

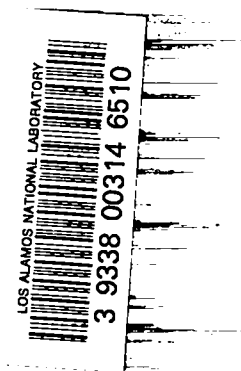
LA-3621

C.3

CIC-14 REPORT COLLECTION  
**REPRODUCTION  
COPY**

**LOS ALAMOS SCIENTIFIC LABORATORY**  
of the  
**University of California**  
LOS ALAMOS • NEW MEXICO

Preparation of a  
Plutonium-0.4 w/o Thulium Alloy  
on a Multikilogram Scale



UNITED STATES  
ATOMIC ENERGY COMMISSION  
CONTRACT W-7405-ENG. 36

## LEGAL NOTICE

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

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

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

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

This report expresses the opinions of the author or authors and does not necessarily reflect the opinions or views of the Los Alamos Scientific Laboratory.

Printed in USA. Price \$1.00. Available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, United States Department of Commerce, Springfield, Virginia

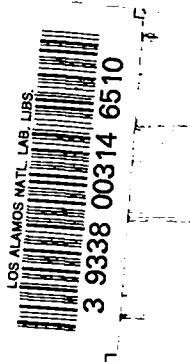
LA-3621  
UC-25, METALS, CERAMICS,  
AND MATERIALS  
TID-4500

**LOS ALAMOS SCIENTIFIC LABORATORY**  
**of the**  
**University of California**  
LOS ALAMOS • NEW MEXICO

Report written: September 1966

Report distributed: December 21, 1966

**Preparation of a**  
**Plutonium-0.4 w/o Thulium Alloy**  
**on a Multikilogram Scale**



Work done by:

D. R. Harbur  
J. W. Romero

Report written by:

D. R. Harbur  
J. W. Anderson  
W. J. Maraman



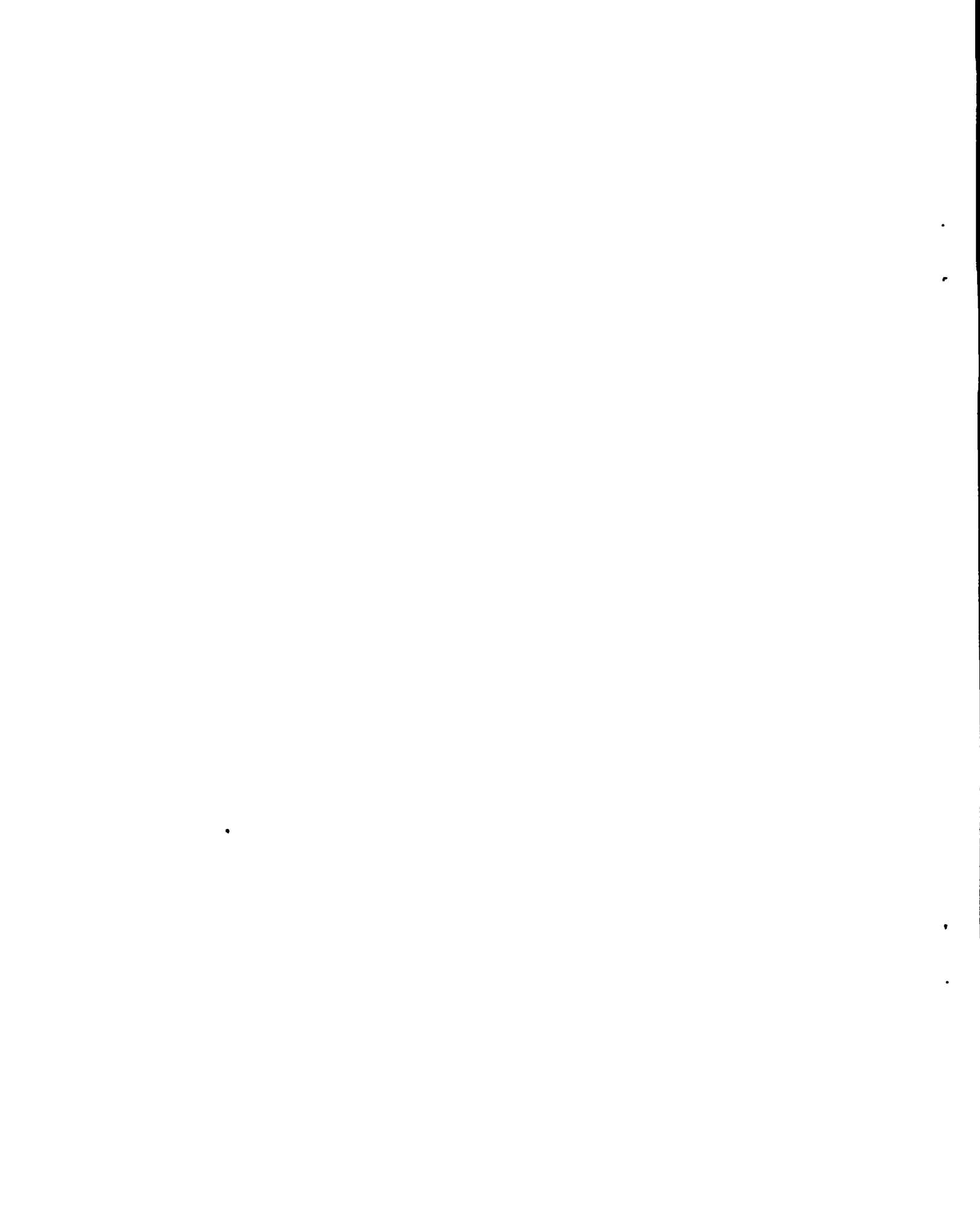


## ABSTRACT

The alloying time necessary to make a homogeneous plutonium-0.4 w/o thulium alloy can be lowered from several hours to less than 30 min by using a dash-pot stirring unit. To avoid macrosegregation of thulium in the casting, the alloy is chill cast into a mold at ambient temperature.

## ACKNOWLEDGMENTS

The authors are indebted to Group CMB-1 under the direction of C. F. Metz for the analytical chemistry.



## TABLE OF CONTENTS

	Page
ABSTRACT	3
ACKNOWLEDGMENTS .	3
INTRODUCTION	7
EQUIPMENT	7
PROCEDURE	9
RESULTS AND DISCUSSION	9
REFERENCES	10





## INTRODUCTION

During the preparation of a plutonium-0.4 w/o thulium alloy, two distinct alloying and casting problems were encountered. The first of these problems was caused by the density extremes between plutonium (19.5 g/cc) and thulium (9.3 g/cc), causing the thulium to form in a layer above the plutonium. Macro-segregation within the cast part was the other problem. These two alloying problems are by no means unique, but rather they are common problems encountered in alloying and casting many plutonium-base alloys.<sup>(1, 2)</sup> This report gives the alloying and casting procedures developed at the Los Alamos Scientific Laboratory for producing a homogeneous plutonium-0.4 w/o thulium alloy. These procedures are quite adaptable for preparing most plutonium-base alloys.

## EQUIPMENT

The equipment used to alloy and cast the plutonium-0.4 w/o thulium alloy is shown schematically in Fig. 1. It consists basically of a copper mold on top of a magnesia insulating stand inside the furnace can. Directly above the mold inside a Vycor tube is the tantalum bottom-pour melt crucible with an off-center tantalum stopper rod and the thulium and plutonium metals. The melt crucible is placed on a magnesia insulating stand. The stopper rod extends through the lid on top of the Vycor tube along with a tantalum dash-pot stirrer. The tantalum melt crucible is heated by an induction coil placed around the Vycor tube.

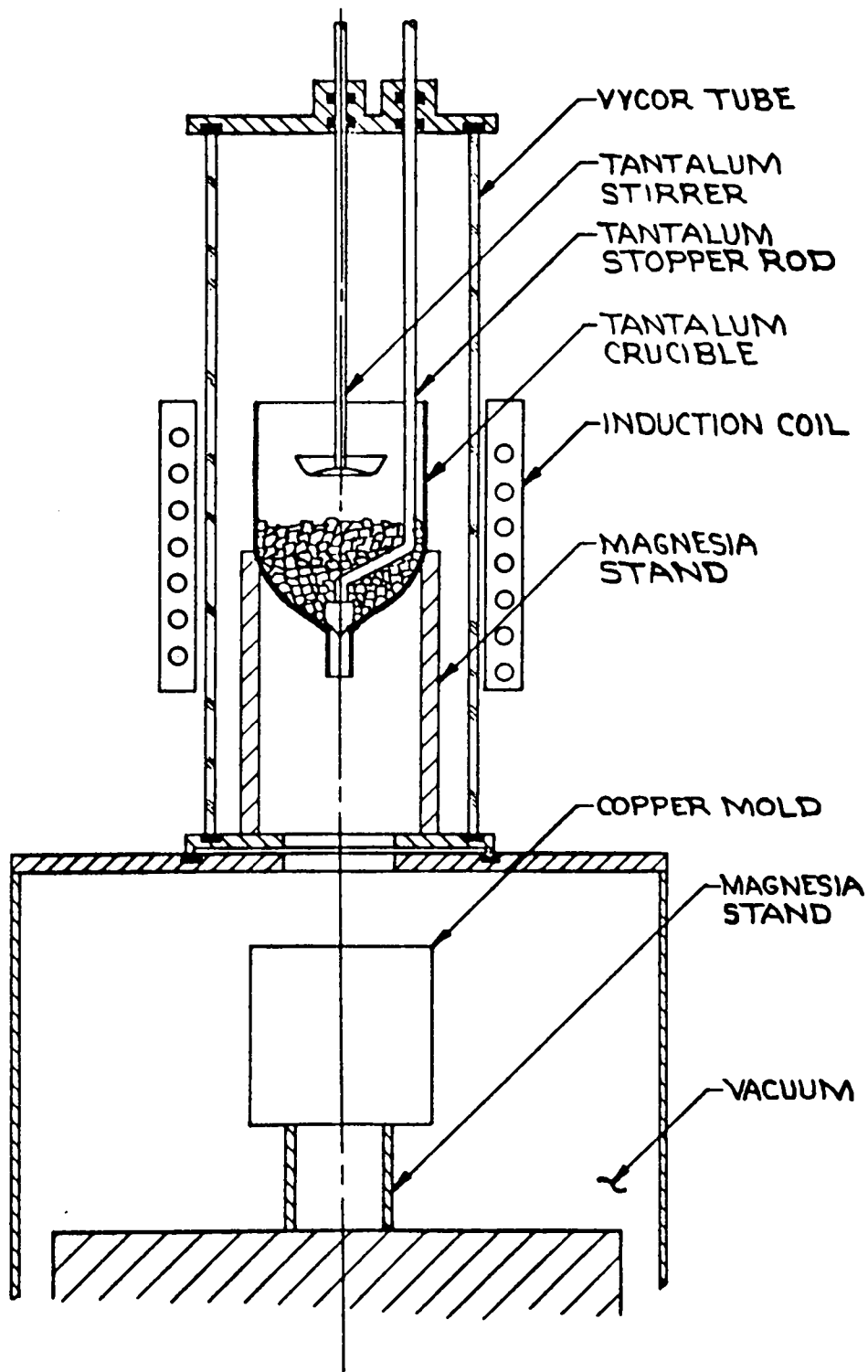


Fig. 1. Alloying and Casting Equipment.

## PROCEDURE

The alloying and casting equipment is assembled. To make a 0.4 w/o thulium alloy, it was found necessary to add 0.5 w/o thulium to the plutonium. After inductively heating the melt crucible to 925°C the melt is stirred up and down for at least 30 min. The copper mold is kept at ambient temperature. After alloying, the melt is bottom poured into the copper mold.

## RESULTS AND DISCUSSION

The largest ingot cast in this manner was a 2.25-in.-diameter, 3-in.-long billet weighing 3.2 kg. The thulium analysis from top, middle, and bottom were 0.41, 0.41, and 0.42 w/o, respectively. Alloying the plutonium and thulium with the aid of the dash-pot stirrer will produce a homogeneous melt in 30 min. Without any stirring device and using small clean chips of thulium it was found necessary to hold the melt at 700° to 950°C for several hours before a homogeneous melt could be achieved. <sup>(3)</sup>

It was found necessary to chill cast the alloyed melt into a copper mold at ambient temperature in order to retain a homogeneous alloy. Casting the melt into a graphite mold heated at 450° to 650°C produced severe macro-segregation in the final casting. The top of such a casting had 3.3 w/o thulium, while the bottom had only 0.1 w/o thulium.

Early in the development program the chemical analysis group (CMB-1) found that the normal methods used to analyze for most other rare earth elements did not produce satisfactory results with thulium. Therefore a new x-ray fluorescence spectrometric method was developed to analyze for thulium. <sup>(4)</sup>

## REFERENCES

1. Harbur, D. R., Romero, J. W., and Anderson, J. W., "Recovery of Mechanically Entrapped Plutonium From Casting Skulls and Turnings," Mod. Castings, 46, 3, 481 (1964).
2. Harbur, D. R., Romero, J. W., and Anderson, J. W., "Plutonium-Cerium-Cobalt Fuel Development for Molten Reactors," Mod. Castings, 48, 2, 124 (1965).
3. Bowersox, D. F. and Leary, J. A., "The Solubilities of Selected Elements in Liquid Plutonium, X. Thulium," Los Alamos Scientific Laboratory Report No. LA-3623 (1966).
4. R. G. Hurley, Hakkila, E. A., and Waterbury, G. R., "The X-ray Fluorescence Spectrometric Determination of Thulium in Plutonium-Thulium Alloys," Los Alamos Scientific Laboratory Report No. LA-3549 (1966).