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### ABSTRACT

Scale-up work on high temperature fuel recovery processes has progressed to the point where the (high temperature) vacuum furnace for several operations in the hot cells has been completed and tested under operating conditions.

Small scale experiments on high temperature methods for processing molten irradiated uranium fuel have been made with spent X-10 fuel slug pieces. The results of direct Pu evaporation, treatment with fused fluorides and oxide scavenging were very similar to those found with tracer experiments.

Small scale tracer experiments on the extraction of Pu from U with both Ca and Mg show that up to 80 per cent of the Pu was extracted with Ca and 20 per cent with Mg. Treatment of uranium amalgams with KCl-LiCl at 350° C removed rare earths to some extent.

This report is based upon studies conducted for the Atomic Energy Commission under Contract AT-11-1-GEN-8.

#### Previous Quarterly Reports in This Series

- NAA-SR-220 Separations Chemistry Progress Report for the Period October, November, December, 1952, March 27, 1953.
- NAA-SR-245 Separations Chemistry Quarterly Progress Report, January-March, 1953, July 10, 1953.
- NAA-SR-273 Separations Chemistry Progress Report for Period April, May, June, 1953, December 1, 1953.
- NAA-SR-844 Separations Chemistry Quarterly Progress Report, July-September, 1953, April 1, 1954
- NAA-SR-943 Separations Chemistry Quarterly Progress Report, October-December, 1953, June 1, 1954.

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## I. INTRODUCTION

The research program under study is chiefly concerned with the development of high temperature processes for fuel recovery and for extracting fissionable elements and fission products from reactor fuels.

## II. HIGH TEMPERATURE COMPACT SEPARATIONS METHODS FOR NUCLEAR FUELS

### A. SCALE-UP WORK (J. R. Foltz, W. J. Gardner, and F. D. Rosen)

The vacuum furnace for the high temperature process experiments to be done in the hot cell was completed and tested. Figure 1 shows the vacuum furnace assembly on the transfer dolly which is used to assemble the experimental equipment and transfer the equipment into the hot cell. In Fig. 2 the experimental assembly is being transferred between the dolly and hot cell. The next figure (Fig. 3) shows the crucible components being lowered into the vacuum furnace.

At present the various associated components for the hot cell experiments are being fabricated and tested. Cold runs with natural uranium have been started.

### B. REMOVAL OF PLUTONIUM FROM URANIUM WITH FUSED HALIDES

1. Fluorides (A. G. Buyers) - Molten uranium-fused salt equilibration at  $1300^{\circ}\text{C}$  have been carried out using ORNL X-10 slug fragments with uranium tetrafluoride and with calcium-lithium fluoride mixtures.

Experiments were performed under  $5/6$  of an atmosphere of purified helium using a graphite crucible to contain the melt. Crucibles and their contents were maintained at  $1300 \pm 100^{\circ}\text{C}$  for a period of  $1/2$  hour. The crucible and other parts of the fusion chamber are shown in Fig. 4. Crucibles were degassed, and salts and salt mixtures were prefused prior to being melted with uranium metal.

The melt was mechanically separated into its different phases by means of the steel mortar and pestle shown in Fig. 5. Figure 5 also shows external

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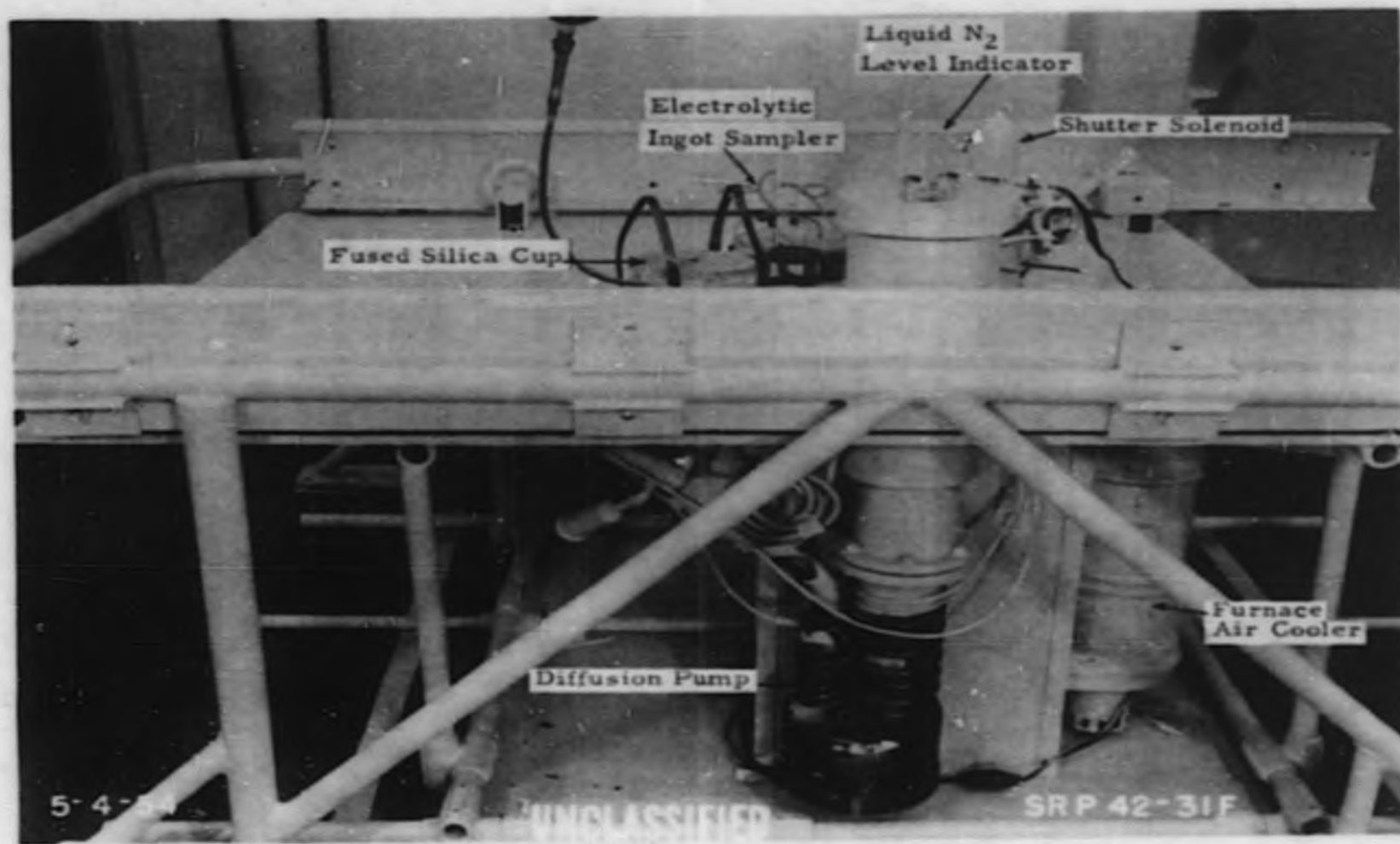


FIG. 1. SCALE-UP VACUUM FURNACE ASSEMBLY ON TRANSFER DOLLY

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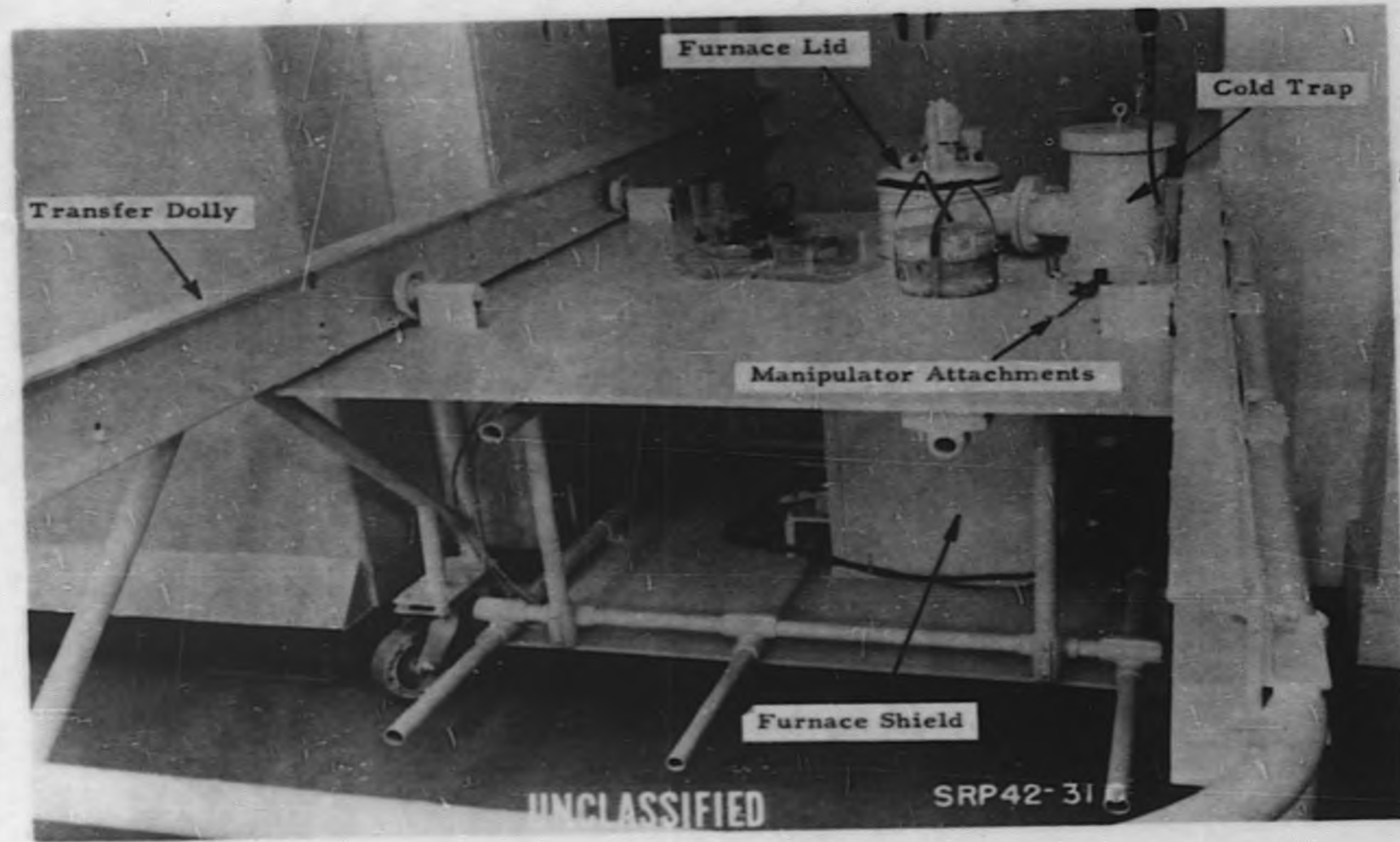
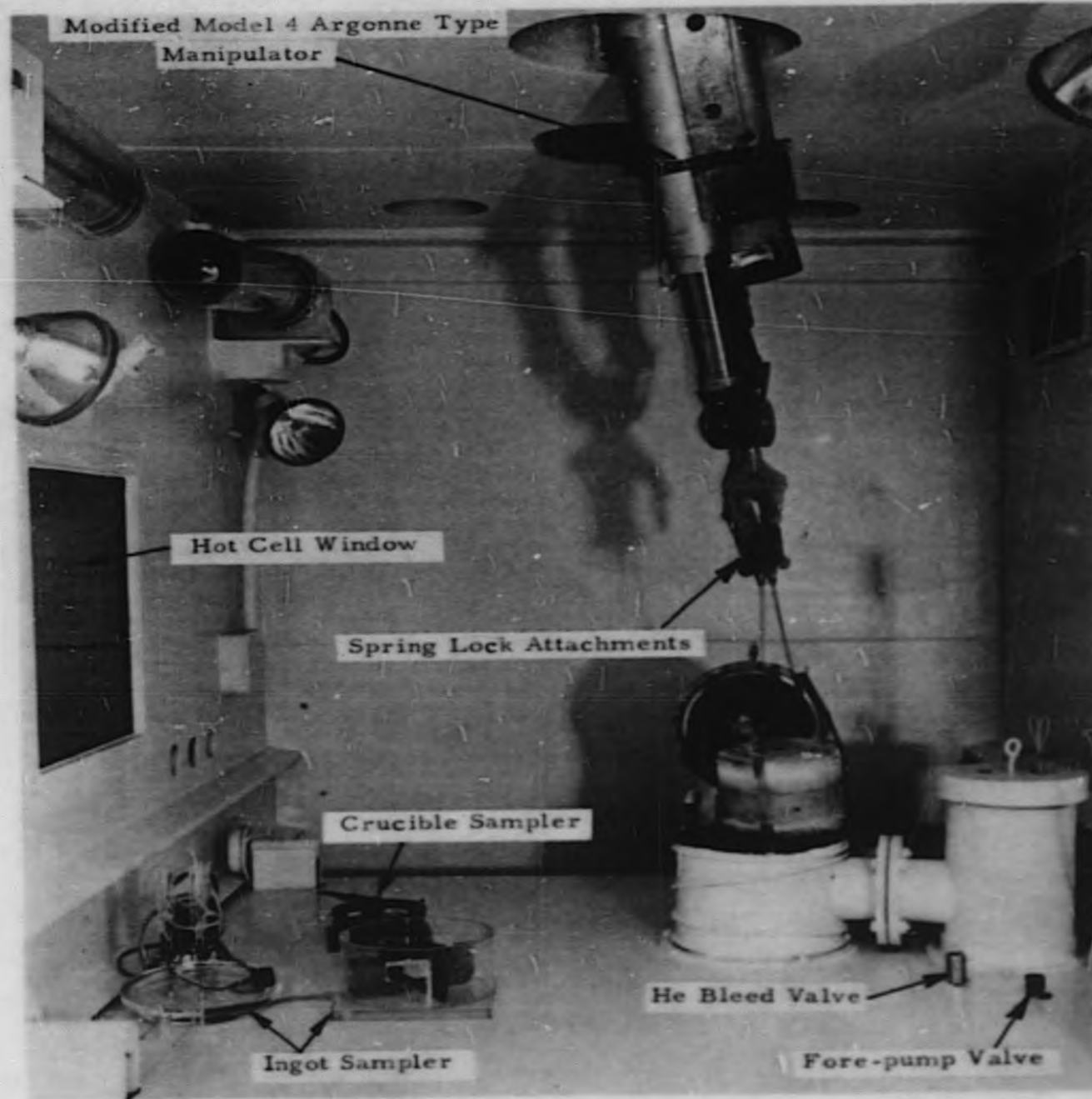


FIG. 2. SCALE-UP UNIT TRANSFER BETWEEN HOT CELL AND DOLLY



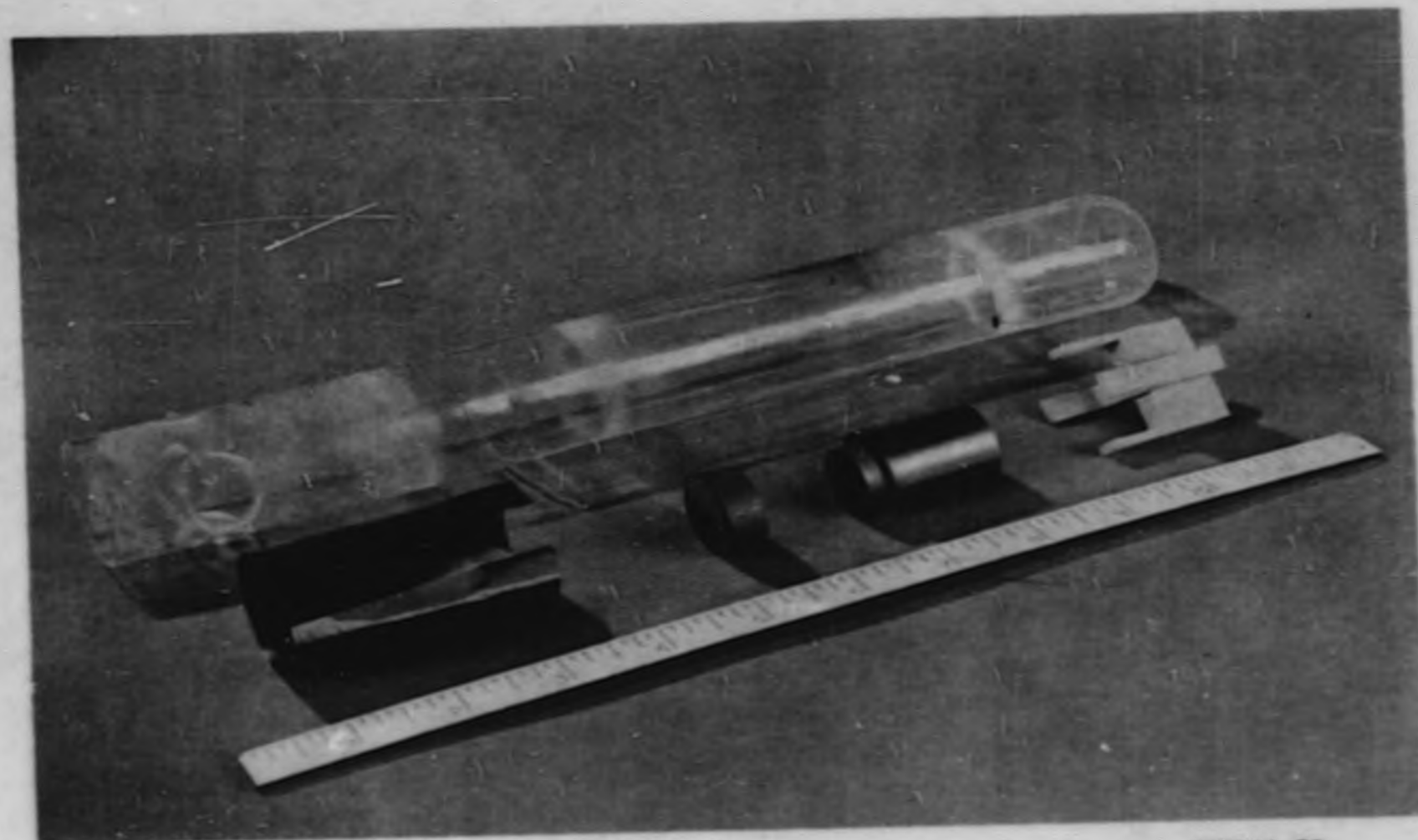


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FIG. 3. LOADING OF VACUUM FURNACE WITH MANIPULATOR ATTACHMENTS



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FIG. 4. FUSION CHAMBER COMPONENTS FOR FLUORIDE FUSIONS

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FIG. 5. EXTERIOR OF FUSION CHAMBER AND GLOVE BOX FITTINGS FOR X-10 FUEL FRAGMENT EXPERIMENTS





arrangement of the fusion chamber and the most important interior fittings of the glove box used to contain these experiments. Cold surface washings and crucible leachings were added to the salt phase solution.

Plutonium distribution values resulting from equilibration of X-10 fragments with  $UF_4$ , and X-10 fragments with 1:1 mixture of  $CaF_2$  and  $LiF$ , were summarized in Table I. Examination of these data has disclosed that both salts were effective in extracting plutonium, 84-91 per cent appearing in the recovered salt. The greater stability of calcium and lithium fluoride over that for plutonium fluorides has suggested that traces of less stable uranium fluorides were formed during the salt-metal fusion and that their presence caused the plutonium to move into this salt mixture.

TABLE I  
PLUTONIUM DISTRIBUTION VALUES OBTAINED BY EQUILIBRATING MOLTEN URANIUM FUEL FRAGMENTS (X-10) WITH FUSED SALTS AT 1300° C

Expt. No.	Salt Used	% Plutonium Activity Measured		
		In Salt	In U Metal Residue	Total
1	$UF_4$	86.7	7.5	94.2
2	$UF_4$	87.2	5.3	92.5
3	$UF_4$	91.3	4.4	95.7
4	$\frac{CaF_2}{LiF} = \frac{1}{1}$ by wt	90.7	10.7	101.4
5	$CaF_2 = \frac{1}{1}$ by wt	84.1	21.0	105.1

Fission product distribution in the foregoing experiments will be determined.

2. Chlorides (W. A. Rosser, Jr.) - The removal of Pu from uranium by chloride salts has been investigated in two ways. In one set of experiments molten uranium containing tracer Pu was equilibrated with molten  $CaCl_2$  containing some  $UCl_3$ . In another set molten uranium was exposed to HCl gas forming  $UCl_3$  which rapidly evaporated from the uranium. Both types of experiments were successful in removing some Pu; however, the degree of removal

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was not as large as that found with comparable fluoride experiments (see NAA-SR-926).

The experiments with  $\text{CaCl}_2$  were conducted by equilibrating tracer irradiated uranium with the salt phase at  $1150^\circ - 1200^\circ \text{C}$  in covered graphite crucibles for 15 to 20 minutes. The rate of evaporation of the salt under about 0.1 atmospheres of helium was not great enough to be troublesome. Uranium trichloride was introduced into the salt phase by mixing small amounts of  $\text{AgCl}$  with the initial salt. The  $\text{UCl}_3$  was produced by the reaction:



The results obtained are shown in Table II. The best Pu removal attained was about 80 per cent. The data shows that the per cent removal did increase with increasing  $\text{UCl}_3$  concentration in the salt phase, so that with pure  $\text{UCl}_3$  the removal of Pu may be essentially complete.

The treatment of molten uranium with  $\text{HCl}$  gas was accomplished by melting the uranium in a graphite crucible in vacuo and then admitting a mixture of a small amount of  $\text{HCl}$  in He gas. The  $\text{UCl}_3$  was observed almost immediately on the cold surfaces of the apparatus, and in a few minutes the reaction was complete.

TABLE II  
TRACER Pu REMOVAL FROM MOLTEN U BY FUSED CHLORIDES

Salt Phase	Wt U, gm	Per Cent Pu Found in Metal	Per Cent Pu Removed
3 gm $\text{CaCl}_2$	6	46	54
1.5 gm $\text{CaCl}_2$	7.7	33	67
0.1 gm $\text{UCl}_3$			
3 gm $\text{CaCl}_2$	6	27	73
0.3 gm $\text{UCl}_3$			
3 gm $\text{CaCl}_2$	6	20	80
1.5 gm $\text{UCl}_3$			





The results obtained by this method are shown in Table III. Under the best conditions the Pu was removed almost quantitatively. The results show that with increasing amount of uranium the per cent Pu removal decreased. Since the same bore crucible was used in all these experiments, this last statement may be rephrased to say: The greater the thickness of the uranium layer, the smaller the per cent Pu removal. Such an effect was not unexpected in these experiments. Since the  $UCl_3$  evaporated so rapidly, it did not have time to equilibrate with the uranium at the bottom of a deep layer. The degree of Pu removal could be improved with the deep uranium layers by reducing the rate of evaporation of the  $UCl_3$ .

TABLE III  
TRACER Pu REMOVAL FROM MOLTEN U BY TREATMENT WITH HCl GAS

Wt U Used gm	Wt U Convected to $UCl_3$ gm	Rare Earths		Pu	
		% Found in Metal	% Found in Salt	% Found in Metal	% Found in Salt
8.1	0.04	-	-	2	-
67	1.7	73	17	40	47
83	2	85	17	38	56
116	1	-	-	73	25

#### C. DISTILLATION OF Pu FROM MOLTEN URANIUM D. Cubicciotti

To date two successful experiments have been completed on the small scale distillation of Pu from pieces of X-10 fuel material. The details of the experiment are essentially the same as those used in the tracer work (NAA-SR-242). The results are shown in Table IV.

The agreement between the observed per cent evaporated and that calculated from Raoult's Law is excellent. The results show that Pu can be evaporated from U to 99 per cent or better in the time expected from theoretical considerations.

#### D. DECONTAMINATION OF URANIUM WITH SOLID SCAVENGERS (A. M. Saul)

The uranium oxide scavenging experiments have been extended to long-cooled ORNL (X-10) fuel.

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TABLE IV  
RESULTS OF DISTILLATION OF Pu FROM X-10 FUEL

Wt, gm	100 gms Pu/gm U 1680° C, CeS Crucible, 0.1 Micron			
	Area, cm <sup>2</sup>	Heating Time, min	% Pu Evaporated	% Pu by Raoult's Law
3.3	2.3	40	90.9	91.5
3.6	1.4	140	98.6	99.5

A series of 4 experiments has been run in which small fragments of this fuel were oxidized and subsequently melted in a graphite crucible. The plutonium and fission product distribution found are listed in Table V. The general experimental procedure employed was as follows:

An approximate 4-gram sample of X-10 fuel was cleaned by successive washes of dilute nitric acid, distilled water, and acetone. The metal was dried, weighed, and placed in a graphite crucible. The crucible was then inserted into a Vycor tube similar to that shown in Fig. 5, and the system was evacuated. When a vacuum of  $10^{-5}$  mm of Hg was obtained, one-half of an atmosphere of oxygen was introduced and allowed to contact the uranium metal. The reaction tube was then heated by means of a resistance furnace to  $250^{\circ} \text{C} \pm 25^{\circ} \text{C}$  and kept at this temperature for 1 hour. This reversal of procedure, i. e., introducing the oxygen before heating the uranium, has been found necessary in order to slow the oxidation rate of the uranium to within controllable limits. By employing such a procedure a surface coating of oxide (representing only a few milligrams of uranium oxide) was formed on the metal. Following this oxidation step, the resistance furnace was removed and the system re-evacuated to a vacuum of  $10^{-5}$  mm of Hg. By means of an induction furnace the uranium metal was rapidly heated to the minimum temperature at which melting of the metal could be observed. In all four experiments this was around  $1175 \pm 50^{\circ} \text{C}$ . In experiments I and II, the uranium was kept at  $1175^{\circ} \text{C} \pm 50^{\circ} \text{C}$  for 1 hour. In experiments III and IV, after the metal melted, it was heated to  $1300^{\circ} \text{C} \pm 50^{\circ} \text{C}$  and kept at this temperature for 1 hour.

After this heating, the system was cooled and opened. The uranium sample was removed from the graphite crucible and the oxide film dissolved from the





metal with hot 16 M  $\text{HNO}_3$ . The cleaned metal was weighed and dissolved in hot 6 M  $\text{HNO}_3$  containing a few drops of 12 M  $\text{HCl}$ . Both the oxide solution and the treated metal solution were then analyzed by standard methods for plutonium and fission products. A correction was made for the amount of Pu and fission product activity carried over with any uranium metal dissolved with the oxide phase.

Unlike the tracer experiments, it has been found impossible to prevent some reaction between the uranium metal and the graphite crucible. It is felt that this is due to bubbling of the metal which occurs when the X-10 fuel is melted. This bubbling breaks the protective oxide film, thus allowing the uranium metal to contact the crucible. This bubbling was not observed in the tracer experiments and may be due to volatilization of impurities such as magnesium present in the X-10 uranium metal.

Carbide interference, then, could well be an explanation for the poor balance indicated in Table V. To avoid this interference, a series of experiments has been initiated in which samples of X-10 fuel are heated in zirconia and calcia crucibles, and the resulting fission product and plutonium removal studied.

#### **E. TREATMENT OF URANIUM WITH OTHER METALS**

1. Mercury (S. J. Yosim, F. J. Keneshea, Jr.) - The use of mercury as an alloying metal for lowering processing temperatures of uranium was suggested last quarter. A liquid uranium amalgam containing 10 atomic per cent uranium can be obtained at a temperature of  $340^\circ\text{C}$ . At this temperature, corrosion of container materials such as glass, graphite, and ceramics by the liquid uranium system should be very slight, if not negligible. In principle it should be possible to carry out all the processes that are carried out with molten uranium at  $1200^\circ\text{C}$  - with the exception, of course, of volatilization. Thus, salt scrubbing and solid scavenging can be carried out on a liquid uranium system at a temperature of only about  $360^\circ\text{C}$ . Therefore, it was decided to conduct some exploratory decontamination experiments on the uranium amalgam.

The amalgam for experiments described here was prepared in a "Soxhlet" type apparatus shown in Fig. 6. By this method fresh mercury continually

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TABLE V

## URANIUM OXIDE SCAVENGING EXPERIMENTS—X-10 FUEL

Wt of U Metal Used (gm)	Temp, °C	Time of Heating	% of Initial Activity	
			Found in Treated Metal	Found in Oxide
2.6	1175 ± 50	1	Pu: 98	2
			Sr: 40	31
			Ce: 56	12
			R. E.: 37	32
			Te: 34	0
			Cs: 10	12
			Ru: 100	0
6.1	1175 ± 50	1	Pu: 96	1
			Sr: 53	16
			Ce: 29	22
			R. E.: 58	15
			Te: 29	9
			Cs: 13	5
			Ru: 100	0
5.0	1300 ± 50	1	Pu: 96	5
			Sr: 23	10
			Ce: 22	14
			R. E.: 20	40
			Te: 17	0
			Cs: 5	3
			Ru: 100	0
4.1	1300 ± 50	1	Pu: 96	5
			Sr: 31	21
			Ce: 21	14
			R. E.: 22	31
			Te: 18	0
			Cs: 6	6
			Ru: 100	0



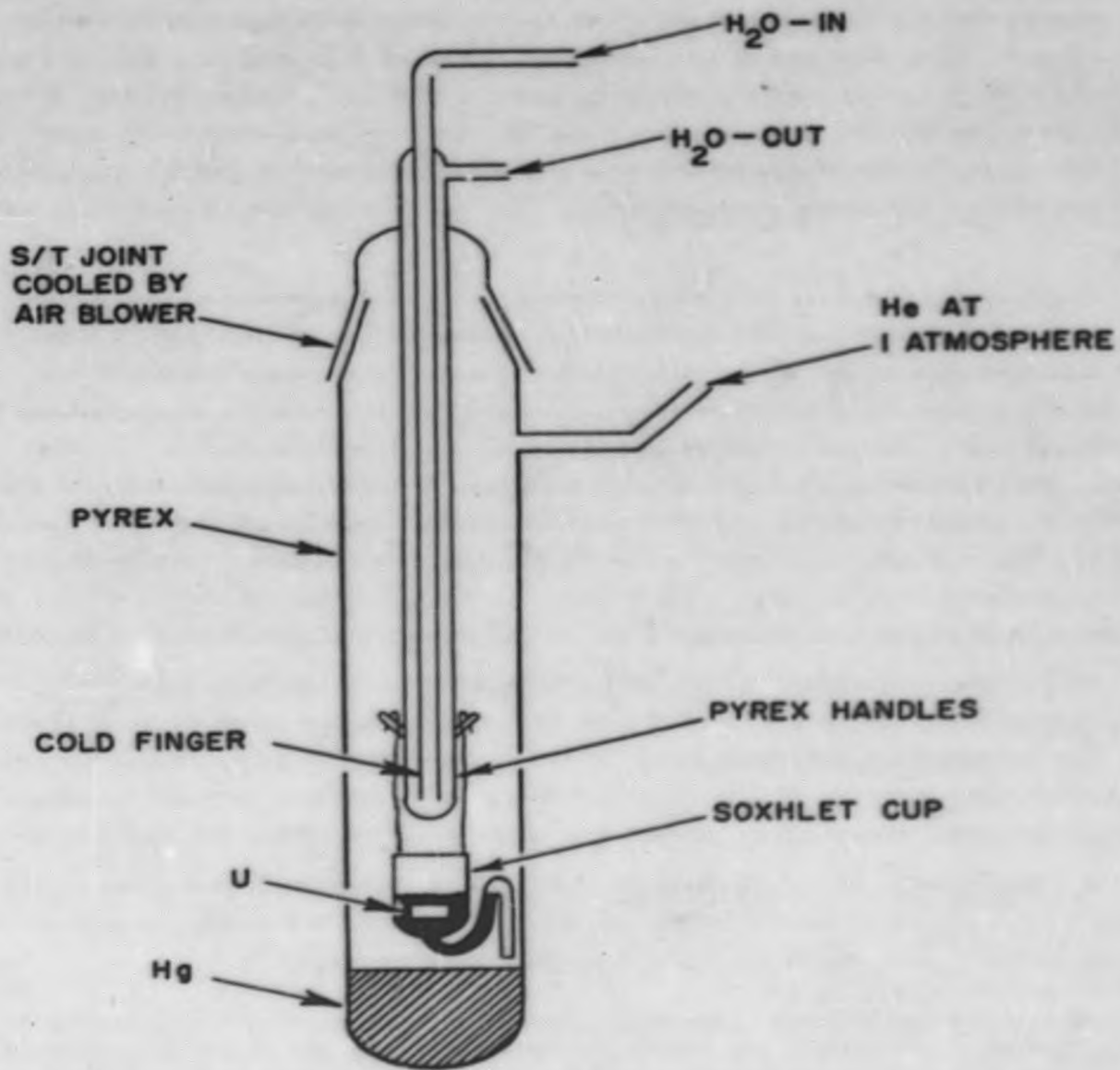


FIG. 6. DIAGRAM OF SOXHLET TYPE APPARATUS USED IN U-H<sub>2</sub> EXPERIMENTS

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introduced to the uranium under a helium atmosphere resulted in a considerably greater amalgam yield than merely boiling the mercury containing the uranium.\*

Salt scrubbing constituted the first decontamination experiments on the U-Hg system. The salt phase was composed of about 9 grams of a KCl (41 mole per cent) LiCl eutectic mixture (melting point = 352° C). When desired,  $UCl_3$  was prepared in situ by adding to the eutectic,  $Hg_2Cl_2$  plus a strip of "cold" uranium. Upon fusion of the mixture, a purple salt appeared which, when dissolved in water, formed a green solution. The purple salt was assumed to be  $UCl_3$ .

The experiment was carried out by placing about 3 grams of irradiated strips in the "Soxhlet" cup and about 300 grams mercury to the "pot." The fused salt was then added to the pot and the entire system evacuated. After heating under vacuum to a temperature of about 100° C, helium was admitted to the system and mercury refluxed until it appeared that the uranium was dissolved. It is interesting to note that as soon as the first amalgam dropped into the pot, the color of salt turned to a deep burgandy, suggesting that  $UCl_3$  was formed. The system was then disassembled, the salt separated mechanically, and the amalgam separated by a filtration in a coarse Pyrex sintered filter. A solution of any residue remaining in the Soxhlet cup also constituted a fraction.

The tentative results of two such salt scrubs performed on irradiated uranium (nvt =  $10^{17}$ ) are shown in Table VI. In both cases most of the plutonium remained in the amalgam while much of the rare earths were extracted into the salt phase. The rare earth activity remaining in the Soxhlet cup was perhaps a result of an oxide scavenging. Oxide scavenging experiments are being planned.

2. Magnesium (F. J. Keneshea, Jr.) - The solubility of uranium in Mg is quite low.† Experiments to study the distribution of Pu and fission products between liquid U and liquid Mg have therefore been initiated.

\*Actually amalgamating the decomposed uranium hydride would probably prove more feasible. However, the "Soxhlet" apparatus was developed for experiments in which it was desirable that no uranium in the form of a suspension be present.

†G. A. Tracy, P. Chiotti, and H. A. Wilhelm, "Magnesium-Uranium Alloy System," ISC-377 (1953).





TABLE VI

## DISTRIBUTION OF TRACER ACTIVITY RESULTING FROM KCl-LiCl SALT SCRUB OF URANIUM AMALGAM

	KCl - LiCl			KCl - LiCl 5w/o $UCl_3$		
	% Activity Found			% Activity Found		
	Gross Beta, Gamma	Pu	Total Rare Earths	Gross Beta, Gamma	Pu	Total Rare Earths
Amalgam	42.5	92.0	35.3	26.3	76.5	19.7
Salt	35.0	0.9	43.0	53.5	14.5	57.6
Soshlet Cup	18.2	5.5	13.8	22.9	-	21.8
Filtrate	0.9	-	0.7	0.7	-	0.8
Total	96.6	98.4	92.8	103.4	91.0	99.9

Table VI, Page 21, NAA-SR-1015

Because of the high vapor pressure of Mg at the melting point of uranium, initial experiments have been carried out at a lower temperature by using a U-Cr alloy (m. p. 859° C).

One experiment has been made using irradiated uranium ( $nvt = 10^{16}$ ). Briefly the procedure was as follows: A piece of U-Cr alloy containing tracer Pu was made by heating together a disc of nonirradiated U-Cr eutectic, a piece of irradiated uranium, and enough Cr metal to maintain the eutectic composition (5.19 w/o Cr). Approximately half of the clean button was then dissolved for a control, and the remainder was contacted with Mg at 950° C. The liquid metal extraction was done in a graphite crucible sealed under vacuum in a steel capsule. The capsule was heated in a crucible furnace. Because of temperature gradients in the furnace it was found that Mg condensed on the upper part of the capsule. To allow the Mg to return to the crucible after condensation, a steel "reflux condenser" was fitted into the top of the capsule. A diagram of the capsule arrangement is shown in Fig. 7.

After heating for 1 hour at 950° C the capsule was removed and cooled rapidly in an air stream. The steel tube was cut open and the U-Cr-Mg button removed from the crucible. The two metal phases were well bonded together. The Mg was separated from the U-Cr by dissolution in 1:1 acetic acid. The U-Cr was then dissolved in HCl and the resulting solution treated with  $HNO_3$ .

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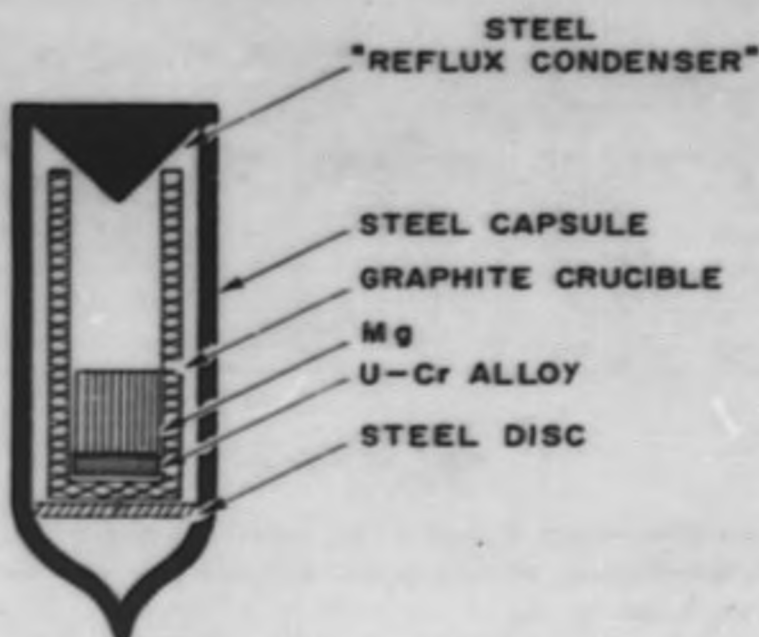


FIG. 7. CAPSULE ARRANGEMENT IN U-Mg EXPERIMENTS

Results of Pu analyses of these phases compared to the Pu in the control solution are shown in Table VII. It is seen that approximately 20 per cent of the Pu was extracted from the U phase by the Mg.

TABLE VII  
EXTRACTION OF TRACER Pu FROM LIQUID U-Cr ALLOY WITH LIQUID Mg

Wt U gm	Wt Ratio U-Cr/Mg	% Pu Found		
		U Phase	Mg Phase	Total
6.21	3.5	80	17	97

Further experiments are being made to check the Pu extraction. Although the fraction of Pu extracted by Mg under the above conditions was low, the use





of a Mg extraction process for the separation of Pu from U is still feasible since a refluxing type of scheme can be devised whereby a small amount of Mg can be recycled over the U fuel.

3. Calcium (R. A. Barney) - Experiments were initiated to study the extraction of tracer plutonium from irradiated uranium with molten calcium metal. A chromium-uranium eutectic containing 5 weight per cent chromium was used in the first experiments because of the low melting point (860° C) and the possibility of its use as a reactor fuel. However, it was found that the presence of this much chromium in the solutions for analysis lowered the plutonium recovery by as much as 20 per cent. It appears the chromium in the melt might decrease the plutonium recovery in the extraction also.

The experiments were made by heating tracer irradiated uranium, or an alloy made of irradiated uranium and 5 weight per cent chromium, with calcium just above the melting point. Samples of uranium from 7 to 15 grams were heated with 6 to 9 grams of calcium in a graphite resistance furnace under an argon atmosphere. The graphite resistance furnace was preferable to induction heating because of closer temperature control and lowered thermal shock for crucibles. Graphite crucibles were found most satisfactory for this study since they are easy to fabricate and were not affected by the metals at the temperatures employed. Calcia crucibles pressed at 15,000 psi and fired to 900° C were also satisfactory. They had the advantage of being easily dissolved to get a more complete material balance. The results obtained to date are given in Table VIII. One-cycle extractions of tracer plutonium from unalloyed uranium removed enough plutonium to warrant further study. In the future, larger quantities of high level irradiated uranium will be used to minimize surface effects and plutonium losses.

### III. SUMMARY

1. The results of treatment of X-10 fuel material with fused fluorides are similar to those found in tracer work. Plutonium was removed to about 90 per cent by  $UF_4$  and  $CaF-LiF$ .

2. Treatment of tracer-irradiated molten uranium with HCl gas removed Pu very well on small samples but not so well on larger samples.



TABLE VIII  
EXTRACTION OF TRACER Pu FROM U BY Ce

Metal	Temp °C	Time, Min	Crucible	Counts/gm of U		
				Control	Treated Uranium	% Pu Removed
U-Cr	700	70	Alumina	1480	910	38
U-Cr	900*	8	Alumina	1160	1040	10
U-Cr	1000	30	Graphite	1220	710	42
U-Cr	1000	30	Graphite			
Pure U	1135	10	Ta	1890	917	51
Pure U	1135	30	CaO	2180	925	79

\*Induction heated

3. The high temperature vacuum furnace for the scale-up distillation of plutonium from molten irradiated uranium was completed and tested at operating conditions.

4. The distillation of Pu from small pieces of X-10 material obeyed Raoult's Law to about 99 per cent removed.

5. Uranium oxide scavenger experiments with X-10 fuel material gave results in good agreement with previous tracer work. Plutonium was largely left in the metal while the rare earth elements, Sr, Te, and Cs were removed.

6. Treatment of a tracer-irradiated uranium amalgam with KCl-LiCl at 350° C showed some promise. Rare earth elements were removed from the amalgam to some degree while Pu was largely left behind.

7. Magnesium metal removed about 20 per cent of the Pu from a U-Cr alloy.

8. Calcium was found to remove as much as 80 per cent of the Pu from a sample of tracer-irradiated uranium.

**END**