

SOSR  
Rec. 2/17  
File  
ANL Library

# Argonne National Laboratory

## SPECIFICATION AND PROCUREMENT OF CP-5 FUEL TUBES

by

A. B. Shuck, J. C. Haugen,  
S. Isserow, and R. F. Huber

The facilities of Argonne National Laboratory are owned by the United States Government. Under the terms of a contract (W-31-109-Eng-38) between the U. S. Atomic Energy Commission, Argonne Universities Association and The University of Chicago, the University employs the staff and operates the Laboratory in accordance with policies and programs formulated, approved and reviewed by the Association.

#### MEMBERS OF ARGONNE UNIVERSITIES ASSOCIATION

The University of Arizona  
Carnegie-Mellon University  
Case Western Reserve University  
The University of Chicago  
University of Cincinnati  
Illinois Institute of Technology  
University of Illinois  
Indiana University  
Iowa State University  
The University of Iowa

Kansas State University  
The University of Kansas  
Loyola University  
Marquette University  
Michigan State University  
The University of Michigan  
University of Minnesota  
University of Missouri  
Northwestern University  
University of Notre Dame

The Ohio State University  
Ohio University  
The Pennsylvania State University  
Purdue University  
Saint Louis University  
Southern Illinois University  
The University of Texas at Austin  
Washington University  
Wayne State University  
The University of Wisconsin

#### NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately-owned rights.

Printed in the United States of America  
Available from  
National Technical Information Service  
U.S. Department of Commerce  
Springfield, Virginia 22151  
Price: Printed Copy \$3.00; Microfiche \$0.65

ARGONNE NATIONAL LABORATORY  
9700 South Cass Avenue  
Argonne, Illinois 60439

SPECIFICATION AND PROCUREMENT  
OF CP-5 FUEL TUBES

by

A. B. Shuck and J. C. Haugen\*  
Argonne National Laboratory

and

S. Isserow and R. F. Huber  
Nuclear Metals Division, Whittaker Corp.

Procurement Services Department

August 1970

\*Reactor Operations Division.



TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT . . . . .	7
I. INTRODUCTION . . . . .	7
II. MANUFACTURE AND QUALITY CONTROL OF CP-5 FUEL TUBES . . . . .	10
A. Process Materials . . . . .	10
B. Charge Makeup . . . . .	10
C. Melting and Casting . . . . .	13
D. Casting, Cropping, and Sampling . . . . .	14
E. Primary Extrusion . . . . .	14
F. Coextrusion Components . . . . .	16
G. Surface Treatment and Coextrusion Billet Assembly and Welding . . . . .	17
H. Leak Detection . . . . .	18
I. Billet Coating, Preextrusion Heating, and Seal-off. . . . .	18
J. Coextrusion . . . . .	19
K. Postextrusion Processing . . . . .	20
L. Fluoroscopy . . . . .	20
M. Blister Test . . . . .	20
N. Radiography, Straightening, and Final Cropping . . . . .	20
O. Final Inspection . . . . .	21
P. Chip Recovery . . . . .	21
III. CONTRACT NO. 31-109-38-2328 DATA EVALUATION . . . . .	22
A. Ultrasonic Test . . . . .	23
1. Grade A Tubes . . . . .	26
2. Grade B Tubes . . . . .	26
3. Grade C Tubes . . . . .	26
B. Visual Inspection . . . . .	27

## TABLE OF CONTENTS

Page

## APPENDIXES

A. Scope of Work. Specifications for CP-5 Fuel Element Subassembly Parts . . . . .	28
B. Specification RF-001a (CP-5) "Alclad Al-U Alloy Fuel Tubes" . . . . .	31
C. Determination of Uranium (10-30%) in Aluminum by Titration with Metrohm Potentiograph for CP-5 Fuel Alloy . . . .	48
D. Inspection Sheets for CP-5 Fuel Tubes . . . . .	50
E. Data Sheets for CP-5 Fuel Tubes . . . . .	54

LIST OF FIGURES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1.	CP-5 Fuel Assembly . . . . .	9
2.	Flow Diagram for Fabrication of CP-5 Fuel Tubes . . . . .	11
3.	Schematic Diagram of Chill Mold . . . . .	13
4.	Schematic Arrangement of Primary-extrusion Billets and Tools. . . . .	15
5.	Schematic Drawing of Coextrusion Billet . . . . .	16

LIST OF TABLES

<u>No.</u>	<u>Title</u>	<u>Page</u>
I.	Core Compositions and Dimensions of Fuel Tubes. . . . .	8
II.	Extrusion-tool Diameters . . . . .	15
III.	Dimensions of Coextrusion Billet Components . . . . .	16
IV.	Dimensions of Coextrusion Tools . . . . .	19
V.	Comparison of Specified vs Statistically Evaluated Data. . . . .	24
VI.	Evaluation of Uranium Assay Data for Inner and Intermediate Tubes . . . . .	25
VII.	Evaluation of Uranium Assay Data for Outer Fuel Tubes . . . . .	25

LIST OF TABLES

1	Table 1. General Information
2	Table 2. General Information
3	Table 3. General Information
4	Table 4. General Information
5	Table 5. General Information
6	Table 6. General Information
7	Table 7. General Information
8	Table 8. General Information
9	Table 9. General Information
10	Table 10. General Information

LIST OF TABLES

11	Table 11. General Information
12	Table 12. General Information
13	Table 13. General Information
14	Table 14. General Information
15	Table 15. General Information
16	Table 16. General Information
17	Table 17. General Information
18	Table 18. General Information
19	Table 19. General Information
20	Table 20. General Information
21	Table 21. General Information
22	Table 22. General Information
23	Table 23. General Information
24	Table 24. General Information
25	Table 25. General Information
26	Table 26. General Information
27	Table 27. General Information
28	Table 28. General Information
29	Table 29. General Information
30	Table 30. General Information

# SPECIFICATION AND PROCUREMENT OF CP-5 FUEL TUBES

by

A. B. Shuck, J. C. Haugen,  
S. Isserow, and R. F. Huber

## ABSTRACT

Fuel assemblies for the Argonne National Laboratory Research Reactor CP-5 have consisted of concentric nested, aluminum-clad, aluminum-uranium alloy fuel tubes. The fuel is prepared by dissolving fully enriched uranium in molten aluminum and casting hollow cylindrical billets. The billets are extruded as tubes from which cylindrical cores are machined. These are clad inside and outside in sealed aluminum jackets, which are bonded to the cores by coextrusion. The resulting aluminum-clad fuel tubes are radiographed, cut to length, blister-tested, cleaned, inspected, and sent to Argonne National Laboratory for construction of CP-5 fuel assemblies.

Data from manufacture and inspection of 37 sets of the fuel tubes were evaluated, and a detailed procurement specification was prepared for future procurement of the tubular elements.

## I. INTRODUCTION

Nuclear Metals, Inc., (now a division of Whittaker Corporation) developed techniques for making aluminum-clad, Al-U alloy fuel tubes during the late 1950's. Argonne National Laboratory (ANL) designed concentric fuel-tube assemblies for Research Reactor CP-5. The center of each assembly provided potential space for materials irradiation or instrumentation. The first CP-5 loading of this design was made in 1959-1960 from coextruded tubes obtained from Nuclear Metals. Subsequent CP-5 orders were made in 1963, 1965, 1967, and 1969. All of these loadings were made under cost-plus-fixed-fee contract arrangement in accordance with a simple scope-of-work specification shown by Appendix A.

When the 1969 Contract No. 31-109-38-2328 was placed, the need for a more formalized specification was recognized. The development of such a specification cooperatively by ANL and Nuclear Metals Division (NMD) was made part of the 1969 procurement effort. The Specification RF-001a (CP-5) is included in this report as Appendix B.

The CP-5 fuel assembly, shown schematically in Fig. 1, consists of three concentrically nested fuel tubes between upper and lower extensions. The lower extension engages the reactor core support grid and provides orificing for controlling the flow of the heavy water moderator-coolant. The upper extension has provision for coolant outflow, shielding, access to the centrally located irradiation space, and a lifting-bracket attachment. These assemblies are constructed at ANL from commercially procured fuel tubes.

The fuel tubes consist of Al-U alloy tubular cores that are clad inside and outside with 0.015-in. thickness of commercial aluminum alloy 1100. The cladding extends beyond the core ends and the end spaces are filled and sealed with Al-1.0 wt % Mg alloy. The core compositions and the dimensions of the fuel tubes are as shown in Table I.

TABLE I. Core Compositions and Dimensions of Fuel Tubes

	Fuel Tube Type		
	I Inner	II Intermediate	III Outer
Tube length, in.	$27 \frac{9}{16}$	$27 \frac{9}{16}$	$32 \frac{5}{32}$
Tube ID, in.	$2.112 \pm 0.005$	$2.516 \pm 0.005$	$2.900 \pm 0.005$
Tube wall, in.	$0.062 \pm 0.003$	$0.062 \pm 0.003$	$0.050 \pm 0.003$
Cladding thickness, <sup>a</sup> in.	0.015	0.015	0.015
Core length, in.	$25 \frac{13}{16}$	$25 \frac{13}{16}$	$25 \frac{13}{16}$
Core thickness, <sup>a</sup> in.	$0.032^{+0.004}_{-0.003}$	$0.032^{+0.004}_{-0.003}$	$0.020^{+0.004}_{-0.003}$
Nominal core alloy	Al-23.3 wt % U	Al-23.3 wt % U	Al-16.4 wt % U

<sup>a</sup>See the specifications for the allowable variations in cladding and core thickness.

Additional requirements for the CP-5 fuel elements are given in Appendix B of this report. The fuel tubes are procured as matched sets of three to produce the specified <sup>235</sup>U content of the set. The <sup>235</sup>U enrichment of the uranium is nominally 93%.

The purposes of this report are as follows: (1) to describe production and quality-control methods used by the contractor for the manufacture of the fuel tubes; (2) to present a specification that may serve for future fixed-price or cost-plus-fixed-fee contracts for CP-5 fuel-tube manufacture, and (3) to evaluate and summarize data from processing and inspection of CP-5 fuel tubes. This report does not cover the ANL receiving inspections or CP-5 fuel-element assembly processes.

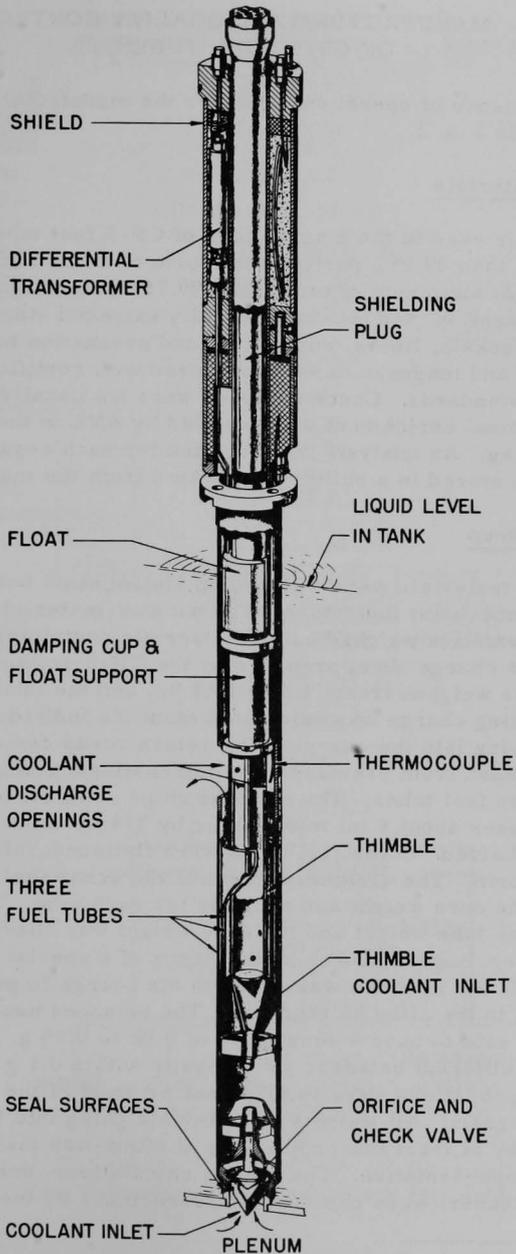


Fig. 1. CP-5 Fuel Assembly. ANL Neg. No. 144-82 Rev. 1.

## II. MANUFACTURE AND QUALITY CONTROL OF CP-5 FUEL TUBES

The sequence of operations used for the manufacture of CP-5 fuel tubes is shown in Fig. 2.

### A. Process Materials

Materials used in the manufacture of CP-5 fuel tubes were (1) aluminum of more than 99.95% purity in the form of scored ingots for core alloy makeup, (2) aluminum of more than 99.75% purity and magnesium for seal alloy preparation, and (3) commercially extruded stock of aluminum alloy 1100 for jackets, liners, end plates, and evacuation tubes. The aluminum alloy 1100 and magnesium were procured with certification of compliance to purity standards. Check analyses were not usually made. Uranium of 93%  $^{235}\text{U}$  nominal enrichment was supplied by ANL in the form of reguli weighing 4-4.5 kg. An analysis was supplied for each regulus. Nuclear materials were stored in a building separated from the main plant.

### B. Charge Makeup

Charge materials were weighed on Metrogram\* balances having electrical balance-point indicators. The nuclear-materials and criticality-control representative weighed out all materials containing uranium in accordance with a charge sheet prepared by the CP-5 project leader. The melting charges weighed from 11.5 to 14.2 kg, and the total uranium content of the melting charge was calculated from the individual analyses of the material going into the charge. The return scrap containing uranium included "hot tops" from previously melted castings, compacted machine chips, and scrap fuel tubes. The machine chips were cleaned and cold-pressed into cakes about 5 in. in diameter by  $3/4$  in. thick. Hot tops from castings were halved. Scrap fuel tubes were flattened, folded, and pressed into compact form. The uranium content of the scrap fuel tubes was calculated from the core weight and analysis for each tube. The difference between the fuel-tube weight and the core weight was charged as aluminum. The uranium reguli were broken up by means of a special die in a hydraulic press and sufficient uranium was added to the charge to provide 16.4 or 23.3% uranium in the alloy as required. The balances used for charge weighing were said to have a sensitivity of 0.02 to 0.05 g, and when properly used, the different balances gave results within 0.1 g of each other. The balance calibrations were verified before start of the CP-5 fuel-fabrication program. All weights of materials going into the alloy charges were checked by at least two people, one of whom was the nuclear-materials management representative. The charge calculations, normally made by the CP-5 project leader, were checked for correctness by the melter.

\*H. A. Hadley, Inc., Burlington, Vermont.

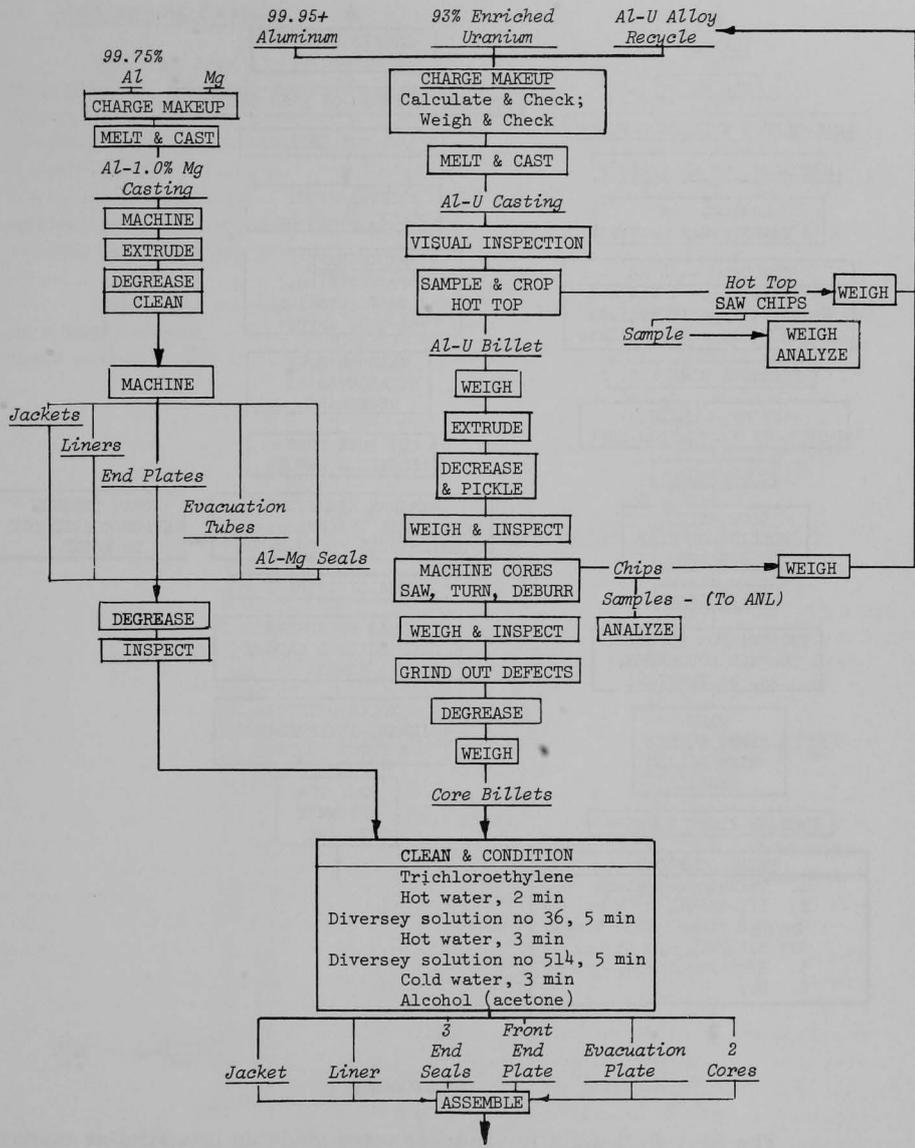


Fig. 2. Flow Diagram for Fabrication of CP-5 Fuel Tubes

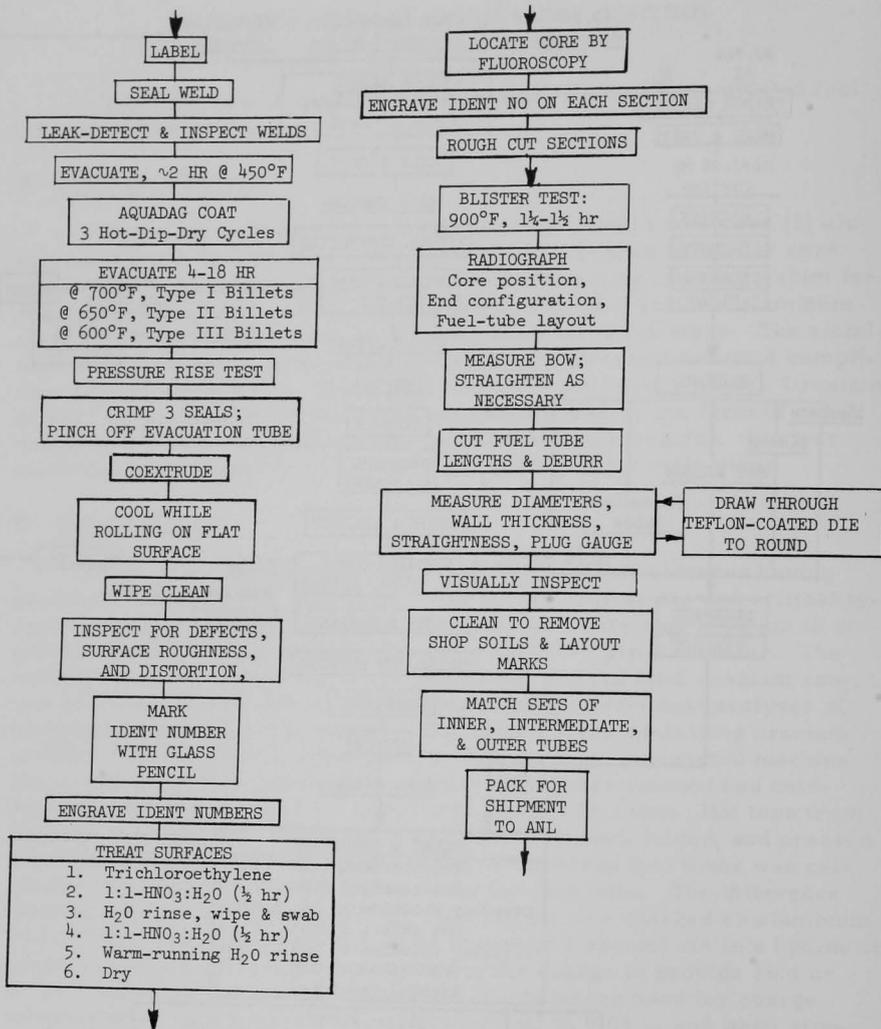


Fig. 2 (Contd.)

The Al-1 wt % Mg alloy charges were made up in a similar manner to that used for preparing the uranium alloy. However, this alloy was not under close nuclear-materials management control.

### C. Melting and Casting

Melting was done in a conventional tilt-pour high-frequency induction furnace. The furnace is lined with a carbon crucible about 9 in. in diameter and 12 in. deep. A carbon-crucible cover was used to prevent splatter losses and to control the argon-gas atmosphere, which was introduced beneath the cover to reduce oxidation during melting. After melting, the alloy is continuously sparged with argon gas by means of a carbon sparge tube, dipped into the bottom of the crucible. A graphite-sheathed dipping thermocouple was used to measure melt temperatures.

The mold configuration is as shown in Fig. 3. The mold consisted of a water-cooled body or chill, a carbon hot top, a carbon core, and a mold bottom. The alloys were poured into the mold through a carbon

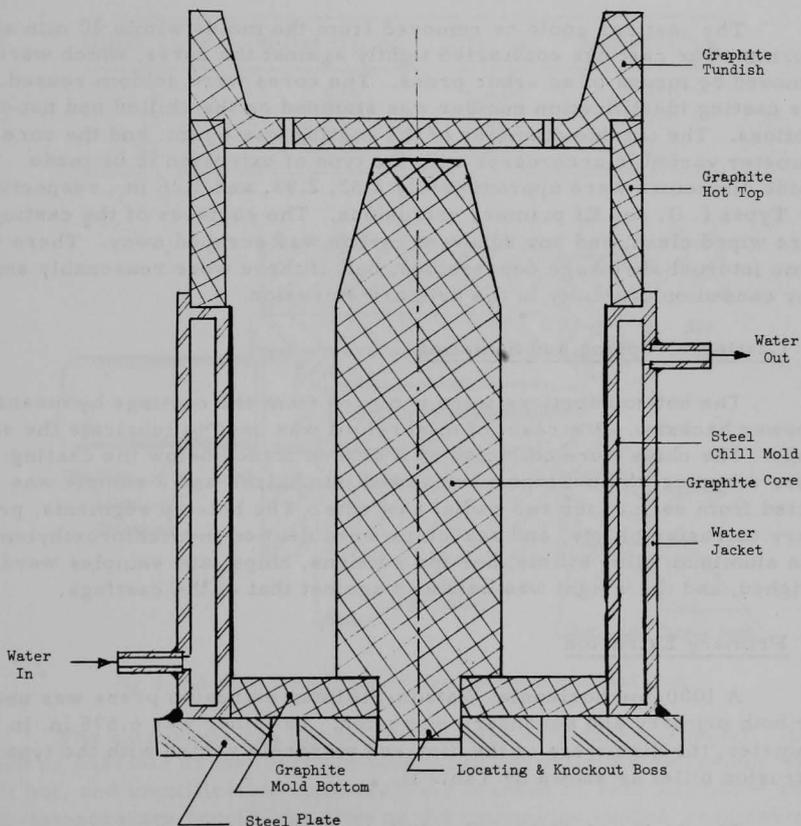


Fig. 3. Schematic Diagram of Chill Mold

tundish. The mold components and the tundish were outgassed in air at 950-1000°F for 1 hr. Except for the tundish and the hot top, these components were stored in a drying oven at about 400°F and assembled 15 min after initiation of the melt sequence (about 1 hr before the pour). The tundish and the hot top were left in the furnace at 900°F until assembly just before the pour.

The aluminum was melted first; the hot tops, fuel tubes, compacted turnings, and uranium were dissolved in the melt in that order. As soon as the additions were melted, the sparge tube was immersed and the melt stirred with argon gas at 4 cu ft/min flow replacing the blanket argon flow previously maintained at 10 cu ft/min. The Al-23.3 wt % U melt was superheated to 1840°F and cooled to 1620°F for pouring. The Al-16.4 wt % U melt was superheated to 1750°F and poured at 1500°F.

The castings could be removed from the molds within 20 min after pouring. The castings contracted tightly against the cores, which were removed by means of an arbor press. The cores were seldom reused. The casting identification number was stamped on the chilled and hot-top sections. The outside diameter of the casting was  $6\frac{1}{2}$  in., and the core diameter varied in accordance with the type of extrusion to be made. The inside diameters were approximately 2.52, 2.93, and 3.26 in., respectively, for Types I, II, and III primary extrusions. The surfaces of the castings were wiped clean, and any adherent carbon was scraped away. There were some internal shrinkage depressions, but, if these were reasonably smooth, they caused no difficulty in the primary extrusion.

#### D. Casting, Cropping, and Sampling

The hot-top sections were removed from the castings by means of a power hacksaw. A stream of mineral oil was used to lubricate the saw blade. The chips were collected on a screen placed below the casting. After cropping, the hot tops were sawed into halves and a sample was collected from each of the two radial saw cuts. The hot-top segments, primary extrusion billets, and saw chips were deoiled in trichloroethylene. The aluminum alloy billets, hot-top sections, chips, and samples were weighed, and the weight was balanced against that of the castings.

#### E. Primary Extrusion

A 1000-ton horizontal Watson-Stillman extrusion press was used for both primary and secondary extrusion. The liner was 6.575 in. in diameter; the diameters of the dies and mandrels varied with the type of extrusion billet as shown by Table II.

The Al-U and Al-Mg alloy billets were extruded in tandem in the same operation. The metal tools were first oxidized by heating to 700-900°F for 4 hr. They were then coated with colloidal graphite. When

several sets of the tools were to be used, they were carefully positioned in the furnaces so that the tool sets could be kept together. The dies, mandrels and liner were heated to 600°F, and the extrusion billets to 800°F, each for not less than 4 hr. Just before the extrusion, the dies and liner were coated with a lead-bentonite grease suspension. The Al-1 wt % Mg billet was then placed on the mandrel with the top end against the backer. A liberal coating of lead-bentonite was painted on the front of the Al-Mg billet, the Al-U billet was placed on the mandrel with the top end toward the die, and the entire assembly was coated with the lead-bentonite lubricant. These operations were done as rapidly as possible in order to conserve heat. The assembly was then placed in the press, as shown schematically in Fig. 4. The press was closed and extrusion accomplished at a ram speed of 13 in./min.

TABLE II. Extrusion-tool Diameters

Billet Type	Billet ID, in.	Die, in.	Mandrel, in.
I	2.520	3.449	2.519
II	2.930	3.881	2.982
III	3.260	3.881	3.259

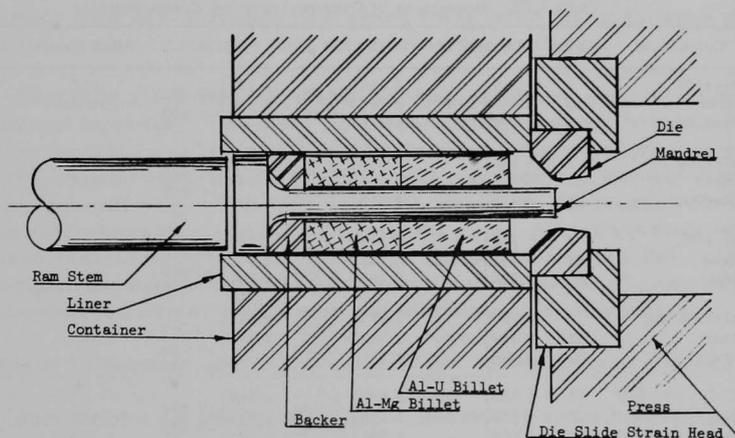


Fig. 4. Schematic Arrangement of Primary-extrusion Billets and Tools

The extrusions ran out on a table lined with asbestos paper. As much as possible of the lubricant was wiped off while the extrusions were still hot, and identification numbers were marked on each extrusion with a high-temperature pencil. As soon as the extrusions cooled, permanent numbers were vibratooled on the front end of each extrusion. The lead-bentonite lubricant is removed from the extrusions by scrubbing in

trichloroethylene and pickling in a 1:1  $\text{HNO}_3:\text{H}_2\text{O}$  solution. The extrusions were weighed before and after pickling, and a record was kept of the cumulative weight loss to the pickling solution.

#### F. Coextrusion Components

Two Al-U fuel sleeves and three Al-1 wt % Mg front, center, and rear spacer sleeves were jacketed with inner and outer cladding cylinders and front and rear seal rings, as shown in Fig. 5. The rear seal ring was fitted with an evacuation tube, which was welded at the front and Al-Si brazed at the rear. The dimensions of the components are shown in Table III. The

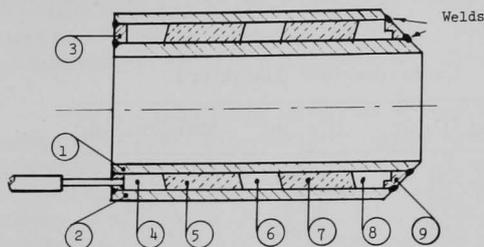


Fig. 5  
Schematic Drawing of Coextrusion Billet. (See Table III for identification of numbered items.)

TABLE III. Dimensions of Coextrusion Billet Components

Item	Component	Type	Length, in.	OD, in.	ID, in.	Dish Front	Angle Rear
1 <sup>a</sup>	Al-1100 Inner Cladding	I	6.410	2.558	2.123	45° 1/32 flat	0°
		II	6.470	2.965	2.530		
		III	6.790	3.295	2.915		
2 <sup>a</sup>	Al-1100 Outer Cladding	I	5.860	3.628	3.325	45°	0°
		II	5.910	4.069	3.748		
		III	6.420	4.069	3.757		
3	Al-1100 Rear Seal	I	0.250	3.319	2.564	0°	0°
		II	0.250	3.742	2.971		
		III	0.250	3.751	3.301		
4 <sup>a</sup>	Al-1.0% Mg Rear Spacer	I	0.789	3.319	2.564	15°	0°
		II	0.789	3.742	2.971		
		III	0.789	3.751	3.301		
5 and 7	Al-U } 23.3% U Fuel } 23.3% U Rings } 16.4% U	I	1.530	3.319	2.564	15°	15°
		II	1.550	3.742	2.971		
		III	1.805	3.751	3.301		
6	Al-1.0% Mg Center Spacer	I	0.850	3.319	2.564	15°	15°
		II	0.850	3.742	2.971		
		III	0.850	3.751	3.301		
8 <sup>a</sup>	Al-1.0% Mg Front Spacer	I	0.825	3.319	2.564	Counterturn 2.950 x 0.125 3.350 x 0.125 3.525 x 0.125	15°
		II	0.825	3.742	2.971		
		III	0.825	3.751	3.301		
9 <sup>a</sup>	Al-1100 Front Seal	I	0.578	3.319	2.564	45°	Counterbore 2.958 x 0.125 3.358 x 0.125 3.531 x 0.125
		II	0.586	3.742	2.971		
		III	0.425	3.751	3.301		

<sup>a</sup>Lengths of dished components are of longest cylindrical surface.

Al-U primary extrusions were inspected for defects, such as extrusion tears or entrapped die lubricants. Core sleeves were power-sawed from the sound sections. Three or four core sleeves were cut from each primary extrusion. The front and tail ends of the extrusions were recycled. Chips were collected from each saw cut and preserved in clear mineral oil. The chips were sent to the analytical laboratory in screw-cap bottles, identified by extrusion serial number and position. The individual core rings were turned on a lathe to the specified dimensions from the sleeves.

After machining, the core rings were measured and inspected for surface and entrained defects. If the defects did not exceed 0.040 in. deep, they were ground out by means of a power tool and steel burr. Grinding was done within a plastic bag in order to catch the fine chips. The core rings were washed, swabbed in acetone, and weighed to the nearest 0.10 g. The weight of the rings and location of any minor defects were recorded on inspection sheets. The content of  $^{235}\text{U}$  was calculated from analyses of the two samples taken from the nearest enclosing pair of saw cuts. All saw chips, grinding chips, and turnings were weighed, and the total weight of the products of machining was balanced against the initial weight of a primary extrusion.

The Al-Mg extrusions were treated in a similar fashion to the Al-U extrusions. The only turnings preserved were lathe turnings from the two ends of the cropped primary that were taken for analysis.

The 1100 alloy end rings and cladding cylinders were machined by conventional methods. The alloy 1100 evacuation tubes were machined from 1/4-in.-OD by 1/16-in.-ID stock tubing, one end of which was turned to a 0.124-in. diameter for 1 1/4 in. This end was inserted through a hole in the rear end seal ring. The tube was TIG-welded to the front of the ring and was Al-Si brazed to the back of the seal ring. All components were degreased in trichloroethylene, inspected dimensionally and the coextrusion components matched for assembly. The dimensions of components were checked for conformance with the machine specifications.

#### G. Surface Treatment and Coextrusion Billet Assembly and Welding

Just before assembly, all components of the coextrusion billets were surface conditioned through: (1) trichloroethylene; (2) hot flowing water for 2 min; (3) alkaline cleaning solution (Diversey No. 36\*) for 5 min; (4) hot flowing water for 3 min; (5) acid deoxidizer (Diversey No. 514) for 5 min; (6) cold-running-water rinse for at least 3 min; and then (7) dry ethanol or acetone. Acetone was preferred to ethanol for final rinse, because it was more completely eliminated and caused less gas in welding. However, difficulty was sometimes experienced with seizing of the components when the assemblies were made dry or lubricated with acetone.

\*Diversey Chemical Company, 212 W. Monroe Street, Chicago, Illinois.

Alcohol was used as an assembly lubricant when this occurred. The alcohol was sprayed onto the components during assembly by means of a laboratory wash bottle.

A tag was attached to the evacuation tubes showing the number and location of each core in the assembly. The assembled billet was allowed to stand for 15-20 min to allow alcohol to evaporate before welding. TIG welds were then made between the cladding and end seal rings with the assembly supported on a hand-operated turntable. The welding torch and welding current were manually controlled. After welding, the coextrusion billets were inspected visually and defects were repaired.

#### H. Leak Detection

The evacuation tubes of the welded coextrusion billets were attached to a vacuum system and a helium leak detector. After pumpdown to less than 0.1 Torr, a valve between the vacuum system and the mass-spectrometer leak detector was opened and the welds were searched for leaks by means of a helium jet. If a leak was indicated, it was precisely located by use of a rubber-tipped probe at low helium flow. The leaks were marked and the coextrusion billet returned for weld repair.

#### I. Billet Coating, Preextrusion Heating, and Seal-off

While under evacuation, the leak-tight billets were outgassed, graphite-coated, and heated to extrusion temperature. The composite billets were attached by means of a flexible tube to a vacuum manifold. Each manifold branch was provided with a thermocouple, a vacuum gauge, and a valve. The billets were heated in vertical tube furnaces for approximately 2 hr at 450°F. While hot, they were dipped in colloidal graphite suspension consisting of 40 parts of distilled water to one part of commercial Aquadag. The mix was stirred immediately before using, and the hot billets were submerged and then quickly removed so that a coating of heat-dried graphite was formed. The dip-drying cycle was performed three times while the billet was still hot.

The billets were now ready for final heating prior to extrusion. They were replaced in the tube furnaces and heated for 4-18 hr at 700°F for Type I billets, 650°F for Type II billets, and 600°F for Type III billets. Although billets could be extruded within 4 hr after coating, it was normal practice to heat the billets overnight for the following day's extrusions. Shortly before extrusion, the valve to the manifold was closed and the rise of pressure was read on the vacuum gauge for 1 min. If the vacuum remained essentially constant, the billet was sealed off immediately before extrusion. This was accomplished by a modified bolt cutter with flattened dies in place of the blades. Three crimps were made at overlapping locations, and the tube was cut off by means of a side cutter at the crimp furthest from the billet.

## J. Coextrusion

The extrusion tools for the three types of fuel tubes are of a dimension shown in Table IV. The liners, dies, mandrels, and backers are prepared by cleaning with trichloroethylene, oxidizing in air for 4 hr at 800-900°F, and coating with a colloidal graphite mix, consisting of one part Aquadag\* and three parts methanol. The coating was applied by brushing to all surfaces of the dies, mandrels, and backers and to the bore of the liner. The coated extrusion tools were placed on stainless steel trays in furnaces and heated for a minimum of 2½ hr. The liners were heated at 650°F, dies at 400°F, mandrels at 600°F, and cutoff cuffs at 800°F. The dies were placed in the liners in the 650°F furnace for 15-30 min before extrusion, the cutoffs were placed on the mandrels in the 600°F furnace approximately 30 min before use. The AZ-31B magnesium alloy cutoff cuff was used to displace the extrusion heel, which would normally remain between the die and the backer.

TABLE IV. Dimensions of Coextrusion Tools

	Type I	Type II	Type III
Liner ID, in.	3.655	4.104	4.104
Die OD, in.	3.654	4.103	4.103
	2.246	2.652	3.016
<u>Mandrel</u>			
Backer diameter, in.	3.654	4.103	4.103
Stem diameter, in.	2.125	2.531	2.916

Immediately before extrusion, lead-grease lubricant was applied to the mandrel, liners, and dies. This lubricant consisted of three parts Plastilube\*\* No. 1, one part fine lead powder, and sufficient motor oil to make up a barely flowing mixture. This mixture was applied by paint roller and saturated cloth pad to the liner, die, mandrel, and backer. The billet was brought from the evacuation room and placed on the mandrel, and the lead-grease lubricant was brushed thinly on the nose and outside by means of a fine brass brush. Excess lubricant was wiped away.

The assembly was placed in the extrusion press, and the billet extruded at a ram speed of 36 in./min with the press stop set to leave a 3/4-in. cutoff butt. During extrusion, the ram pressure gauge read between 200 and 300 tons. The reduction ratios for the various types of tubes were 16.7:1 for Type I extrusions producing the inner tubes, 16.6:1 for the Type II extrusions producing intermediate tubes, and 14.3:1 for the Type III extrusions producing the outer tubes.

\* Achison Colloids Co., Port Huron, Michigan.

\*\* Plastilube is a product of Warren Refining and Chemical Company, 750 Prospect Avenue, Cleveland, Ohio.

As the extrusions emerged from the press, they were caught in the gloved hands of two operators. One man supported the front of the tube, while the second man supported the rear to prevent its dropping on the extrusion table and becoming marred or scarred. The hot extrusion was transferred to an asbestos-paper-covered table and rolled back and forth by hand for 10 min to prevent bowing or distortion. The lubricants were wiped from the tube while it was still hot, and the core identification numbers were transferred to the front end of the extrusion by means of a hot-materials marking pencil. As soon as the tubes were cooled, these numbers were engraved by vibratool. Each tube was inspected visually, as soon as it was made, so that defect-producing conditions could be corrected on subsequent extrusions. The safe handling limit was six billets.

#### K. Postextrusion Processing

The extruded tubes were transferred to the postextrusion processing and inspection area on the second floor of the plant. A zero-degree reference mark was engraved on the front end of each extrusion, and the surfaces were cleaned and treated through the following sequence of operations:

- (1) Degrease in trichloroethylene; (2) wipe inside and outside; (3) immerse in 1:1  $\text{HNO}_3:\text{H}_2\text{O}$  solution; (4) wash and swab in warm running water; (5) treat in 1:1  $\text{HNO}_3:\text{H}_2\text{O}$  solution for 1/2 hr; (6) rinse in warm running water; and (7) swab dry inside and outside.

#### L. Fluoroscopy

The core locations were determined by fluoroscopy in a cabinet-type, 150-kVA X-ray radiography and fluoroscopy machine (Picker X-ray Corporation). The positions were marked temporarily by means of a felt marking pen, and a rough-cut index mark and an identification number were vibratooled at the end of each core section. The tube surfaces were then protected by wrapping paper, and sections were cut at the index marks by means of a bandsaw. The cut sections were degreased, swabbed inside and outside, placed in stainless steel trays, and covered with aluminum foil and placed in foil for blister testing.

#### M. Blister Test

The blister test consisted of heating the tube at 900°F for  $1\frac{1}{4}$  to  $1\frac{1}{2}$  hr without flux in an air atmosphere. The tubes were then cooled, and the inner and outer surfaces were inspected for blisters. Any blisters larger than 1/16 in. in diameter were cause for rejection.

#### N. Radiography, Straightening, and Final Cropping

Precise location of the core was determined before final cutting by Polaroid radiography. Lead inch marks, identification numbers, and index

marks were affixed at the front and rear of the tube by means of pressure-sensitive tape. Radiography was done at 150 kVA, 5 mA, on Polaroid Radiography Film Packet No. 1001 with a 5.5- to 7-min rotated exposure or with Polaroid Radiographic Packet No. 3000 with a  $1\frac{1}{2}$ - to 3-min rotating exposure. The film packet was wrapped around a brass spring holder, which held the film packet in contact with the inside tube wall. The film was exposed one end at a time and then was removed from the fuel tube and processed through a Polaroid film processor.

The position and maximum lengths of the cores were measured, and the 90%-thickness lengths were estimated by measurement from the radiograph films. The locations of the fuel tube ends were laid out as specified. The finished cuts were made by means of a fine-tooth bandsaw. A fixture and very slow feed speeds were used to maintain end perpendicularity and produced a smooth cut. Both ends were deburred, and the tubes were swabbed with acetone and stored in cardboard mailing tubes.

#### O. Final Inspection

Final inspection was done on polyethylene surface plates. A gauge consisting of height blocks was set up. The tubes were carefully rotated against the square edge to measure bow and end perpendicularity. The length was measured by means of a 4-ft vernier. Polyethylene cylindrical blocks were inserted at least 3 in. and flush with the tube ends, and the tubes were straightened. Any bulges noted near the core were removed by die sizing or gently tapping on the bulged area with the inside of tube supported on a mandrel. The outside diameter and wall thickness were measured by micrometers, as required by the specification. If excess ovality occurred, the tubes were rounded by pulling through a die, which was lubricated by a Teflon coating. The tube was coated with Johnson No. 15 WaxDraw.\* All tubes were carefully cleaned with acetone to remove the lubricants, shop soils, layout marks, or fingerprints. The tubes were visually inspected for scratches, striations, dents, or other defects listed in the specification. The inner, intermediate, and outer tubes were assembled in sets to the required  $^{235}\text{U}$  content. The sets of tubes were packed for shipment to ANL.

#### P. Chip Recovery

The turnings from the U-Al primary extrusion were deoiled by washing with trichloroethylene after weighing. The clean turnings were returned to the fabrication area where they were pressed into compacts about 5 in. in diameter by  $1/2$  to 1 in. thick.

Chips from each saw cut were collected in separate bottles labeled with the billet number and position number. About 15 g of chips were

---

\*Johnson's Wax Company, 1521 Howe St., Racine, Wisc.

collected for analysis from each saw cut through the primary extrusion. This material was deoiled through trichloroethylene and acetone.

Argonne National Laboratory required two 5-g samples from each primary extrusion. One of these was taken from the saw chips ahead of the first core sleeve, and the second was taken from behind the last core sleeve. Each sample was placed in a screw-cap bottle and flooded with clean mineral oil, and the bottle was marked with the extrusion number and position.

The Nuclear Metals Division analyzed 1-g samples for uranium content from each saw cut, including those at the ends of the extrusion. The method was to dissolve the sample in sulfuric acid, reduce  $U^{+6}$  to  $U^{+4} + U^{+3}$  with zinc; aerate to  $U^{+4}$ ; and, in the presence of ferric chloride and phosphoric acid, titrate to  $U^{+6}$  with potassium dichromate on a potentiograph. One sample from each casting was analyzed for iron, and a correction factor (1 mg Fe  $\div$  2.13 mg of U) was applied to the uranium determination. The procedure used for uranium analysis is given in Appendix C.

The method is standardized by a synthetic Al-U standard sample prepared from analytic reagent grade aluminum and National Bureau of Standards,  $U_3O_8$  Standard Sample, NBS-950a. The standard sample is checked for each run of analyses of a given composition. A run usually is not more than 10 analyses. Precision of the method was predetermined by repeated analyses of the NBS standard and has been controlled by comparing the daily analyses of the NBS standard.

The Nuclear Metals Division determines magnesium on samples from the front and rear of Al-1.0 wt % Mg primary extensions. The method described by ASTM E-34-58 is used for determination of magnesium in the alloy. Where raw materials are procured with certified analyses, NMD does not usually run check analyses for impurities of these materials.

### III. CONTRACT NO. 31-109-38-2328 DATA EVALUATION

The Nuclear Metals Division (NMD) furnished the following records for each shipment of fuel tubes on Contract No. 31-109-38-2328:

- a. X-radiographs (Polaroid) of the ends of each fuel tube showing the core end configuration and location.
- b. CP-5 Fuel Data Sheet summarizing core measurements and calculated  $^{235}U$  content of each tube and tube set.

- c. CP-5 Fuel Tube Inspection Sheets.
- d. Chemical Analysis Report Forms showing analyses made on the cast billets and primary extrusions.

The CP-5 (fuel tube) Data Sheets, which summarize core and tube dimensions and calculated  $^{235}\text{U}$  content, are shown in Appendix E. The Polaroid X-radiographs were used to measure end-seal lengths and maximum and minimum core lengths. These measurements were made by scale to the nearest  $1/64$  in. and reported decimally on the data sheets. Examples of the CP-5 inspection sheets and the Chemical Analyses Report Forms are also given in Appendix D. One objective of this work was to determine whether the specified dimensions and  $^{235}\text{U}$  content can be realistically met by the NMD process. This was done by statistically determining the average,  $\bar{x}$ , and  $\pm 2\sigma$  (s) deviation for each of the 37 sets of fuel tubes. Twice the standard deviation  $2\sigma$  (corrected for the small sample,  $n = 37$ ) very closely approximates the 95% acceptability of a normally distributed population. Although the size of the sample was smaller than desirable, the results shown in Table V indicate that the dimensions and  $^{235}\text{U}$  content can be met more than 95% of the time. The exception was bow, which is subject to additional straightening.

The results of uranium assay are shown in Tables VI and VII. In Table VI, the uranium assay of sections remelted are shown by capital-letter designations, while the assay results taken between sections usable for coextrusions are numbered. Only the usable (numbered) assays were used for calculations of averages  $\bar{x}$ , grand averages  $\bar{X}$ , standard deviations, and ranges. There are no specified values for percent uranium in the core alloy, but the averages of the two adjacent samples were used to calculate what weight of the coextrusion core section is required to give the  $^{235}\text{U}$  content specified.

#### A. Ultrasonic Test

Shortly after the fuel-tube inspection was started, the Nuclear Metals ultrasonic inspection was not showing nonbond areas that were not also shown by blister testing. The ANL ultrasonic test was found to be more sensitive than that used at NMD. The NMD test was eliminated from the work under contract to avoid duplicate effort. Ultrasonic testing was retrained in the specification, however, for future fixed-price contracts.

TABLE V. Comparison of Specified vs Statistically Evaluated Data

Type of Fuel Tube Specified Values/Calculated from Data		I Inner		II Intermediate		III Outer		
		Specified or Nominal	Calculated from Data	Specified or Nominal	Calculated from Data	Specified or Nominal	Calculated from Data	
Core Length, in.	Nominal	$\bar{x}$ (of mean)	24.0625	23.8821	24.0625	23.7208	24.0625	23.6622
	Minimum	$\bar{x}_{\min.}$		22.926		22.648		22.967
		$\sigma_{\min.}$	21.8125	22.048	21.8125	21.867	21.8125	22.349
Maximum	$\bar{x}_{\max.}$		24.839		24.811		24.358	
	$\sigma_{\max.}$	26.3125	25.375	26.3125	25.301	26.3125	24.968	
Outside Diameter, in.	Nominal	$\bar{x}$ mean	2.236	2.236	2.640	2.642	2.112	2.113
		$\bar{x} - 2\sigma$	2.231	2.232	2.635	2.636	2.107	2.100
		$\bar{x} + 2\sigma$	2.241	2.240	2.645	2.648	2.117	2.128
Overall Length, in.	Nominal $\pm 1/32$	$\bar{x}$	27.5625	27.569	27.5625	27.569	32.1562	32.164
		$\pm 2\sigma$	0.0156	0.010	0.0156	0.023	0.0156	0.020
Bow, in.	Maximum	$\frac{\bar{x}}{\bar{x} + 2\sigma}$	0.025	0.029	0.025	0.028	0.025	0.030
Grams of $^{235}\text{U}$ (Core Wt x % U x % $^{235}\text{U}$ )	Nominal	$\bar{x}$	62.00	62.77	73.00	73.63	35.00	34.94
	Minimum	$\bar{x} - 2\sigma$	59.00	60.969	70.00	71.27	32.00	33.41
	Maximum	$\bar{x} + 2\sigma$	65.00	64.572	76.00	75.99	38.00	36.47
Grams of $^{234}\text{U}$ per set	Nominal Minimum Maximum	$x$			170.00	171.23		
		$x - 2\sigma$			168.00	168.74		
		$x + 2\sigma$			176.00	173.90		

TABLE VI. Evaluation of Uranium Assay Data for Inner and Intermediate Tubes

	Casting No.: AX 2698	AX 2818	AX 2703	AX 2850	AX 2829	AX 2843
Primary Extrusion:	1D	1E	2E	1F	2F	
Recycle Sections						
A	-	22.6	-	22.5	22.2	-
B	-	22.8	-	21.2	22.9	-
C	-	22.2	-	22.0	23.6	-
D	-	-	-	-	-	23.3
E	-	-	-	-	-	23.6
Coextrusion Sections						
1	22.8	23.0	23.2	22.8	22.8	22.8
2	23.4	23.2	23.6	23.1	22.9	23.5
3	23.5	23.5	23.5	24.1 <sup>a</sup>	23.3	23.4
4	23.5	23.6	23.7	24.0 <sup>a</sup>	23.2	23.1
5	23.4	23.8	-	25.0 <sup>a</sup>	-	-
Coextrusion avg, $\bar{x}$	23.32	23.42	23.50	23.80	23.05	23.20
Range, $\Delta$	0.7	0.8	0.5	2.2	0.5	0.7
Total range, R				25.0 - 22.8 = 2.2		
Number of analyses of coextrusion sections, n				27		
Grand average coextrusions, $\bar{X}$				23.50 wt % U		
Standard deviation, $\sigma$				0.48 wt % U		
95% lower limit $\bar{X} - 2.05\sigma$				22.40 wt % U		
95% upper limit $\bar{X} + 2.05\sigma$				24.40 wt % U		

<sup>a</sup>Two sections were rejected because of the extreme variation of analysis of these samples.

TABLE VII. Evaluation of Uranium Assay Data for Outer Fuel Tubes

	Casting No.: AX 2725	AX 2833
Primary Extrusion:	3E	3F
	wt % U	wt % U
Coextrusion Sections		
1	16.1	16.2
2	15.8	16.2
3	16.1	16.3
4	16.0	16.3
5	16.5	16.4
6	15.8	16.1
Casting, $\bar{x}$	16.0	16.2
Range, $\Delta$	0.7	0.3
Number of analyses	12	
Grand average, $\bar{X}$	16.1 wt % U	
Standard deviation, $\sigma$	0.215 wt % U	
95% lower limit, $\bar{X} - 2.2\sigma$	15.63 wt % U	
95% upper limit, $\bar{X} + 2.2\sigma$	16.57 wt % U	

The following ultrasonic classification system for the CP-5 fuel tubes serves as a guide for the acceptance or rejection of the tubes.

### 1. Grade A Tubes

Grade A tubes are tubes suitable for reactor use, from an ultrasonic viewpoint, having no internal flaws.

A1: 100% inspection, unobscured, indicates no internal defects.

A2: 100% inspection impaired by the presence of surface marks, dimensional deformities, or other flaws external in nature. These flaws obscure the area immediately below them and could, therefore, possibly conceal an internal flaw. No internal defects are indicated in any unobscured area.

### 2. Grade B Tubes

Grade B tubes are questionable tubes having internal flaws indicated ultrasonically, but none accompanied by visible superficial evidence.

B1: Internal flaws occur at random points.

B2: Internal flaws form a definite pattern (lines, square, patch, etc.).

### 3. Grade C Tubes

Grade C tubes are generally unsatisfactory tubes, which have internal flaws with one or more flaws supported by the presence of a visible blister on the tube surface. These tubes will be rejected.

C1: Blisters occur at random points only.

C2: Blisters occur in a definite pattern (line, square, patch, etc.). The ultrasonic inspection summary for the 1970 fuel shipment is as follows:

	<u>A-1</u>	<u>A-2</u>	<u>B-1</u>	<u>B-2</u>	<u>C-1</u>	<u>C-2</u>
Inner fuel tube	18	1	8	9	1	0
Intermediate fuel tube	22	1	4	8	0	2
Outer fuel tube	16	0	6	11	1	1

B. Visual Inspection

The greatest process rejection was for visual defects, pits, tears, and striations on the surfaces of the extrusions. Where such defects were frequent, it was often possible to assign a cause. An isolated pit or scratch occurring on the extruded surface was more difficult to explain. Sometimes a small fragment of die lubricant or metallic particle would be found imbedded in the pit. This did not explain the isolated clean pits with smooth, rounded bottoms, or the minute tears that were sometimes seen. A developmental investigation of die-lubricant preparation and control might improve the surfaces. Very careful training and supervision of operators are necessary to prevent damage to the soft aluminum cladding of the fuel tubes.

Concentration. Fuel tubes that contain a low concentration of hydrogen are...  
 The greatest process rejection was for visual defects, pits, tears, and striations on the surfaces of the extrusions. Where such defects were frequent, it was often possible to assign a cause. An isolated pit or scratch occurring on the extruded surface was more difficult to explain. Sometimes a small fragment of die lubricant or metallic particle would be found imbedded in the pit. This did not explain the isolated clean pits with smooth, rounded bottoms, or the minute tears that were sometimes seen. A developmental investigation of die-lubricant preparation and control might improve the surfaces. Very careful training and supervision of operators are necessary to prevent damage to the soft aluminum cladding of the fuel tubes.

Outer Tube	Inner Tube	Extrusion Thickness	Core Thickness	Wall Thickness	Average D	Average ID
0.12 ± 0.01	0.08 ± 0.01	0.02 ± 0.005	0.01 ± 0.002	0.01 ± 0.002	0.004 ± 0.001	0.002 ± 0.001
0.10 ± 0.005	0.07 ± 0.005	0.01 ± 0.002	0.005 ± 0.001	0.005 ± 0.001	0.003 ± 0.001	0.001 ± 0.001
0.09 ± 0.005	0.06 ± 0.005	0.01 ± 0.002	0.005 ± 0.001	0.005 ± 0.001	0.003 ± 0.001	0.001 ± 0.001
0.08 ± 0.005	0.05 ± 0.005	0.01 ± 0.002	0.005 ± 0.001	0.005 ± 0.001	0.003 ± 0.001	0.001 ± 0.001
0.07 ± 0.005	0.04 ± 0.005	0.01 ± 0.002	0.005 ± 0.001	0.005 ± 0.001	0.003 ± 0.001	0.001 ± 0.001
0.06 ± 0.005	0.03 ± 0.005	0.01 ± 0.002	0.005 ± 0.001	0.005 ± 0.001	0.003 ± 0.001	0.001 ± 0.001
0.05 ± 0.005	0.02 ± 0.005	0.01 ± 0.002	0.005 ± 0.001	0.005 ± 0.001	0.003 ± 0.001	0.001 ± 0.001
0.04 ± 0.005	0.01 ± 0.005	0.01 ± 0.002	0.005 ± 0.001	0.005 ± 0.001	0.003 ± 0.001	0.001 ± 0.001

## APPENDIX A

Scope of Work. Specifications for  
CP-5 Fuel Element Subassembly Parts

- Title: Fuel subassembly parts for CP-5 tubular fuel elements.
- Number: 32 sets of three tubes as shown on drawing C-MD-20431 un-assembled; in addition, four (4) each of the intermediate and eight (8) each of the outer tubes.
- Composition: Fuel tubes must contain a core of aluminum-uranium alloy formed from uranium approximately 93% enriched in U-235. These core sections must have an extreme alloy variation of no more than  $\pm 1$  wt % U-235 between like tubes and no more than  $\pm 1/2$  wt % U-235 within one tube. The mass of U-235 in the core section of the inner fuel tubes (C-MD-20431) must be  $62.0 \pm 3$  grams. The mass of U-235 in the core section of the intermediate fuel tubes (C-MD-20431) must be  $73.0 \pm 3$  grams. The mass of U-235 in the core section of the outer fuel tubes (C-MD-20431) must be  $35.0 \pm 3$  grams.

Fuel tubes (thirty-two (32) sets only) must be delivered in matched sets such that the total mass of U-235 in each set of three tubes (inner, intermediate, and outer) must be  $170.0^{+6}_{-2}$  grams.

All fuel tubes are to be coextruded with 1100 aluminum cladding and integrally extruded end caps of aluminum-silicon alloy or an aluminum-magnesium alloy to give the same extrusion characteristics as the aluminum-uranium alloy.

## Dimensions:

	<u>Inner Tube</u>	<u>Intermediate Tube</u>	<u>Outer Tube</u>
Cladding Thickness	$.015 \pm .033$ "	$.015 \pm .003$ "	$.015 \pm .003$ "
Core Thickness	$.032 \pm .004$ " $.003$ "	$.032 \pm .004$ " $.003$ "	$.020 \pm .004$ " $.003$ "
Wall Thickness	$.062 \pm .003$ "	$.062 \pm .003$ "	$.050 \pm .005$ "
Average O.D.	$2.236 \pm .005$ "	$2.640 \pm .005$ "	$3.000 \pm .005$ "
Average I.D.	$2.112 \pm .005$ "	$2.516 \pm .005$ "	$2.900 \pm .005$ "

The inner and intermediate fuel tubes must be round to within plus or minus .005". The outer tube must be round to within plus or minus .007".

The bow in all tubes, measured as maximum deviation of the O.D. surface from a straight edge parallel to the longitudinal axis of the tube held so as to contact the tube at extreme ends and cannot exceed 0.025".

The maximum core length, measured from extreme locations of core material must be no more than 26-5/16". The mean mass distribution of the core material must be no less than 23-13/16".

The minimum length of full thickness core should be not less than 21-13/16". However, ANL will agree to take tubes which have a shorter minimum length of full thickness core providing that the core length measured between points where the core thickness is 90% of the core thickness at the longitudinal midpoint of the core is not less than 21-13/16". Densitometer readings will be the basis for arriving at the core thickness. Note: Exceptions could be made at the Laboratory's discretion to accept tubes which exhibit areas with less than 90% relative thickness core at the minimum core length providing the following conditions are met:

- A) The width of any such area shall be no greater than 1/4".
- B) The total width of such areas at the minimum core length is no more than 3/4" for the Intermediate Tube and the Outer Tube and no more than 1/2" for the Inner Tube.
- C) No area with less than 90% relative thickness core may extend more than 3/8" past the minimum core length position toward the center of the length of the tube.

Cladding thickness may be allowed to decrease to no less than 0.009" in the area outside the minimum full core length, and the core thickness can increase proportionately.

Other dimensions and requirements are included in drawing C-MD-20431, which is hereby incorporated into and made a part of Appendix C.

#### Surfaces:

The surface of the finished fuel tubes shall be free from seams, metallic or non-metallic inclusions, imbedded foreign material or visible blisters. No surface indentations, scratches, or pits deeper than 0.003" over the fuel portion or

0.005" at the non-fueled ends shall be permitted. The surface finish shall be as generated in the fabrication process. No operations such as grinding, machining, abrasive grit blasting or buffing to alter or improve the surface shall be permitted except as agreed upon by the Laboratory and, in any case, only provided that no more than 0.003" of surface material is removed.

**Testing:** All fuel tubes shall be blister tested at 500° by holding each as extruded tube for one hour in air without flux. Tubes exhibiting blisters must be rejected.

All fuel tubes shall be ultrasonically tested in the as extruded, blister tested condition to establish the presence of a metallurgical bond over the full length of the tube.

**Test Reports:** Each shipment of tubes must be accompanied by the following:

1. Radiographs of both ends of each individual fuel tube relating the location of the end of the core to the end of the tube. In cases where there is question as to minimum core length, densitometer data comparing core thickness at minimum core length locations must also be submitted.
2. Data sheets containing the following information for each tube:
  - A. Identity number
  - B. End seal length
  - C. Maximum core length
  - D. Minimum core length
  - E. Shift and taper at each end
  - F. Blister test results
  - G. Ultrasonic test results
  - H. U-235 loading
  - I. Inspection remarks.

**Marking:** Each tube must be marked with its identity number at one end.

## APPENDIX B

ARGONNE NATIONAL LABORATORY  
9700 South Cass Avenue  
Argonne, Illinois 60439

SPECIFICATION  
RF-001a CP-5

TECHNICAL SPECIFICATION for the FABRICATION of  
ALCLAD AL-U ALLOY FUEL TUBES  
CP-5 FUEL ELEMENT SUBASSEMBLIES

Prepared by A. B. Shuck  
August 29, 1969  
Revision 1  
October 2, 1969  
Revision 2  
January 29, 1970

 <small>U of C-AUR-USAC</small>	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 0	Approved	Date 8/29/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>1</u> of <u>16</u>		

## TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page</u>
1.0	Scope.....	2
2.0	Applicable Documents.....	2
3.0	Special Conditions.....	3
4.0	Technical Requirements.....	4
5.0	Quality Control.....	13
6.0	Reports and Records.....	15
7.0	Delivery.....	16

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 2	Approved	Date 1-29-70
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>2</u> of <u>16</u>		

### 1.0 Scope

- 1.1 This specification defines the requirements for processing, fabricating, quality control, and shipment of aluminum-clad, aluminum-uranium alloy fuel tubes to be used in CP-5 fuel element subassemblies.
- 1.2 The fuel tubes shall consist of aluminum-uranium alloy tubular cores containing a nominal 23.3 (intermediate and inner tubes) and 16.4 w/o (outer tubes) of total uranium of 93% U<sup>235</sup> enrichment. The cores shall be clad inside and outside with 1100 aluminum. The cladding shall extend beyond the ends of the fuel as shown by the drawing. The space shall be filled with aluminum-1 w/o magnesium alloy. The fuel, cladding, and filling shall be metallurgically bonded for heat transfer and leak-tightness.
- 1.3 The Contractor shall furnish samples of core materials as required by paragraph 5.3.4.

### 2.0 Applicable Documents

- 2.1 Standard Specification *ASTM-B 211-67 Aluminum Alloy Bars and Wires.*
- 2.2 Drawing No. *C-MD-20431, Rev. 2/67, CP-5 Fuel Tube (Mark V) Procurement Drawing.*
- 2.3 RDT Standard *RDT-F-2-4T, Quality Verification Program Requirements (October, 1969).*

 <small>U S C A M - U S A E C</small>	A R G O N N E N A T I O N A L L A B O R A T O R Y		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>3</u> of <u>16</u>		

### 3.0 Special Conditions

- 3.1 The contractor shall be responsible for the development of, and adherence to, written fabrication, analysis, and inspection procedures and quality control plans. All such procedures and plans shall be submitted to Argonne National Laboratory for review, comment, or rejection prior to fabrication of the fuel tubes.
- 3.2 The contractor shall be responsible for providing all chemical analyses, measurements, and inspections required by the specification. The contractor shall provide certified process inspection and analysis records traceable to individual fuel tubes on forms approved by the Laboratory. The contractor shall provide process procedures, inspection procedures, and a quality control plan as described in Section 5.0 of this specification.
- 3.3 The Laboratory's representatives and inspectors representing the Laboratory shall be permitted free access at all times, while work under this contract is being performed, to the portions of the contractor's and subcontractor's plants and laboratories engaged in the processing, manufacturing, inspection, analyzing or storing materials to be supplied under this specification. The Laboratory's representatives shall have the right to review all process, inspection and calibration records, to request recalibration of gauges and test equipment, and to reject any of these that do not meet the requirements of the specification at any time the work is being performed.
- 3.4 The Laboratory shall be notified in advance of the schedule of fabrication and inspection operations so that during the course of the work the Laboratory's representative can observe any fabrication or inspection operations. Argonne National Laboratory will designate certain processes and inspections that it will witness.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 0	Approved	Date 8/29/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>4</u> of <u>16</u>		

- 3.5 The contractor shall provide schedules for the supplying of government or Laboratory furnished materials and manufacturing and delivery schedules on a Laboratory-approved form. Each schedule shall include status columns to be filled in showing weekly requirements of materials, production and delivery progress against the schedule. A copy of the updated schedule shall be submitted each month with the contract status report.

#### 4.0 Technical Requirements

##### 4.1 Design

- 4.1.1 The CP-5 reactor is a research reactor used at Argonne National Laboratory for Physics and materials experiments. The CP-5 fuel element consists of a central section having three concentric fuel tubes, a lower section, which engages the reactor bottom grid and provides flow control of the heavy-water coolant-moderator and an upper section containing the flow discharge openings. The element is attached to the reactor shield plug at the top end. Experiments and samples may be placed in the thimble within the innermost fuel tube.

 <small>UIC/MA-USAC</small>	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10-2-69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page 5 of 16		

4.1.2 The fuel tubes supplied in accordance with this specification shall contain uranium of approximately 93%  $U^{235}$  enrichment. The uranium shall be in aluminum-uranium alloy tubular cores clad inside and outside with 1100 aluminum. The cladding shall extend beyond the ends of the cores and the space at the core ends shall be filled with aluminum-1 w/o magnesium alloy. The cores and cladding shall be metallurgically bonded.

4.1.3 The dimensions and the  $U^{235}$  content of the fuel tubes shall be as follows:

TABLE I. Dimensions and  $U^{235}$  Content of Fuel Tubes

All Dimensions in Inches	Inner Tube	Intermediate Tube	Outer Tube
a Cladding Thickness <sup>1</sup>	0.015 ± 0.003	0.015 ± 0.003	0.015 ± 0.003
b Core Thickness <sup>1</sup>	0.032 + 0.004 - 0.003	0.032 + 0.004 - 0.003	0.020 + 0.004 - 0.003
c Wall Thickness	0.062 ± 0.003	0.062 ± 0.003	0.050 ± 0.005
d Avg. Outside Dia.	2.236 ± 0.005	2.640 ± 0.005	3.000 ± 0.005
e Avg. Inside Dia.	2.112 ± 0.005	2.516 ± 0.005	2.900 ± 0.005
f Roundness (max. O.D.-min. O.D.)	0.010	0.010	0.014
g Bow, max.	0.025	0.025	0.025
h Core Length			
1)@ 90% Thickness min. <sup>2</sup>	21-13/16	21-13/16	21-13/16
2)Maximum	26-5/16	26-5/16	26-5/16
i $U^{235}$ Content, grams	62.0 ± 3.0	73.0 ± 3.0	35.0 ± 3.0
j $U^{235}$ Content of a Matched Set, grams		170.0 + 6.0 - 2.0	

<sup>1</sup> Thickness of core and cladding are not directly measurable but are inferred from extrusion reductions and core areas as measured from X-radiographs.

<sup>2</sup> See paragraph 4.3.4.2 for definition of 90% of full thickness.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>6</u> of <u>16</u>		

## 4.2 Materials

### 4.2.1 Aluminum

- 4.2.1.1 Aluminum used for inner and outer cladding shall be 1100 aluminum of 99.0 w/o purity or better.
- 4.2.1.2 Virgin aluminum used for core alloy production shall be of not less than 99.95 w/o pure.
- 4.2.1.3 Aluminum used for end-seal alloy preparation shall be of not less than 99.5 w/o pure.
- 4.2.1.4 The spectrochemical analyses of all aluminum used shall be furnished and shall include determination of at least the following elements: silicon, iron, copper, manganese, magnesium, zinc, cadmium, boron, hafnium, cobalt, and silver.

### 4.2.2 Uranium

- 4.2.2.1 Uranium will be furnished by the Laboratory. The uranium will be of approximately 93%  $U^{235}$  content in the form of reduction buttons. Only uranium that is supplied by the Laboratory or approved by the Laboratory shall be used for this work.
- 4.2.2.2 Chemical and isotopic analyses will be provided with the uranium, which shall be used in calculation of alloy compositions. Alloy compositions and core weight shall be prepared on the basis of the specified  $U^{235}$  content of the fuel element, and on the analyses of the alloy, not on the nominal compositions.
- 4.2.2.3 The contractor shall be fully responsible for maintaining nuclear and radiological safety in accordance with established and AEC approved procedures.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a (CP-5)		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page 7 of 16		

#### 4.3 Fabrication Requirements

##### 4.3.1 Alloy Preparation

The aluminum-uranium alloy shall be prepared by comelting aluminum and uranium. Superheat and time at temperature shall be controlled to completely dissolve the uranium in the aluminum. To prevent alloy segregation, the melt shall be cast at as low temperature as practical into chilled or water-cooled molds.

- 4.3.1.1 The contractor shall demonstrate that analysis of any one-gram sample taken from any location in a core alloy extrusion billet shall not deviate by more than 0.5 weight percent from the average analysis of that billet.
- 4.3.1.2 The range of average analyses of all billets of the same nominal composition made under this specification shall be within 0.7 weight percent in nominal composition.
- 4.3.1.3 The total of elements other than aluminum and uranium in the core alloy shall not exceed 5000 ppm by weight of which iron shall not exceed 1500 ppm.
- 4.3.1.4 Argonne National Laboratory will evaluate fuel element impurities in terms of equivalent boron content EBC. An equivalent boron content greater than 5 ppm of core alloy or cladding will be cause for rejection. Equivalent boron will be calculated by multiplying ppm of impurities by the following factors and summing the results:

 <small>U of C-ANL-USAE</small>	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 2	Approved	Date 1-29-70
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page 8 of 16		

$\phi^2$	4.3.1.5	Al	NA	F	0.000001	P	0.000092
		Ba	0.000125	Fe	0.000672	Si	0.000082
		Be	0.000016	Pb	0.000012	Ag	0.008368
		B	1.00000	Sm	0.533741	Sn	0.000075
		Ca	0.000157	Eu	0.405388	W	0.001496
		Cd	0.312325	Mg	0.000041	V	0.001406
		Ga	0.000575	Mn	0.003443	Zn	0.000241
		Cl	0.013660	Mo	0.000403	Gd	4.19
		Cr	0.000854	Ni	0.001201	Dy	0.083770
		Co	0.009239	N	0.001923	Zr	0.000029
		Cu	0.000868	O	0.000000	Hf	0.008429

These factors are based upon microscopic absorption cross sections for 2200 m/sec neutrons as tabulated in ANL-5800 (2nd ed.) Table 2-1, pp. 30-31.

- 4.3.1.6 If the contractor can show evidence that any of these elements are not present in the feed materials, nor introduced by the process in amounts that raise the total impurity content of the finished elements to exceed 5 ppm boron equivalent, the contractor may request permission from the Laboratory to waive analysis for the specific elements.

#### 4.3.2 Coextrusion

- 4.3.2.1 All components of the coextrusion assemblies shall be fully inspected for process suitability, shall have clean, machined surfaces, and shall be free from inclusions, laps or foreign matter. Spot grinding of cores may be done to remove local included matter.
- 4.3.2.2 The extrusion components shall be preshaped to compensate for distortion of the core ends.

$\phi^2$  Factors are recalculated with cross-section value from ANL-5800. Paragraph 4.3.1.6 was added.



ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 2	Approved	Date 1-29-70
For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>9</u> of <u>16</u>		

$\phi^2$  4.3.2.3 The core thickness between the full thickness section<sup>3</sup> and core end shall be not greater than 0.042 in. for inner and intermediate tubes and not greater than 0.032 in. for outer fuel tubes providing that reasonable assurance can be provided that clad thickness is not less than 0.009 in.

4.3.2.4 Surface Finish

The inner and outer surfaces of the fuel tubes shall be free from seams, laps, die lines or extrusion defects such as scaling. No scratches nor roughness greater than 0.003 in.-deep over the fuel, nor greater than 0.005 in.-deep over the nonfuel ends will be acceptable. The overall surface roughness shall be not more than 125-microinches RMS except for local areas accepted by the Laboratory Representative. The surface finish shall be generated by the extrusion process. No operations such as machining, grinding or abrasive grit blasting shall be permitted. Polishing to improve surface finish shall be permitted only as agreed upon by the Laboratory Representative, and shall remove no more than 0.003 in. of surface material.

<sup>3</sup> As defined in paragraph 4.3.4.2.

$\phi^2$  Paragraph 4.3.2.3 and the beginning of 4.3.2.4 were added to this page.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 0	Approved	Date 8/29/69
	For	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>10</u> of <u>16</u>		

4.3.3 Each fuel tube shall be radiographed to show (1) the location of the core ends, (2) x-ray density as a measure of core thickness and, (3) local segregation of U-Al compounds.

4.3.3.1 The contractor shall radiograph each fuel tube by rotating the tube on a film-wrapped drum under a collimated x-ray beam. The film shall overlap at least 1/2 inch and the overlap shall be under the engraved identification arrow and numbers. A standard of known core thickness and penetrometer for calibration of the densitometer shall be included in each x-radiogram.

4.3.3.2 X-ray standards and penetrameters used for densitometry shall be subject to Laboratory approval.

4.3.4 Each fuel tube shall be cut to the lengths listed in Table II. The core positions, and lengths shall be as shown.

Table II, Tube, Core, and End Plug Length

Fuel Tube	Tube Length In.	Core Length, Inches		End Length Min. In.
		Min. 90% Thickness	Max. Length	
Inner	27-9/16 ± 1/32	21-13/16	26-5/16	1/2
Intermediate	27-9/16 ± 1/32	21-13/16	26-5/16	1/2
Outer	32-5/32 ± 1/32	21-13/16	26-5/16	1-3/8 Top 3-7/8 Bottom

4.3.4.1 The tube length shall be measured to an accuracy of 1/64 in.



ARGONNE NATIONAL LABORATORY

Specification No.

RF-001a CP-5

Title: ALCLAD AL-U ALLOY FUEL TUBES

Rev.

Approved

Date

1

10/2/69

For: CP-5 FUEL ELEMENT SUBASSEMBLIES

Page 11 of 16

4.3.4.2 The minimum length at greater than 90% of full thickness core shall be determined from x-radiograms by comparison with visual standards or densitometry. Full thickness is defined as a core thickness of  $0.032 + 0.004$  for inner and intermediate tubes and  $0.020 + 0.004$  for outer fuel tubes.

4.3.4.3 The maximum core length shall be measured from the extreme locations of core material.

#### 4.3.5 Measurements

4.3.5.1 Wall thickness measurements shall be taken by tube micrometer to read wall thickness over the end filler. Deep-throat micrometers shall be used to measure wall thickness over the cores. The maximum and minimum readings shall be recorded at each end and over the core material.

4.3.5.2 The average outside diameter shall be measured by taking three micrometer readings at  $60^\circ$  intervals at each end of the tube and four readings at  $45^\circ$  intervals at the center of the tube. The 10 readings shall be averaged. The average, maximum, and minimum readings shall be recorded.

4.3.5.3 Inside diameter readings shall be taken at the ends of the tube only, or may be derived from outside diameter and wall thickness measurements taken at corresponding locations.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page 12 of 16		

- 4.3.6 The maximum bow of any tube shall not exceed 0.025 in. Bow shall be measured as 1/2 maximum indicator runout when the tube is rotated supported on V-blocks at the ends. Alternately bow shall be measured as deviation of outside of tube from a straight edge.
- 4.3.7 Care shall be taken in all handling and gauging operations to avoid scratching or marking the tubes.

#### 4.4 Blister Tests

- 4.4.1 Each tube shall be blister tested by heating in an air furnace without flux for one hour at  $500^{\circ} \pm 25^{\circ}\text{C}$ . The tubes shall be supported during heating as required to prevent distortion.
- 4.4.2 A visible blister on the inside or outside surface shall be cause for rejection.
- 4.4.3 All tubes shall be ultrasonically tested by the immersion techniques. A focused ultrasonic beam shall be sent into the tube wall and the resulting reflections displayed on a cathode ray tube. Flaw locations shall be recorded in a manner that allows nonbonded areas to be located on the tubes. Ultrasonic test equipment shall be approved by the Laboratory. Nonbonded areas larger than that of a 1/16 in. diameter standard shall be cause for rejection.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>13</u> of <u>16</u>		

#### 4.5 Fuel Tube Handling

- 4.5.1 All personnel handling fuel tubes shall wear clean, lintless gloves. All surfaces contacting the fuel tubes during handling shall be clean and free from oil, dirt, and grit.
- 4.5.2 Extreme care and control shall be exercised at all times to prevent damage to the fuel tubes. All surfaces contacting the fuel tubes during handling shall be smooth, free from sharp edges and lined with clean teflon, felt or polyethylene to eliminate the possibility of fuel tube marring. Micrometers and gauges shall be used with care to prevent damage to the fuel tubes.

#### 4.6 Marking

Each fuel tube shall have an identification number that shall consist of a Laboratory supplied prefix number and a contractor supplied serial number that will provide traceability to process data. The number shall be engraved or applied by "vibra-tool" on the top inside surface over end seal material.

#### 5.0 Quality Control

- 5.1 The contractor shall produce the fuel tubes in accordance with written procedures for alloy production, fabrication, analyses, inspection tests, quality control, and shipping. These procedures shall be submitted to Argonne National Laboratory for review, comment, or rejection prior to start of fabrication of the fuel element.
- 5.2 The contractor shall maintain and verify the quality of the fuel tubes supplied under this specification in compliance with the requirements of RDT F2-4T AEC RDT Standard, Quality Verification Program Requirements.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>14</u> of <u>16</u>		

5.3 The contractor shall submit a Quality Control Plan containing the following elements as a minimum.

- 5.3.1 Project and quality control organization.
- 5.3.2 Inspection and control of contractor procured materials.
- 5.3.3 Control of materials furnished by the Laboratory or the AEC.
- 5.3.4 Any special gauges or fixtures, and the calibration maintenance and control of gauges.
- 5.3.5 Process controls (such as melt, extrusion and test temperature, analytical controls, in process inspections).
- 5.3.6 Inspection control, audit and certification of analytical and inspection reports.
- 5.3.7 Preservation, handling and packaging controls for process supplies and fabricated items.
- 5.3.8 Data report forms, data evaluation.
- 5.3.9 Deficiency analysis and corrective action.
- 5.3.10 Control of nonconforming materials.

5.4 Qualification of Process

- 5.4.1 After completion of process development and before start of the remaining production, the contractor shall submit for Laboratory approval or comment, three fuel tubes of each size to be manufactured under this specification. These fuel tubes shall be accompanied by any process development reports, and complete process, analytical and inspection data. The contractor shall certify the data to be in compliance with this specification.

	ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
	Title:	ALCLAD AI-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
	For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>15</u> of <u>16</u>		

- 5.4.2 The Laboratory will evaluate the data and the fuel elements and will comment or authorize production of the remaining fuel elements ordered.
- 5.4.3 Throughout the production run, the contractor shall maintain quality, qualify surveillance over processes, inspections, and handling procedures, and shall perform a deficiency analysis and correction function.
- 5.4.4 The contractor shall furnish two four- to-eight-gram samples from each lot of core alloy. The samples shall be clean part-off turnings or facings identified with the adjacent extrusion billets.

#### 6.0 Reports and Records

- 6.1 No shipment of fuel tubes will be considered acceptable for record purposes unless radiographs, inspection reports, ultrasonic test results, and analyses and all other data required by this specification, or by the contract, have been delivered to the Laboratory or accompany the shipment.
- 6.2 The forms on which such data is submitted shall be subject to approval by the Laboratory representative.



ARGONNE NATIONAL LABORATORY		Specification No. RF-001a CP-5		
Title:	ALCLAD AL-U ALLOY FUEL TUBES	Rev. 1	Approved	Date 10/2/69
For:	CP-5 FUEL ELEMENT SUBASSEMBLIES	Page <u>16</u> of <u>16</u>		

7.0 Delivery

7.1 Preparation for Delivery

- 7.1.1 After inspection, fuel tubes shall be washed clean and dried. All cleaning agents shall be subject to Laboratory review.
- 7.1.2 The fuel tubes shall be packed in approved shipping cases. Fuel tubes shall be supported on shock-resistant spacers, which will prevent relative motion and will prevent marring, or distortion during shipment.
- 7.1.3 The shipping containers shall meet all applicable regulations or license requirements of the Atomic Energy Commission and Department of Transportation.
- 7.1.4 The shipping containers' design shall be submitted to Argonne National Laboratory, and ANL approval shall be received by the contractor prior to shipment of fuel tubes.

7.2 Shipment

- 7.2.1 Shipment of fuel shall be made to:  
Argonne National Laboratory  
9700 South Cass Avenue  
Argonne, Illinois 60439  
Attention: Special Materials Division
- 7.2.2 Copies of all records shall be sent to Argonne National Laboratory to the attention of the Contract Administrator or his designated representative.

## APPENDIX C

Determination of Uranium (10-30%) in Aluminum by Titration  
with Metrohm Potentiograph for CP-5 Fuel Alloy1. Background

After a sample in solution in sulfuric acid is obtained, the uranium is reduced to a mixture of +3 and +4 oxidation states with zinc amalgam. Complete conversion to the +4 state is obtained by aeration. Ferric chloride and phosphoric acid are added as catalysts to permit room temperature titration. Titration is conducted on the potentiograph, using potassium dichromate (0.2 N) to oxidize the +4 uranium to the +6 state.

For enriched uranium, the volumetric factor will depend on the ratio  $^{235}\text{U}/^{238}\text{U}$ . Iron is the only interference in the titration (1 mg Fe  $\div$  2.13 mg U). For greatest precision, iron can be determined separately by O-phenanthraline and a suitable correction made. For CP-5, it should be necessary to determine the iron only on each casting (three samples) and apply the derived correction to subsequent core samples.

2. Method

a. Place  $1 \pm 0.05$  g of degreased chips in a 250-ml beaker. Add 20 ml of 1:1  $\text{H}_2\text{SO}_4$  + 20 ml  $\text{H}_2\text{O}$ ,\* and apply low heat to promote reaction. After the reaction slows down, cool, add five drops 1:1 HCl, and leave on low heat overnight. Cool, wash down the sides of beaker, add several drops of concentrated  $\text{H}_2\text{O}_2$ , heat to gentle boil, hold 15 min to destroy excess peroxide, and cool. (The result should be a bright yellow solution.)

b. Dilute to 125 ml, heat to boiling, add 2%  $\text{KMnO}_4$  dropwise to permanent pink (destruction or organic), and let cool to room temperature.

c. Put 10 g of 20-mesh amalgamated zinc in a 400-ml beaker, and pretreat with 100 ml of 5%  $\text{H}_2\text{SO}_4$  with magnetic stirring. Decant and wash. Transfer the sample solution to this beaker, and stir magnetically for 1/2 hr with the beaker covered (reduction of  $\text{U}^{+6}$  to  $\text{U}^{+4} + \text{U}^{+3}$ ). Decant the solution back into the sample beaker through a glass wool filter and wash/decant amalgam twice into sample beaker (dark green solution).

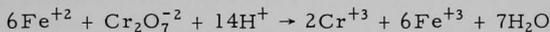
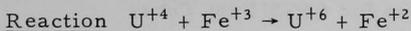
d. Aerate the solution for 1/2 hr in vacuum filtrators, slow bubbling rate ( $\text{U}^{+3} \rightarrow \text{U}^{+4}$ ) (light green solution). Be sure the room temperature is below 75°F.

e. Move the beaker to the titration stand, start the magnetic stirrer, and add 20 ml of 4% ferric chloride solution (prepared daily, mix cold) (catalyst) (solution yellow). Then add 15 ml of a mixture of two parts concentrated  $\text{H}_3\text{PO}_4$  and one part concentrated  $\text{H}_2\text{SO}_4$  (complex excess ferric ion).

\*For  $\text{U}_3\text{O}_8$  + 99.99 Al standard sample, omit 20 ml  $\text{H}_2\text{O}$  in dissolving solution.

f. Insert the electrodes, cover the beaker with a split watch glass, and wait for constant potential (-1000 mV measuring range, +1000 mV compensation). Titrate, using 20-ml burette and 0.2N  $K_2Cr_2O_7$ . Carry out the entire titration at a speed of 2 with a platinum electrode referenced against a calomel cell.

Titer            200 mg U  $\Leftrightarrow$  ~8.5 ml 0.2N  $K_2Cr_2O_7$  (freshly prepared)  
                           ↑↑  
                           normal uranium



### 3. Standardization

Standardization is with NBS  $U_3O_8$  chemical standard 950a fired at 900°C. Approximately 270 mg  $U_3O_8$  (for 23% alloy) or 190 mg  $U_3O_8$  (for 16% alloy) is carried through the procedure, along with sufficient aluminum (low in iron) to comprise a 1-g sample.

APPENDIX D  
Inspection Sheets for CP-5 Fuel Tubes







APPENDIX E  
Data Sheets for CP-5 Fuel Tubes

JOB NO. 3360 000 005NUCLEAR METALS DIVISION  
CP-5 DATA SHEETSHIPMENT NO. 1  
DATE 12-30-69

D R N U O. M	T U N B O. E*	T Y P E	FUEL LENGTH		END SEAL		BLISTER TEST	ULTRA- SONIC TEST **	LOAD Gms. U-235	T S O E T A L	INSPECTION REMARKS
			Max.	Min.	No. End	Un- No. End					
			(in)	(in)	(in)	(in)					
28	1D14	1	25.031	23.687	1.265	1.265	Passed	--	62.33		No serious defects on any of the tubes in shipment. Refer to individual inspection sheets.
	2D25	2	24.532	22.500	1.515	1.515	Passed	--	72.43		
	3D11	3	24.593	23.781	2.609	4.953	Passed	--	36.21	170.97	
31	1D13	1	25.250	22.687	1.156	1.156	Passed	--	61.97		
	2D32	2	24.687	22.906	1.437	1.437	Passed	--	72.83		
	3D34	3	24.469	23.000	2.671	5.015	Passed	--	35.88	170.68	
32	1D32	1	24.938	23.906	1.312	1.312	Passed	--	63.47		
	2D21	2	24.562	22.875	1.500	1.500	Passed	--	72.52		
	3D53	3	24.098	23.125	2.859	5.203	Passed	--	35.92	171.91	
33	1D12	1	24.968	23.407	1.297	1.297	Passed	--	61.60		
	2D33	2	24.594	23.344	1.484	1.484	Passed	--	73.06		
	3D12	3	24.844	23.156	2.484	4.828	Passed	--	35.84	170.50	
34	1D31	1	24.688	23.062	1.437	1.437	Passed	--	63.40		
	2D31	2	24.750	22.469	1.406	1.406	Passed	--	72.65		
	3D31	3	24.563	22.968	2.624	4.968	Passed	--	35.91	171.96	
35	1D11	1	24.688	23.562	1.437	1.437	Passed	--	61.35		
	2D34	2	24.688	23.406	1.437	1.437	Passed	--	73.32		
	3D51	3	24.094	23.250	2.859	5.203	Passed	--	35.74	170.41	

\* All tubes bear a 6-character identification number, consisting of "28" followed by the numbers listed in Column 2.

\*\*Ultrasonic test not performed.

JOB NO. 3360 000 005

NUCLEAR METALS DIVISION  
CP-5 DATA SHEET  
Sheet 1 of 2 sheets

SHIPMENT NO. 2  
DATE 1-15-70

D R N U O. M	T U N B O. E (1)	T Y P E	FUEL LENGTH		END SEAL		BLISTER TEST	ULTRA- SONIC TEST	LOAD Gms. U-235	T S O E T A L	INSPECTION REMARKS
			Max.	Min.	No. End	Un- No. End					
			41	1D42	I	24.750					
41	2E14	II	24.625	22.718	1.468	1.468	Accept.	---	75.29	-----	for detailed remarks on all tubes,
41	3D32	III	24.469	22.937	2.672	5.016	Accept.	---	35.86	174.30	noting remarks for tube #3D44 in particular
36	1B35	I	24.875	23.125	1.343	1.343	Accept.	---	62.77	-----	
36	2E15	II	24.750	22.750	1.406	1.406	Accept.	---	75.30	-----	
36	3D41	III	24.625	22.688	2.593	4.937	Accept.	---	36.01	174.08	
40	1B34	I	24.875	23.000	1.343	1.343	Accept.	---	62.56	-----	
40	2E12	II	24.782	23.187	1.390	1.390	Accept.	---	74.49	-----	
40	3D44	III	24.125	22.718	2.843	5.187	Accept.	---	35.61	172.66	
39	1C31	I	24.812	22.656	1.375	1.375	Accept.	---	61.31	-----	
39	2E34	II	24.688	22.500	1.437	1.437	Accept.	---	75.70	-----	
39	3E12	III	24.344	23.344	2.734	5.078	Accept.	---	34.08	171.09	
38	1C15	I	24.750	22.718	1.406	1.406	Accept.	---	61.86	-----	
38	2D23	II	24.688	22.281	1.437	1.437	Accept.	---	72.60	-----	
38	3D13	III	24.531	23.250	2.640	4.984	Accept.	---	35.80	170.26	
37	1D24	I	24.875	23.031	1.343	1.343	Accept.	---	63.18	-----	
37	2F11	II	24.812	23.032	1.375	1.375	Accept.	---	72.14	-----	
37	3D14	III	24.562	23.250	2.625	4.969	Accept.	---	35.67	170.99	
45	1D23	I	24.968	22.969	1.297	1.297	Accept.	---	63.21	-----	
45	2F12	II	25.187	22.750	1.187	1.187	Accept.	---	71.98	-----	
45	3D33	III	24.531	23.188	2.640	4.984	Accept.	---	35.99	171.18	

- (1) All tubes in shipment bear a 6-character identification number, consisting of "28" followed by the numbers listed in Column 2.
- (2) Ultrasonic test not performed.



JOB NO. 3360 000 005

NUCLEAR METALS DIVISION  
CP-5 DATA SHEET  
Sheet 1 of 2 sheets

SHIPMENT NO. 3  
DATE 2-13-70

D R N U O. M	T U B E (1)	T Y P E	FUEL LENGTH		END SEAL		BLISTER TEST	ULTRA- SONIC TEST (2)	LOAD Gms. U-235	T O T A L	INSPECTION REMARKS
			Max.	Min.	No. End	Un- No. End					
			28	1E25	I	25.093					
28	2F31	II	24.969	22.407	1.296	1.296	Accept.	----	73.62	-----	
28	3E31	III	24.062	23.000	2.875	5.219	Accept.	----	34.42	171.10	
31	1D33	I	24.750	23.063	1.406	1.406	Accept.	----	63.35	-----	
31	2E24	II	24.968	22.812	1.297	1.297	Accept.	----	75.26	-----	
31	3E21	III	24.469	23.000	2.671	5.015	Accept.	----	63.78	172.39	
33	1C32	I	24.875	23.313	1.343	1.343	Accept.	----	61.49	-----	
33	2F34	II	25.062	22.000	1.250	1.250	Accept.	----	73.48	-----	
33	3D42	III	24.718	23.156	2.547	4.891	Accept.	----	65.78	170.75	
34	1D41	I	25.062	22.500	1.250	1.250	Accept.	----	63.19	-----	
34	2F15	II	24.875	22.313	1.343	1.343	Accept.	----	72.27	-----	
34	3F22	III	24.437	22.875	2.687	5.031	Accept.	----	64.48	169.94	
35	1D21	I	25.157	23.156	1.562	0.843	Accept.	----	63.10	-----	Note unequal end seal lengths on 1D21
35	2F13	II	25.125	22.625	1.218	1.218	Accept.	----	72.20	-----	
35	3F23	III	24.125	23.125	2.843	5.187	Accept.	----	64.61	169.91	
32	1E42	I	24.875	23.532	1.343	1.343	Accept.	----	63.74	-----	
32	2F22	II	24.907	22.750	1.327	1.327	Accept.	----	72.52	-----	
32	3E41	III	24.156	22.875	2.828	5.172	Accept.	----	64.25	170.51	
47	1E44	I	24.594	22.750	1.484	1.484	Accept.	----	63.94	-----	
47	2F23	II	25.157	22.094	1.202	1.202	Accept.	----	72.88	-----	
47	3E14	III	24.844	22.781	2.484	4.828	Accept.	----	63.77	170.59	

- (1) All tubes in shipment bear a 6-character identification number, consisting of "28" followed by the numbers listed in Column 2.
- (2) Ultrasonic test not performed.

JOB NO. 3360 000 005

NUCLEAR METALS DIVISION  
 CP-5 DATA SHEET  
 Sheet 2 of 2 sheets

SHIPMENT NO. 3  
 DATE 2-13-70

D R N U O. M	T U N B O. E (1)	T Y P E	FUEL LENGTH		END SEAL		BLISTER TEST	ULTRA- SONIC TEST (2)	LOAD Gms. U-235	T S O E T A L	INSPECTION REMARKS
			Max.	Min.	No. End	Un- No. End					
			48	1E34	I	24.968					
48	2F21	II	25.031	21.813	1.265	1.265	Accept.	----	72.37	-----	
48	3F21	III	24.406	23.000	2.703	5.047	Accept.	----	34.47	170.45	
50	1E23	I	24.843	23.063	1.359	1.359	Accept.	----	62.84	-----	
50	2F32	II	25.156	22.125	1.203	1.203	Accept.	----	73.63	-----	
50	3F24	III	24.344	22.844	2.734	5.078	Accept.	----	34.76	171.23	
46	1F11	I	25.593	23.000	0.984	0.984	Accept.	----	61.36	-----	
46	2F33	II	24.844	22.656	1.359	1.359	Accept.	----	73.26	-----	
46	3F32	III	24.406	22.657	2.703	5.047	Accept.	----	34.86	169.48	
49	1F12	I	24.563	22.812	1.500	1.500	Accept.	----	61.37	-----	
49	2G22	II	24.969	22.750	1.296	1.296	Accept.	----	74.99	-----	
49	3F33	III	23.625	22.656	3.093	5.437	Accept.	----	34.76	171.12	

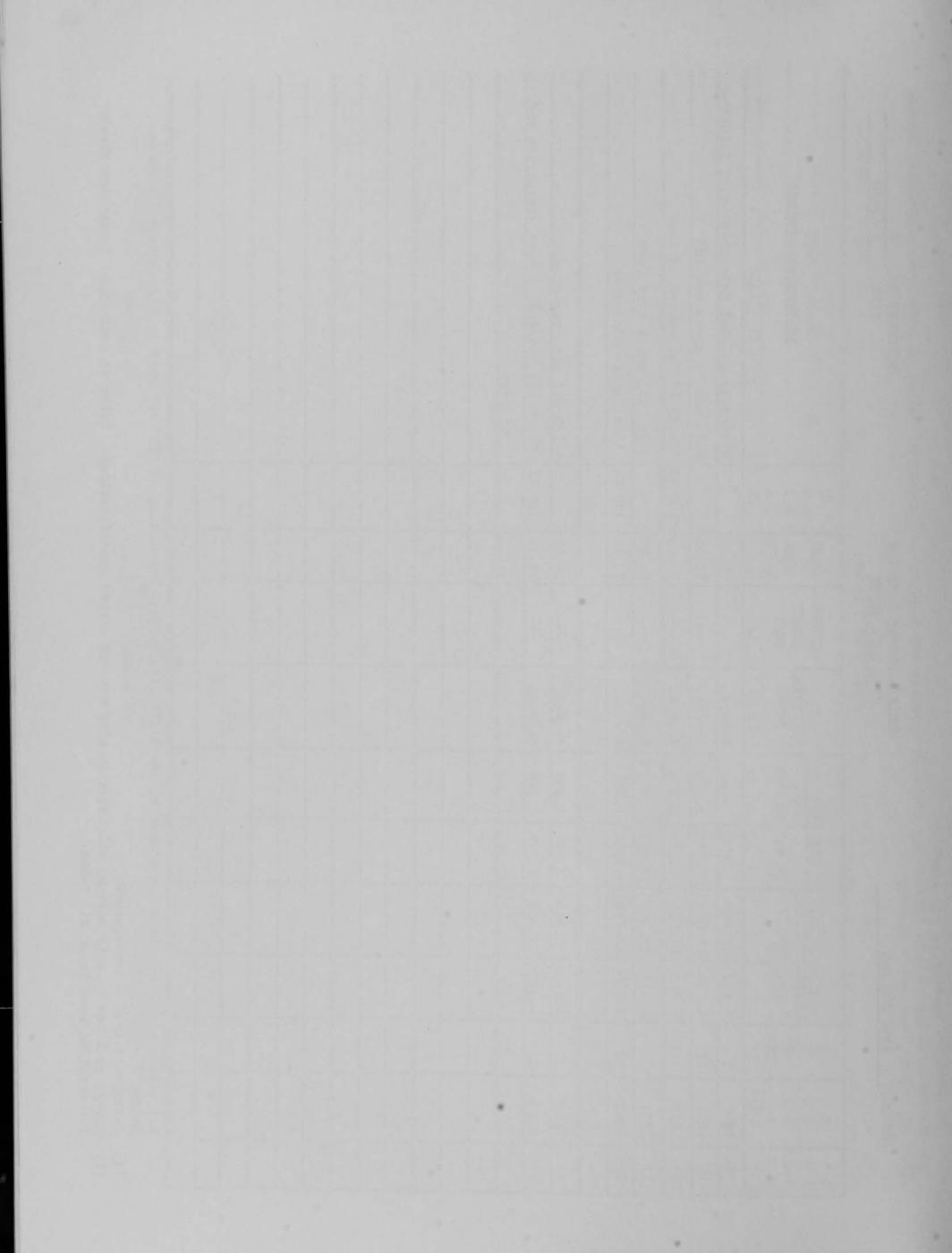
(1) All tubes in shipment bear a 6-character identification number, consisting of "28" followed by the numbers in Column 2.  
 (2) Ultrasonic test not performed.

JOB NO. 3360 000 005NUCLEAR METALS DIVISION  
CP-5 DATA SHEET  
Sheet 1 of 2 sheetsSHIPMENT NO. 4  
DATE 3-12-70

D R N U O. M	T U N B O. E (1)	T Y P E	FUEL LENGTH		END SEAL		BLISTER TEST	ULTRA- SONIC TEST (2)	LOAD Gms. U-235	T S O E T A L	INSPECTION REMARKS (3)
			Max.	Min.	No. End	Un- No. End					
43	1F24	I	25.188	21.875	1.187	1.187	Accept.	---	64.34		
43	2G32	II	24.750	22.906	1.406	1.406	Accept.	---	74.55		
43	3F44	III	24.375	22.813	2.718	5.062	Accept.	---	34.89	173.78	
39	1F15	I	24.594	22.875	1.484	1.484	Accept.	---	62.15		
39	2G25	II	24.969	22.625	1.296	1.296	Accept.	---	74.51		
39	3F14	III	24.812	23.031	2.500	4.844	Accept.	---	34.43	171.09	
38	1F14	I	24.531	22.844	1.515	1.515	Accept.	---	61.86		
38	2G31	II	24.625	22.688	1.468	1.468	Accept.	---	74.43		
38	3F12	III	24.593	22.844	2.609	4.953	Accept.	---	34.45	170.74	
42	1F13	I	24.563	22.656	1.500	1.500	Accept.	---	61.57		
42	2E13	II	24.968	23.094	1.297	1.297	Review	---	74.80		Blister midway on end seal
42	3F34	III	23.782	22.250	3.015	5.359	Accept.	---	34.75	171.12	
40	1E22	I	25.312	22.031	1.125	1.125	Accept.	---	62.70		
40	2G11	II	24.688	23.000	1.437	1.437	Accept.	---	72.18		
40	3F31	III	24.093	23.062	2.859	5.203	Accept.	---	34.74	169.62	5 mil pit over core
44	1F23	I	24.656	21.937	1.453	1.453	Accept.	---	63.84		
44	2C35	II	24.250	22.000	1.656	1.656	Accept.	---	73.44		3 1/2 mil indenta. inside end of core +0D pit
44	3E44	III	24.281	23.218	2.765	5.109	Accept.	---	35.16	172.44	5 mil pit over core
41	1F21	I	24.969	23.062	1.296	1.296	Accept.	---	62.57		
41	2G34	II	24.469	22.781	1.546	1.546	Accept.	---	74.00		3-3/4 mil gouge over core
41	3F13	III	24.282	22.812	2.765	5.109	Accept.	---	34.34	170.91	7 mil pit end seal; 3 1/4 & 3 1/2 mil pit core

- (1) All tubes in shipment bear a 6-character identification number, consisting of "28" followed by the numbers listed in Column 2.
- (2) Ultrasonic test not performed.
- (3) Except as noted in this column, all tubes in shipment are within specifications. Refer to individual inspection sheets for detailed remarks on all tubes.





ARGONNE NATIONAL LAB WEST  
[Barcode]

X

ARGONNE NATIONAL LAB WEST  
[Barcode]

X