400	57,033	75,045	90,974	122,807	143,284	263,829
c Inspection \$8.65/hr	9,000	36,000	40,738	53,600	64,981	87,720	102,345	188,450
d Supervision \$15.60/hr	16,300	64,700	73,215	96,338	116,787	157,750	183,938	338,685
e Subtotal Labor and Supervision	\$128,080	\$ 519,920	\$ 588,345	\$ 774,156	\$ 938,482	\$1,266,961	\$1,478,095	\$ 2,721,626
<u>Materials and Supplies</u>								
f Plutonium Required, kg	(20)	(615)	(695)	(915)	(1,110)	(1,500)	(1,750)	(3,220)
h Kilogram-days, Pu	(1,800)	(9,000)	(10,100)	(15,000)	(18,000)	(22,000)	(26,000)	(47,000)
<u>Analytical</u>								
i Feed Material Analysis	\$ 260	\$ 6,640	\$ 7,800	\$ 9,900	\$ 12,000	\$ 16,300	\$ 18,800	\$ 35,000
j Primary Product Analysis	5,500	22,275	25,200	33,165	40,200	54,277	63,300	116,603
k Secondary Analysis	3,000	18,000	20,000	26,600	32,000	43,500	56,000	93,282
l Subtotal Analytical	\$ 8,760	\$ 46,915	\$ 53,000	\$ 69,665	\$ 84,200	\$ 114,077	\$ 138,100	\$ 244,885
<u>Residue Recovery</u>								
m kg Pu (7.0% of line g)	(10)	(43.8)	(48.7)	(64.0)	(78.0)	(105)	(122.5)	(225)
n Recovery Subcontract \$8,000/kg	\$ 80,000	\$ 350,400	\$ 389,600	\$ 512,000	\$ 624,000	\$ 840,000	\$ 980,000	\$ 1,800,000
p Equipment and Tooling (See Table IV)	489,331							
q Shipping Containers (20 @ \$375)	7,500							
r Shipping Exclusive-use Truck \$1,900/100 kg Pu Shipped		11,400	13,300	17,100	19,000	26,600	30,400	55,100
s Total Varying Costs		\$ 960,635	\$1,080,245	\$1,420,921	\$1,723,682	\$2,325,638	\$2,717,595	\$ 4,898,111
t Total Fixed Costs	\$721,671	721,671	721,671	721,671	721,671	721,671	721,671	721,671
u Contingency on Varying Costs (20%)		192,127	216,049	284,184	344,736	465,128	543,525	1,142,156
v Subtotal Varying + Fixed + Contingency (Cost Normal to ANL) \$/kg Pu		\$1,874,433	\$2,017,965	\$2,426,776	\$2,790,089	\$3,512,437	\$3,982,791	\$ 6,852,938
		\$ 3,383	\$ 3,229	\$ 2,942	\$ 2,791	\$ 2,602	\$ 2,529	\$ 2,363
<u>Prices Not Normal to ANL</u>								
w Loss kg (0.4% of line g)	(0.1)	(2.46)	(2.78)	(3.66)	(4.44)	(6.00)	(7.00)	(12.88)
x Loss \$43,000/kg	\$ 4,300	\$ 105,780	\$ 119,540	\$ 157,380	\$ 190,920	\$ 258,000	\$ 301,000	\$ 553,840
y Uneconomically Recovered Residue	4,300	105,780	119,540	157,380	190,920	258,000	301,000	553,840
z Subtotal Loss and Residue	\$ 8,600	\$ 211,560	\$ 239,080	\$ 314,760	\$ 381,840	\$ 516,000	\$ 602,000	\$ 1,107,680
<u>Factors Applied to Line v</u>								
aa General and Administrative 15%		\$ 281,165	\$ 302,695	\$ 364,016	\$ 418,515	\$ 526,866	\$ 597,423	\$ 1,027,941
bb Profit 15%		323,340	348,099	418,618	488,267	605,895	687,037	1,182,132
cc General and Administrative and Profit on Fixed Costs		8,600	8,600	8,600	8,600	8,600	8,600	8,600
dd Total Estimated Price		\$2,699,098	\$2,816,439	\$3,532,770	\$4,087,321	\$5,169,798	\$5,877,851	\$10,179,291
ee Total \$/kg Pu		\$ 4,872	\$ 4,506	\$ 4,282	\$ 4,087	\$ 3,829	\$ 3,732	\$ 3,510

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