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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

PYROMETALLURGY EXPERIMENTS ON PLUTONIUM-RICH REACTOR FUELS

(Project Status Report as of March 31, 1958)

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PYROMETALLURGY EXPERIMENTS
ON PLUTONIUM-RICH REACTOR FUELS

(Project Status Report as of March 31, 1958)

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ABSTRACT

A laboratory survey of various pyrometallurgical methods for purifying synthetic spent plutonium fuel, fissium, is being conducted. The methods that have been investigated are (1) liquation and filtration, (2) oxide drossing, (3) carbide slagging, (4) halide slagging, (5) chloride conversion cycle, (6) electrorefining, (7) recrystallization from mercury and (8) liquid-liquid metal extraction with calcium-magnesium eutectic.

By holding a melt of fissium for prolonged periods, insoluble fission products segregate either in elemental or compound form. Filtration of the liquated melts has indicated that zirconium, niobium, molybdenum and lanthanum are removed by this simple method.

Results obtained by oxide drossing, as well as by carbide and halide slagging, are qualitatively in agreement with thermodynamic arguments. Rare earth fission products are concentrated in the dross and halide slag, while zirconium is concentrated in a carbide slag. Slagging with chloride melts appears to offer the best removal of rare earth elements within the shortest time.

It is possible to remove zirconium, molybdenum and ruthenium by a chloride conversion cycle. In this method, the fissium was dissolved in a zinc chloride - sodium chloride melt and the resulting plutonium chloride - sodium chloride eutectic was filtered to remove the zinc. The salt phase contained 98 percent of the plutonium, almost all of the rare earth impurities and essentially no zirconium, molybdenum, ruthenium or zinc. Reduction of the plutonium(III) chloride in this melt has not yet been attempted.

Excellent purification of fissium has been achieved by electrorefining at 550°C in a lithium chloride - potassium chloride - plutonium chloride electrolyte. Results were in accordance with predictions based on standard free energies of formation of the chlorides. Zirconium, molybdenum and ruthenium remained in the impure fissium anode, while lanthanum and cerium concentrated in the salt phase but were not deposi-

ted with the plutonium product at the cathode. The purified product was collected as a liquid iron eutectic by using an iron cathode.

Measurements of the individual solubilities of typical fission product elements in mercury indicate that separation from plutonium by this method also may be feasible. The solubility of plutonium over the temperature range 20°C to 325°C has also been determined.

Extractions of fission products from fissium melts by liquid calcium-magnesium eutectic have indicated that this method does not have any particular merit for processing spent fuel.

FOREWORD

This report is essentially identical to a paper that has been submitted for inclusion in the proceedings of the International Conference on the Peaceful Uses of Atomic Energy to be held at Geneva in 1958. However, because the proceedings probably will not be published within the next year, this document is being distributed as a project status report at the present time. A previous report (LA-2132) summarizes the status of this project up to March 31, 1957.

ACKNOWLEDGEMENTS

All chemical analyses were conducted by members of Group CMB-1 under the direction of C. F. Metz. The advice of R. D. Baker on all phases of the project is also gratefully acknowledged.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF TABLES (continued)

INTRODUCTION

The general scope of this project was introduced in the previous status report, LA-2132. At the present time, all experiments are being conducted with the synthetic spent fuel "fissium".

PREPARATION OF FISSIUM ALLOY

The proposed initial fuel for the Los Alamos Molten Plutonium Reactor Experiment (LAMPRE)(1) is the 9.5 atomic percent iron-plutonium eutectic melting at 406°C. Consequently, this plutonium-rich fuel composition has been used to prepare a synthetic spent fuel for pyroprocessing studies. The fissium was prepared on a 1 kg. scale by dissolving representative fission product elements in molten iron-plutonium alloy at 1000°C. Each element was added in an amount that corresponded approximately to 10 percent burnup after 130 days of fast fission. After dissolution, the melt was chill-cast into segments weighing 80-100 g. each. The experimental arrangement for alloying and casting is shown schematically in Fig. 1. Chemical analyses of samples from each segment indicated that the fission products were distributed quite uniformly through the segments. A more detailed discussion of preparation and composition has been published previously.(2) The composition of a typical chill-cast segment is shown in Table 1.

Table 1

Composition of Iron-Fissium Alloy

Element	Concentration of Element, weight percent
Zr	0.82
Mo	0.74
Ru	1.28
La(a)	1.31
Ce	0.74
Fe	2.76
C	0.01
O	0.01
Pu (by difference)	92.33

(a) Excess lanthanum was added as a stand-in for all rare earth fission products except cerium.

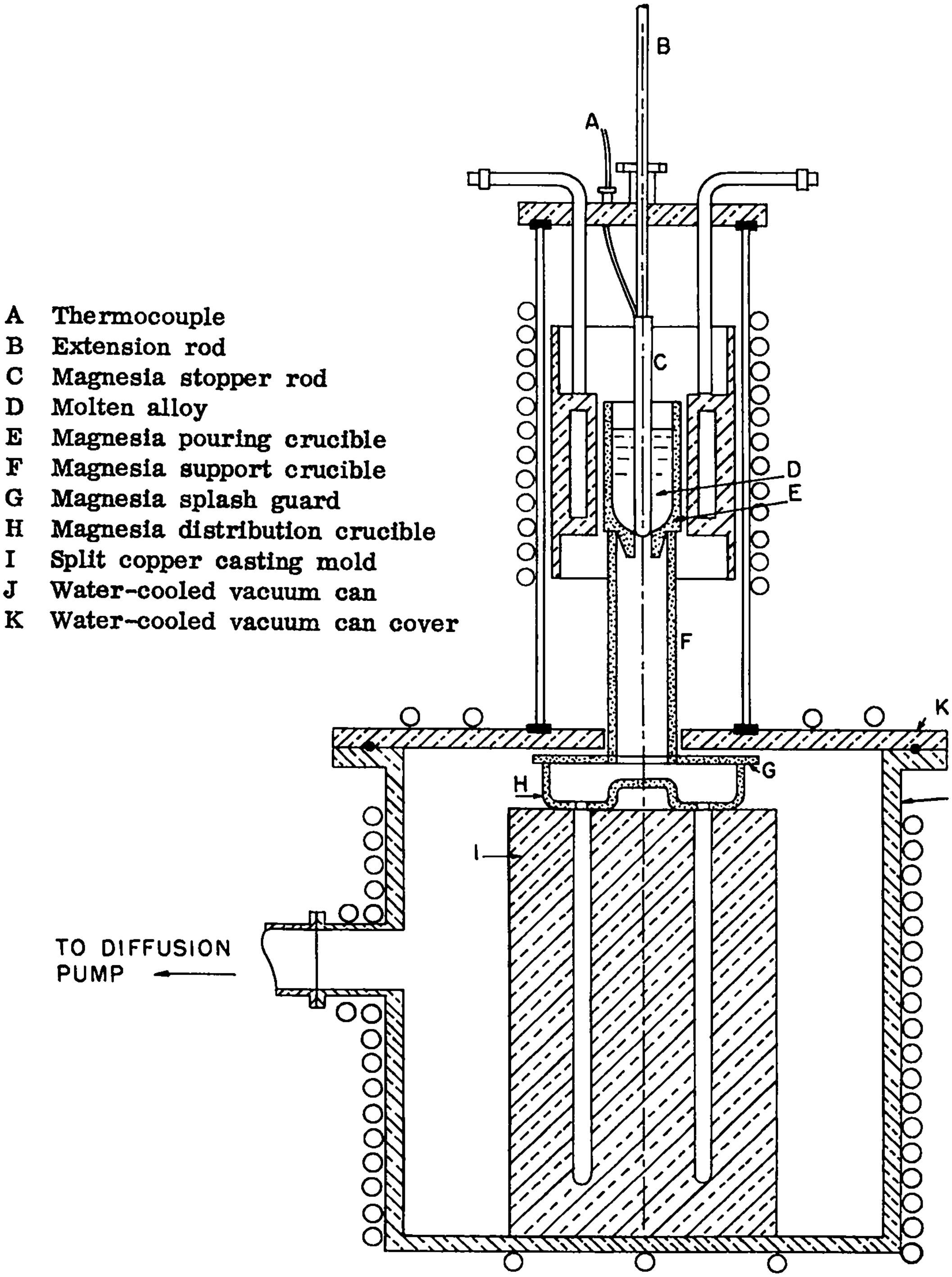

Figure 1. Vacuum Alloying and Casting Furnace

In addition to iron-plutonium fissium, a few experiments were conducted with fissium prepared from 12 atomic percent cobalt-plutonium base alloy melting at 410°C. The concentrations of added fission product elements were similar to those in the iron-fissium, except that no lanthanum had been added.

PYROMETALLURGICAL METHODS INVESTIGATED

1. Liquation and Filtration

If a fission product element is present in an amount that exceeds its solubility, it should be possible to effect a separation simply by holding the fissium in the molten state. This allows insoluble constituents to segregate, either in elemental or compound form, and they may then be separable by filtration.

A series of experiments was conducted wherein approximately 100-g. fissium melts were held at constant temperature in a tantalum crucible in a vacuum (10^{-7} atmosphere) resistance furnace. The melts were sampled periodically by filtering a 3-g. aliquot through a graphite filter plug fitted into the end of a 6-mm. quartz tube. In all samples, the filter tube was heated to the liquation temperature, then immersed in the melt for less than 1 min. Consequently, it is assumed that the composition of the liquid phase had not been altered significantly by reaction with the graphite filter in such a short time.

The results of these experiments are summarized in Table 2. These results are expressed in terms of C_o and C. C_o is the concentration of element before processing, expressed as grams of element per gram of plutonium and C is the concentration of element after processing. From samples taken after 100, 150 and 200 hr. (not shown in Table 2), it was apparent that liquation times of approximately 250 hr. are necessary to reach approximate equilibrium concentrations. However, residual concentrations of zirconium, molybdenum and lanthanum indicate that these elements may be removed to a large degree.

Table 2

Residual Fission Product Element Concentrations in Liquated Fissium as a Function of Temperature

Temperature, °C	Liquated Time, hr.	C/C_o					
		Zr	Nb	Mo	Ru	La	Ce
500	2.8	0.87	0.45	0.45	1.03	0.23	0.90
	250	0.16	0.15	0.16	0.90	0.06	0.79
550	2	0.03	..	0.26	0.92	0.19	0.90
	250	0.01	..	0.05	0.77	0.12	0.71
650	17	0.58	0.75	0.42	0.97	0.40	0.94
	260	0.18	0.36	0.32	0.92	0.35	0.88
800	3	0.60	1.03	0.80	1.02	0.89	1.09
	260	0.45	0.65	0.68	1.10	0.71	0.83

In one experiment 500 g. of fissium was liquated for 250 hr. at 600°C in a tantalum crucible, and the entire melt was then filtered. Although a yield of only 60 percent was obtained because of mechanical difficulties, the fission product removal shown in Table 3 was comparable to that obtained on the smaller scale.

Table 3

Residual Fission Product Element Concentration in Fissium Alloy After Liquation at 600°C and Filtration

(500-g. scale)

C/C_o						
Zr	Nb(a)	Mo	Ru	La	Ce	Fe
≤ 0.015	≤ 0.29	0.063	1.16	0.16	0.82	1.03

(a) Niobium concentration in fissium before processing was 0.030 weight percent.

2. Oxide Drossing

This method, which has been studied extensively with uranium fuels,(3a, 3b, 4) consists of holding the molten impure metal in a refractory oxide crucible for a prolonged period to accomplish selective oxidation of impurities. Fission product removal is effected by formation of an insoluble dross at the crucible-melt interface. Thermodynamic considerations indicate that strontium, barium and rare-earth elements should concentrate in the oxide phase while molybdenum and ruthenium should become concentrated in the melt.

Oxidative drossing experiments have been conducted with both iron- and cobalt-fissium alloys by melting approximately 80-g. charges in dry-pressed magnesia crucibles and holding the melt at temperature in a vacuum induction furnace at a pressure of 10^{-7} atmosphere. The ratio of crucible area that was wetted by the melt to the weight of melt was 0.17 sq. cm. per g. During the experiments at 1450°C, visible quantities of magnesium were distilled out of the charge by reaction with plutonium and some of the fission products, viz.,

$$2\ \mathrm{La}\ (l)\ +\ 3\ \mathrm{MgO}\ (s)\ =\ \mathrm{La_2O_3}\ (s)\ +\ \mathrm{Mg}\ (g).$$

After the melt had been held at temperature for the desired time, it was cooled to room temperature and the resulting ingot was recovered by breaking the crucible. The ingot was then sampled by machining in an inert atmosphere.

Results of one typical experiment are shown in Table 4. As anticipated, rare earth elements did concentrate in the oxide phase while molybdenum and ruthenium did not. Zirconium also appears to have concentrated slightly in the oxide phase. The increases in zirconium, lanthanum and cerium concentrations at the ingot top apparently are due to both drossing and partial liquation.

Table 4

Effect of Oxide Drossing on the Composition of Fissium Alloy

Ingot Region Sampled	W/W_o (a)					
	Zr	Mo	Ru	La	Ce	Fe
Metal Top	2.80	0.77	0.81	2.03	2.83	0.79
Metal Core	0.74	1.00	1.07	<0.05	0.05	0.95
Oxide Skin	1.73	0.41	0.11	28.	9.3	0.26

(a) W = weight percent of element in sample after processing
W_o = weight percent of element in fissium before processing

The effect of varying the temperature is shown in Table 5. A negligible amount of oxide was formed in 4 hr. at 1000°C and there was no change in the cerium concentration. Although some reaction occurred at 1200°C (above the normal boiling point of magnesium), significantly faster removal was obtained at 1450°C.

Table 5

Effect of Drossing Temperature on the Composition of Fissium Alloy

Temperature, °C	Time, hr.	W/W_o						
		Zr	Mo	Ru	La	Ce	Fe	Co
1000	4	0.88	0.70	0.91	..	1.00	..	0.92
1200	2	0.79	0.67	0.99	0.44	0.89	1.10	..
1450	2	0.79	0.73	0.90	0.26	0.62	1.06	..

As shown in Table 6, a reaction time of 5 hr. was required at 1450°C to reduce the total rare earth concentrations by a factor of at least 20.

Table 6

Effect of Drossing Time at 1450°C on the Composition of Fissium Alloy

Time, hr.	W/W_o						
	Zr	Mo	Ru	La	Ce	T.R.E.(a)	Fe
2	0.79	0.73	0.90	0.26	0.62	0.39	1.06
3	1.14	1.37	1.04	<0.07	0.27	<0.15	1.14
5(b)	0.74	1.00	1.07	<0.05	0.05	<0.05	..

(a) Total rare earths

(b) Concentration of magnesium in processed ingot was <0.1 weight percent.

Several 1-kg. scale drossing experiments were conducted in the apparatus shown in Fig. 1. The experimental procedure was similar to that used in the small-scale experiments, except that the melt was bottom-poured out of the crucible by withdrawing a magnesia stopper rod at the completion of each run. Dry-pressed magnesia crucibles and stopper rods having a wetted area of 0.10 sq. cm. per g. of melt were used in all experiments. These experiments have led to the following findings which are offered without detail.

(1) Drossing times in excess of 5 hr. are required at 1400°C for good rare earth removal on the 1-kg. scale.

(2) Long drossing times result in high plutonium losses, i.e., yields of only 72 to 90 percent are attainable after drossing for 5 to 8 hr. at 1400°C. Increasing the pouring temperature from 1000°C to 1400°C is beneficial.

(3) The poured product contains less than 200 p.p.m. of magnesium, less than 100 p.p.m. of carbon and less than 20 p.p.m. of oxygen.

(4) The use of zirconia in place of magnesia crucibles may improve the yield of poured product.

3. Carbide Slagging

One carbide slagging experiment was conducted to evaluate the effectiveness for removing zirconium. Approximately 80 g. of cobalt fissium was held at 1000°C for 2 hr. with a graphite rod immersed 1/4 in. into the melt. Analysis of the ingot core indicated that the zirconium had been reduced to 60 percent of the initial concentration, while analysis of the carbide slag indicated that zirconium had concentrated by a factor of approximately 2.3 in this phase. Molybdenum, ruthenium and cerium concentrations were not affected significantly.

Although carbide slagging did improve zirconium removal somewhat over that obtained by oxidative drossing, the improvement was insufficient. Moreover, the large amount of plutonium carbide formed in this experiment indicated that this method would result in poor plutonium yields.

4. Halide Slagging

In theory, halide slagging should be a better method for separating fission products from plutonium fuels than oxide drossing. The alkaline earth and rare earth elements form oxides that are more stable than plutonium oxide. On the other hand, the alkalies, the alkaline earths and the rare earths form more stable fluorides and chlorides. Thus, halide slagging potentially can remove a greater number of impurities. Of greater importance is the fact that halides, and particularly chlorides, have much lower melting points than the oxides. Therefore, it should be possible to employ a liquid slag, thereby eliminating the problem of the slow reactions experienced with solid oxide phases.

Two preliminary fluoride slagging experiments were conducted with cobalt fissium. In both experiments 40 g. of melt was held in a cobalt difluoride crucible in a helium atmosphere. Only the ingot was sampled after processing. Although the results shown in Table 7 indicated that good cerium removal is possible by this method, no additional fluoride slagging experiments were performed.

Table 7

Effect of Fluoride Slagging on the Composition of Fissium Alloy

Experimental Conditions	W/W_o				
	Zr	Mo	Ru	Ce	Co
30 min. at 550°C	0.81	0.85	0.91	1.03	0.93
60 min. at 900°C	0.71	0.88	1.13	<0.08	1.65

The removal of fission product elements by chloride slagging was tested in the following experiment. Approximately equal volumes of fissium (100.44 g.) and 22 weight percent sodium chloride - plutonium trichloride eutectic (32.46 g.) were contacted at 600°C in a tantalum crucible under an argon atmosphere in a resistance furnace. In order to avoid the presence of solid phases, fissium that had been liquated at

600°C and filtered was used. The liquid metal and liquid salt phases were periodically stirred, then allowed to separate. After the phases had separated, each phase was sampled at temperature and the distribution of fission product elements was determined by chemical analysis of these samples. Samples taken after 3, 6 and 30 hr. indicated that each phase had attained a constant composition within 3 hr. The results shown in Table 8 are in agreement with thermodynamic arguments, i.e., the rare earth elements are nearly completely transferred to the salt phase, while ruthenium and molybdenum are not. The behavior of zirconium and niobium could not be determined because of the limits of analytical methods employed.

Table 8

The Distribution of Fission Product Elements Between Molten Fissium and $PuCl_3$-NaCl Eutectic at 600°C

Element	Concentration in Initial Alloy, w/o	Percent of Original Amount in	
		Metal Phase After 3 hr.	Salt Phase After 3 hr.
Zr	<0.003	...	...
Nb	<0.006	...	...
Mo	0.036	97	<3
Ru	1.22	97	<3
La	0.24	...	100
Ce	0.75	<0.9	100
Fe	2.54	100	<0.1

5. Halide Conversion Cycles

In contrast to halide slagging, it may be feasible to convert all of the plutonium in a fuel to a halide compound which may then be selectively reduced back to the metal. Purification could be affected by (1) using an appropriate oxidizing agent so that the more noble elements, such as ruthenium, are not oxidized, (2) physical separation of volatile or insoluble fission product halides and finally, (3) selective reduction of the plutonium halide.

Several fluoride conversion experiments were conducted in which an 85-g. ingot of fissium was directly converted to anhydrous plutonium tetrafluoride by reaction at 600°C with hydrogen fluoride in the presence of a small amount of oxygen to prevent the formation of plutonium trifluoride. The entire fluoride powder was then reduced with calcium by the bomb method that is used routinely to prepare plutonium metal. Moderate purification from molybdenum, lanthanum and cerium was obtained, while zirconium and ruthenium concentrations were not affected. This fluoride conversion method does not appear to have the advantages of either good purification or engineering simplicity. Apparently a physical separation is required to remove the "noble" elements.

The first portion of a chloride conversion cycle also has been studied. In this method, sodium chloride was added to lower the liquidus temperature. The stoichiometric quantity of zinc chloride in a 30 weight percent sodium chloride solution was added to 21 g. of fissium alloy and slowly heated to 850°C in an argon atmosphere. Both a liquid metal and a blue liquid salt phase were visible at this temperature. The system was then cooled to 550°C and a portion of the salt phase was filtered through a medium grade sintered Pyrex disc. In addition to halides of the fission product elements, this phase was predominantly the 22 weight percent sodium chloride-plutonium trichloride eutectic. After cooling to room temperature, the unfiltered salt and metal phases were recovered and also analyzed chemically. The distribution of each element after reaction is shown in Table 9. Because the composition of the unfiltered salt was essentially identical to that of the filtered sample, they are reported as a single phase in Table 9.

Table 9

Distribution of Elements After Reaction of Fissium with Zinc Chloride

Element	Percent of Original Amount	
	Salt Phase	Zinc Button
Zr	< 0.5	1.4
Mo	< 0.4	98
Ru	< 1	99
La	89	11
Ce	101	< 0.7
Fe	< 0.2	...
Zn	< 0.2	97
Pu	98	1.5

The following conclusions may be drawn from these results:

(1) The conversion of plutonium to the trichloride proceeded smoothly and in good yield.

(2) Zirconium, molybdenum, ruthenium, iron and zinc concentrations in the salt phase are negligible.

(3) The rare earth elements are oxidized by zinc chloride and concentrate with the plutonium in the salt phase. (Removal of these impurities in a subsequent reduction step may be difficult.)

(4) As anticipated from vapor pressure data, zirconium chloride volatilizes from the melt.

6. Electrorefining

Good purification has been obtained by transferring uranium from an impure uranium anode to a cathode, using a molten salt electrolyte.(5) Theoretically this process is well adapted to give good purification by controlling the deposition potential. Fission products having chlorides that are less stable than plutonium chloride should concentrate as an anode sludge, while fission products having chlorides that are more stable should concentrate in the molten salt electrolyte.

An electrorefining experiment was conducted with iron-fissium using the cell shown in Fig. 2. Approximately 20 g. of fissium melt was contained in the anode compartment at the start of the electrorefining. A Pyrex-sheathed thermocouple (not shown in Fig. 2) was immersed in the molten anode. In order to eliminate the problems associated with dendritic cathode deposits, an iron cathode was used to permit the collection of the product in a tantalum reservoir as a liquid plutonium-iron alloy. The molten salt composition was 41 - 50 - 9 weight percent lithium chloride-potassium chloride-plutonium chloride. The experiment was conducted at 550°C in an argon atmosphere. After passing a current of 0.25 amp. for 17 hr. at a potential of one volt, the cell was disassembled and a shiny button of refined plutonium weighing 6 g. was recovered in the tantalum reservoir. This button was dissolved completely and analyzed chemically, as was the anode residue.

The results shown in Table 10 indicate that this method has excellent purification potentialities.

Table 10

Purification of Fissium Alloy by Electrorefining

Element	Concentration of Element, weight percent		
	Feed Material	Product	Anode Residue
Zr	0.672	< 0.01	2.08
Mo	0.751	0.009	2.20
Ru	0.968	< 0.05	3.19
La	1.59	< 0.097	0.35
Ce	0.735	< 0.01	< 0.10
Fe	2.38	4.56	7.3
Pu	92.3	96.5	81.0
Li	0	< 0.06	..

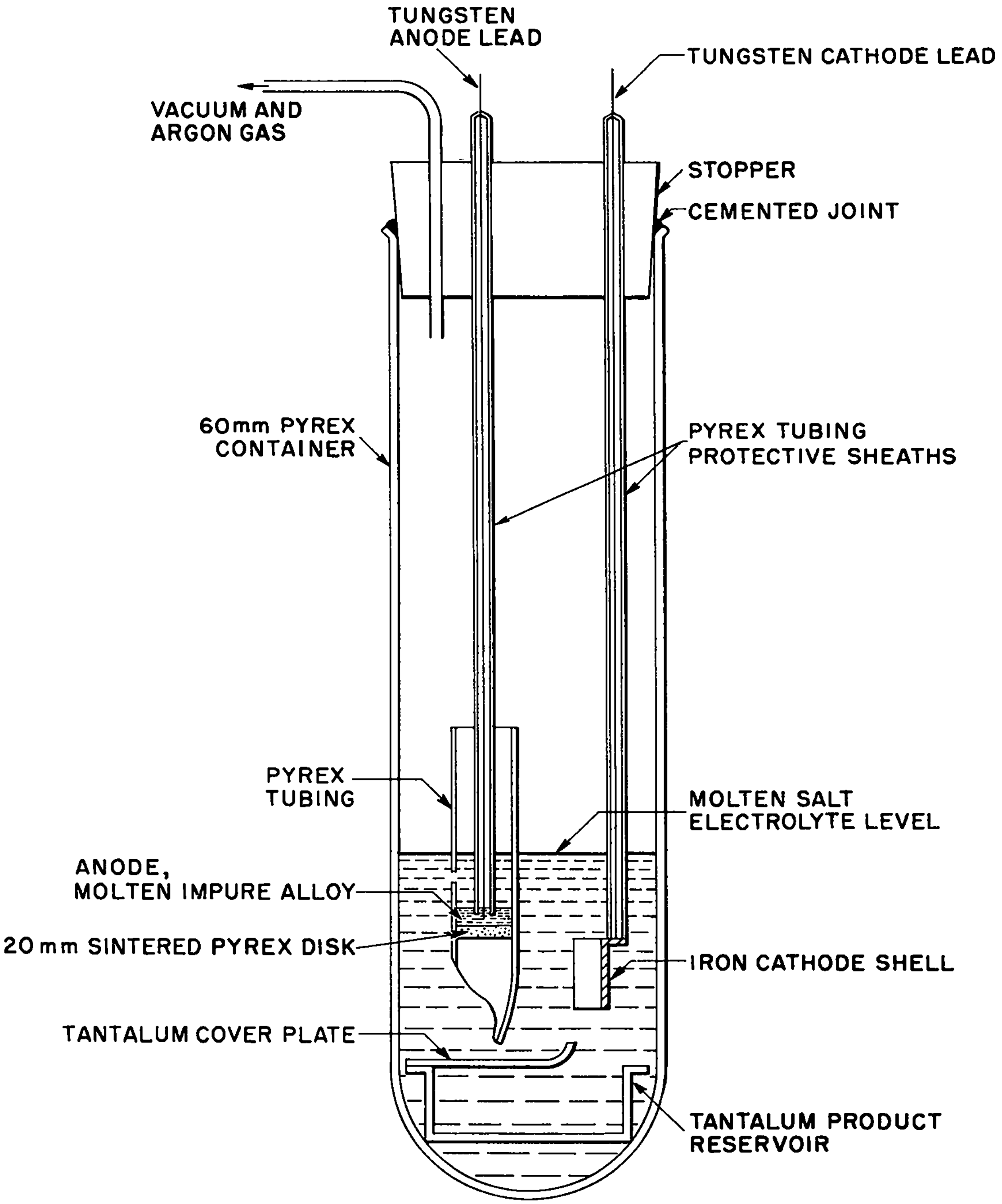

Figure 2. Plutonium Electrorefining Cell

The elements zirconium, molybdenum and ruthenium concentrated at the anode, while the rare earths apparently concentrated in the salt phase (which was not analyzed). The iron-plutonium ratio in the product material corresponds to 17 atomic percent iron. This is identical to the liquidus composition at 550°C on the iron-rich side of the 9.5 atomic percent iron eutectic.

7. Recrystallization from Mercury

Purification by a mercury extraction-recrystallization cycle has been proposed for uranium fuels.(6) In such a process, uranium is dissolved in mercury at an elevated temperature, then filtered. Insoluble fission products presumably remain behind in the dissolver. The amalgam is then cooled to precipitate uranium tetramercuride and filtered at the lower temperature. Soluble fission products should remain in the filtrate. Uranium tetramercuride may then be decomposed to recover the uranium.

The feasibility of using this method for plutonium pyroprocessing has been studied by measuring the solubility of individual elements in mercury at various temperatures. The solubility of "soluble" elements was determined by equilibrating with mercury and filtering at temperature. The filtrate sample was then cooled, weighed and analyzed for the appropriate element. Except for ruthenium, the solubility of "insoluble" elements was determined by immersing weighed coupons in a known amount of boiling mercury and periodically measuring the coupon weight. Thus, the weight loss corresponded to the amount of element dissolved. Because ruthenium formed a quasi-amalgam that did not pass through the filter, the solubility of this element was determined by the filtration method used for the "soluble" elements. Results obtained in these experiments are summarized in Table 11.

Table 11

The Solubilities of Selected Elements in Mercury

Element	Temperature, °C	Solubility, g. element per l. mercury
Zr	350	0.093
Nb	350	< 0.001
Mo	350	< 0.001
Fe	350	0.002
Ta	350	< 0.001
Pu	20	2.65
	325	85.7
Ru	20	< 0.002
	250	< 0.002
La	20	2.87
	250	36.8
Ce	20	1.31
	250	74.5

These results indicate that good purification may be attainable by this method, provided that no insurmountable difficulties are encountered in dissolution or co-precipitation. Apparently the rare earths are sufficiently soluble at 20°C, while niobium, molybdenum and ruthenium are relatively insoluble at 250°C. On the other hand, the solubility of zirconium at 350°C is high in comparison to the fission yield, so that the behavior of this impurity is more ambiguous.

8. Liquid-Liquid Metal Extraction

The extraction of fission products and plutonium from uranium by liquid rare earth elements and silver has been reported.(3c) The choice of a suitable extractant metal for plutonium fuels is complicated by the fact that most low melting elements and alloys are miscible with plutonium to an appreciable extent. However, the 68 atomic percent calcium-32 atomic percent magnesium eutectic has a very limited mutual solubility with liquid plutonium-iron alloy. Consequently, the use of liquid calcium-magnesium was evaluated in a series of experiments in which equal volumes of the two liquid phases were equilibrated for 24 hr. by stirring in a helium atmosphere at 600°C. After equilibration, each phase was sampled at 600°C and analyzed chemically. Iron-fissium alloy that had been liquated and filtered was used in these experiments to avoid the presence of solid phases. The results are shown in Table 12.

Table 12

Distribution of Elements Between Liquid Calcium-Magnesium and Molten Fissium at 600°C

Distribution Ratio(a)							
Zr	Nb	Mo	Ru	La	Ce	Fe	Pu
0.35	<0.7	0.16	<0.06	61.	1.02	0.011	0.015

(a)Distribution ratio defined as weight percent of element in calcium-magnesium divided by weight percent of element in iron-plutonium phase.

Apparently lanthanum is extracted very favorably, cerium is extracted partially and extraction of the other elements is unfavorable.

SUMMARY

Methods for purifying spent plutonium reactor fuel have been surveyed experimentally. From these results it appears that most of the procedures studied are worthy of further experimentation.

In general, the results have been in accordance qualitatively with the trends that may be predicted from thermodynamic considerations.

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