Liquid-metal Extraction for Processing of Spent Fuel

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Plutonium and the fission products can be removed from irradiated uranium by liquid-metal extraction by use of another metal immiscible with uranium. Metals studied have been silver, cerium, and lanthanum. Plutonium removal by silver is high, by the rare-earth metals moderate. In all cases volatile elements, including cesium, strontium, and barium, are removed. Rare earths are efficiently removed. Ruthenium and molybdenum are largely unaffected. Experiments with synthetic fuels corresponding to long burn-up periods show improved removal of most elements. Repeated batch extractions indicate that a continuous process separating the fuel into uranium, plutonium, and fission-product fractions could be developed.

As the development of reactors to produce competitive electrical power will require low processing costs, some very different types of processing are being investigated. A successful process may mean the difference between a nuclear power industry which can truly compete in price with other fuels and one which must be subsidized to meet competition.

The basic idea behind the processes under investigation is quite different from that behind the ones that have been used to date. In the conventional process for the clean-up and reuse of nuclear fuel, the fuel metal is dissolved in acid and separated from fission products and other fissionable material (e.g., plutonium) with a high degree of purification before being refabricated into fuel elements. The degree of removal of the intensely radioactive fission products is sufficient to permit direct handling of the fuel elements in the refabrication and hence an elaborately designed fuel element is possible. However, another possible technique would be to provide in the separation only enough removal of fission products to negate any poisoning or impairment of metallic properties for which they might be responsible and at the same time by reforming the metal out of the molten state, to counteract any radiation damage which the metal may have sustained. Refabrication in this case would have to be done as a remote operation, and the fuel-element design would necessarily have to be simple to allow this.

The economic success of such a processing cycle would depend on the relative cost of complete chemical separation and direct refabrication on one hand and less complete, simple separation and remote fabrication on the other. Because there is a strong possibility that the latter alternative may be less costly, the authors have actively investigated simple types of processing.

The processing to be described falls in the general class of so-called "pyro-metallurgical" processes. Very early work (*1*) in this laboratory showed that this type of processing could give separations of considerable interest and promise. Renewed work from 1952 on has added much to the early data and carried the program to the stage at which scale-up experiments are in progress.

As has been described (*2*) the work at this laboratory has been concerned with liquid-metal extraction. In the phase systems of a number of metals, including silver, rare earths, magnesium, calcium, thorium, and others, with uranium there is an extensive immiscibility in the molten state. At temperatures just above the melting point the solubility of each phase in the other is a few per cent or less. Consequently a liquid-liquid extraction above the melting point is possible if provision is made for adequate mixing of phases and subsequent separation.

Experiments (*2*) showed that silver, cerium, and lanthanum were some of the metals of choice in such a process. They also indicated that silver was a good extractant for plutonium as well as for many of the fission products, but that cerium and lanthanum extracted less of the plutonium. With all these elements rather large amounts of the important fission products, with the exception of molybdenum and ruthenium, were extracted.

PREPARATION OF MATERIALS

Most of the previously reported experiments were done with irradiated uranium, which contained the fission products and plutonium at rather low concentrations. One of the observations from these experiments was that the concentration of the material extracted affected the extent of the extraction. Since actual power reactor fuels will in all probability be operated to a degree of burn-up quite in excess of any of the irradiated uranium which was available for experimentation, it became necessary to go to artificial fission-product–uranium mixtures, which have been called "fissium."

The composition of the fissium material used was determined by a thorough study of the fission chains and the feasibility of preparing alloys. The possible effect of varying the energy of the neutron spectrum responsible for the fission acts was considered, as were the recent observations on fission yield, fine structure, and the burn-up of materials of high cross section. Calculations for a 30-day irradiation period to 2% burn-up of U^{235} and no cooling gave the values in Table 1 for the atomic percentages of the most important elements resulting from the fission of U^{235}.

A practical artificial spent fuel was made up to be much simpler than this would indicate. The elements xenon, cesium, strontium, barium, krypton, rubidium, and iodine were eliminated from consideration because they are so volatile that their addition would be very difficult, and in any case their removal in any melting operation is easy and complete. In some cases a single rare earth, neodymium, was added to represent all the group, the amount added being equivalent to the sum of all the rare-earth percentages. In other cases cerium was considered separately and neodymium was

TABLE 1. ATOMIC PERCENTAGES OF THE ELEMENTS IN THE FISSION PRODUCTS

Element	At. %	Element	At. %	Element	At. %
Zr	15.0	Sr	6.6	Kr	2.14
Mo	9.20	Ba	4.57	Te	1.60
Xe	9.17	La	3.18	Rb	1.21
Ce	9.00	Tc	3.14	Sm	1.11
Nd	7.90	Nb	2.75	Pm	0.73
Ru	7.88	Y	2.66	I	0.73
Cs	7.83	Pr	2.26	All others	1.45

added equivalent to all the other rare earths. Examination of the table shows that with these reservations, the addition of four or five elements, zirconium, molybdenum, and ruthenium plus the chosen rare earth or earths, would take care of the first ten elements in the table plus a number of others. The elements added amounted to 59% of the total atoms and those known to be volatile amounted to another 32.2%, or a total of 91.2%. The actual composition of the fissium used is given in Table 2.

The fissium was made up as needed with radioactive isotopes of the elements of interest to permit analysis by radiochemical methods. In most of the experiments to be described, the added elements were made radioactive by irradiation in the Argonne National Laboratory CP-5 reactor. As the isotopes producible in cerium and ruthenium by n, γ reactions are less satisfactory for analytical purposes than Ce^{144} and Ru^{106}, which can be obtained as fission products from the Oak Ridge National Laboratory, cerium and ruthenium metals containing these tracers were made by reduction of the salts by the conventional methods. Table 3 lists the properties of the isotopes used.

The fissium was prepared in kilogram quantities by a process involving several steps. The refractory nature of some of the metals made a high-temperature melting process necessary in order to get them into the alloy. This was accomplished by arc melting all the alloying metals with about 100 g. of uranium. The resulting button of concentrated alloy was sectioned into quarters, which were placed in a tantalum crucible about 1 in. in diameter alternately among pieces of uranium metal weighing about 200 g. each. This assembly was melted by induction heating *in vacuo*, removed, inverted, and remelted.

The homogeneity of the samples was checked by means of radioautographs of the resulting cylinders and by scanning with a gamma-ray scintillation spectrometer. The radioautographs showed considerable concentration of some of the fission products at the crucible interface. Removal of the surface by machining removed most of this inhomogeneity, though there was still indication of a nonuniform distribution. The scintillation spectrometer was set at the characteristic gamma-ray energy of the particular nuclide of interest, e.g., 0.72 Mev. for zirconium, and by scanning the length of the cylinder through a collimating system one could observe the distribution of the particular element. Though some lack of uniformity was observed, it did not appear that repeated melting would help the situation, and the samples were used as produced. Other methods of preparing the material, such as a tilting resistance furnace, gave less satisfactory results.

TABLE 2. FISSIUM COMPOSITION, 2% BURN-UP

Element	Atom ratio	Weight %
U	98	99.5
Zr	0.30	0.117
Ru	0.156	0.068
Mo	0.184	0.076
Ce	0.180	0.108
Nd	0.358	0.220

TABLE 3. ISOTOPES USED FOR FISSIUM ANALYSIS

Element	Isotope	Half-life	Radiation energy, Mev.	Remarks
Zr	95	65d	$\beta 0.37$, $\gamma 0.72$	35-day Nb^{95} daughter introduces problems
Mo	99	66h	$\beta 1.2$, γ's	Short half-life is a severe limit
Ru	103	40d	$\beta 0.22$, $\gamma 0.5$	Soft β is difficult to measure accurately
Ru	106	1y	$\beta 0.04$ Rh$\beta 3.5$, γ's	Hard β of Rh daughter is easily measured
Ce	141	33d	$\beta 0.58$	Irradiated Ce contains 14-day Pr^{143}, which confuses issue
Ce	144	280d	$\beta 0.3$ Pr $\beta 3.0$	Hard β of Pr daughter is easily measured
Nd	147	11d	$\beta 0.83$	

TABLE 4. DISTRIBUTION COEFFICIENTS

Fission products	Extractants Ag	Ce	La	Legend Letter	Range
Mo	*B*	*C*	*B*	*B*	0.003–0.03
Ru	*B*	*D*	*D*	*C*	0.03–0.3
Zr	*D*	*D*	*D*	*D*	0.3–3
Nd	*E*	*E*		*E*	3–30
Ce	*D*		*E*		

TABLE 5. DECONTAMINATION

Fission products	Extractants Ag	Ce	La	Legend Letter	Dec. fact.	% removed
Mo	*P*	*P*	*P*	*P*	1–1.3	0–23
Ru	*P*	*Q*	*Q*	*Q*	1.3–1.8	23–45
Zr	*S*	*Q*	*S*	*S*	2.5–5	60–80
Nd	*T*	*U*		*T*	5–10	80–90
Ce	*S*		*S*	*U*	10–50	90–98

TABLE 6. FORWARD AND REVERSE EXTRACTIONS*

Fission products	Extractants Ag For.	Ag Rev.	Ce For.	Ce Rev.	La For.	La Rev.	Legend Letter	Distn. coeff.
Mo	*B*	*D*	*C*	*C*	*B*	*D*	*B*	0.003–0.03
Ru			*D*	*D*	*D*	*D*	*C*	0.03–0.3
Zr	*D*	*D*	*D*	*E*	*D*	*E*	*D*	0.3–3
Nd	*E*	*G*	*E*	*G*			*E*	3–30
Ce	*D*	*G*			*E*	*F*	*F*	30–100
							G	100

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*The ranges for the forward extractions are those given in Table 4; those for the reverse extractions represent two runs on each extractant.

The extraction experiments to be reported were each run with about 150 g. of uranium and an equal volume, roughly half this weight, of the extracting metal. Tantalum crucibles were used for the extractions. The samples were heated inductively *in vacuo* to 1,200° to 1,220°C. for 20 min. in all cases unless otherwise indicated.

The induction heating is known to provide sufficient stirring to mix the lighter metal and uranium phases. It is not known whether this stirring is vigorous enough to permit the system to approach equilibrium in the time taken for the experiments.

ANALYSIS

The original fissium melts were sectioned into samples for extraction, and between each extraction sample a sample was taken for analysis. Radiochemical analysis of these samples showed a non-

Table 7. Distribution Coefficients in Repeated Extractions

Fission products	Silver 1	Silver 2	Cerium 1	Cerium 2	Lanthanum 1	Lanthanum 2
Mo	*A*	*A*	*C*	*C*	*B*	*B*
Ru	*A*	*A*	*D*	*D*	*D*	*D*
Zr	*D*	*D*	*C*	*D*	*D*	*C*
Nd	*D*	*E*	*E*	*F*		
Ce	*D*	*D*			*E*	*E*

Legend

Letter	Range
A	0–0.003
B	0.003–0.03
C	0.03–0.3
D	0.3–3
E	3–30
F	30–100

uniform distribution of various activities along the cylinder which did not show up in the homogeneity studies previously mentioned. In general the fission-product content of the extraction samples, the original material for each extraction, was taken as the average between the two adjacent analytical samples, but if one of these appeared out of reason, a weighted average was used.

A decontamination factor was determined for each extraction, defined as the ratio of the amount of the element of interest in the original metal to that of the final extracted metal. For most purposes this is not considered as important a quantity as the distribution coefficient, which in our usage is defined as the ratio of the amount of the element of interest per gram of metal in the extractant phase to that quantity in the uranium phase.

Table 8. Decontamination in Repeated Extractions

Fission products	Silver 1	Silver 2	Silver Over-all	Cerium 1	Cerium 2	Cerium Over-all	Lanthanum 1	Lanthanum 2	Lanthanum Over-all
Mo	*P*	*P*	*P*	*P*	*P*	*P*	*P*	*P*	*P*
Ru	*P*	*P*	*P*	*P*	*Q*	*R*	*Q*	*P*	*Q*
Zr	*S*	*S*	*T*	*R*	*Q*	*S*	*R*	*S*	*S*
Nd	*S*	*T*	*U*	*T*	*U*	*V*			
Ce	*S*	*U*	*V*				*S*	*S*	*T*

Legend

Letter	D.F.	% rem.
P	1–1.3	0–23
Q	1.3–1.8	23–45
R	1.8–2.5	45–60
S	2.5–5	60–80
T	5–10	80–90
U	10–50	90–98
V	>50	>98

Analyses were by standard radiochemical techniques. In many cases the amount of an element in a particular phase was too small to follow through a chemical procedure without the addition of carrier. In these cases known amounts of carrier were added and the principles of the isotope dilution technique were used.

RESULTS

It was found that the reproducibility of the experiments was rather poor by the standards which are accepted for aqueous solution chemistry. The trends and orders of magnitude are, however, clearly established and the tables of data have been arranged to show the trends by indicating ranges of values rather than actual values. In the extraction and decontamination experiments summarized in Tables 4 and 5 an average of six determinations is represented by each given range.

In these first experiments in a new field it has not been possible to find the reasons for the lack of reproducibility and to obtain improvement. However, the principal cause of the lack of agreement is probably the sampling. It is quite possible that small particles of one phase may remain suspended in the other after the induction furnace has been turned off. As soon as the induction heating is stopped, the stirring ceases and settling begins. Cooling starts at the same time, however, and is fairly rapid and so the phases may not have time to separate completely. To remedy this situation a procedure has been initiated of reheating the melt in a resistance furnace to allow complete phase separation after the melt is removed from the induction furnace.

In order to ascertain whether or not the extraction represented equilibrium conditions, the extractant metal phase from some extractions was reextracted with inactive uranium. The results of these experiments (Table 6) show that in some cases distribution coefficients in the same range were obtained independently of the direction of approach, for example, in the extraction of ruthenium into cerium or lanthanum. More frequently, however, the reverse-direction extraction showed a higher value of the distribution coefficient than the forward extraction, which can be explained by a lack of attainment of equilibrium in the system. More vigorous stirring or longer heating was a possible aid in the solution of this difficulty.

Another series of experiments was designed to determine whether initial decontaminations and distributions could be duplicated in a second extraction of the uranium phase with the same metal. Two parallel runs were made for each extractant, and the results for distribution coefficient and decontamination are given in Tables 7 and 8, respectively. It can be seen that similar distributions and decontaminations were obtained in the first and second extractions. Comparison of duplicate runs shows that consecutive extraction experiments give just as good agreement as duplicate runs. The indication is that scale-up to continuous or repeated batch extraction would be possible.

The fissium used in these experiments did not contain plutonium and no plutonium data are available from these experiments, but a restatement of the earlier runs on plutonium is important in order to consider the process as a whole. Table 9 contains a summary of the plutonium data (*2*) at two levels of plutonium content: medium, about 18 mg. of plutonium/kg of uranium, and high, about 230 mg. of plutonium/kg. of uranium. The distribution coefficients for silver are high enough to indicate complete removal if the process is repeated a small number of times. With cerium, a number of repetitions would be necessary; with lanthanum, removal of plutonium would be difficult. Somewhat lower removal by lanthanum would allow its use to remove fission products while leaving the plutonium in the fuel, but with the removal as large as is indicated, this would probably give too large a loss of plutonium to be feasible.

Table 9. Plutonium Extraction

Extractant	Distribution coefficient: Medium level	Distribution coefficient: High level	Decontamination factor: Medium level	Decontamination factor: High level
Ag	4.6	7.0	3.7	5.1
Ce	0.95	1.02	1.50	1.58
La	0.65	0.56	1.28	1.22

TREATMENT OF THE EXTRACTANT PHASE

Removal of fission products and plutonium from uranium by such an extraction process must be followed by a step to clean up the extractant phase if the process is to be of much value in a fuel cycle. Methods of cleaning up the extractant phases have been under investigation for some time with particular emphasis on the silver phase resulting from extraction of uranium with silver.

Some of the procedures tried to clean up the silver have been electrorefining with an aqueous bath, electrorefining with fused salt baths, and treatment with molten salts containing silver chloride. In the electrorefining experiments the contaminated silver is made the anode and a silver rod is used as the initial cathode. As the electrolysis progresses the cathode grows at the expense of the anode. A silver cathode which is decontaminated from those impurities which were present in the silver anode is obtained as a result either of lack of oxidation of the impurities at the anode leaving them in the form of a sludge or lack of reduction at the cathode leaving them in the electrolyte. Large decontamination factors were obtained from both of the electrorefining procedures, but the aqueous studies were abandoned. The aqueous bath required to obtain a smooth adherent deposit was so filled with complexing agents and other reagents that its stability toward the radiation which it would receive in actual use appeared highly problematical.

The fused-salt electrorefining studies are continuing with quite promising results. However, problems exist. Deposits tend to be crystalline and to form long streamers from the electrode. Owing to the large surfaces of these crystalline deposits the amount of adsorption and trapping of the electrolyte within the cathode deposit is considerable. Solutions of these problems appear to be at hand to permit further studies on decontamination.

The direct treatment of the contaminated silver with molten silver chloride has difficulties also, particularly in the choice of materials for use as containers. Glass is unusable at the temperature of molten silver, and metals are rapidly chlorinated by hot silver chloride. Silica crucibles have been found to hold the mixture and these are at present in use. Decontamination results are not available for many elements as, for example, so little ruthenium or molybdenum go into silver that their removal is hardly a problem. Results for fission-product cerium, which does extract into silver, indicate a removal of 97 to 99.6% in a single heating with silver chloride or silver chloride–sodium chloride mixtures containing 10 to 90 mole % silver chloride. Results for zirconium similarly indicate removal of 96 to 99.9% of the fission product from silver in such extractions. Thus it appears that the silver layer can be freed of at least some of its fission-product load after the silver extraction of uranium.

CONCLUSIONS

In estimating the value of this type of processing it is important to review the reasons for reprocessing fuel. Foremost for many reactors will be the cure of radiation damage. Changes occur in thermal conductivity, strength, and even shape of solid fuel elements in which fission is occurring at a high rate. These make it necessary to limit the time of exposure of the fuel elements and to reprocess at shorter intervals than would be required for any other reason. The fission products which in being slowed down give rise to this radiation damage may also just by their presence in the metal change its strength or other metallic properties or its resistance to radiation damage. Hence, their removal may be essential. Little is known about this aspect of the problem as it involves studies on complex alloys with small amounts of many elements very uniformly mixed through the alloy, a highly complicated system which it has not been possible to duplicate.

A third reason for reprocessing is the removal of fission products because of their neutron-absorbing tendency. The urgency for this depends on the type of reactor in question, as the neutron absorption by different fission-product nuclides is highly dependent on the neutron energy. Thus for a thermal reactor, removal of certain poisons may be of prime importance while for a fast reactor there are no nuclides of particularly serious poisoning ability. The reprocessing step should also provide for the replenishment of the used fissionable material in the fuel element, for example, by allowing the addition of highly enriched U^{235} to a fuel mixture of moderate enrichment.

Most of these ends can be accomplished by pyrometallurgical processing. The healing of radiation damage in a solid fuel can certainly be done by a melting and recasting process provided the fuel element is of such design that it can be fabricated remotely. Since the resistance of an alloy to radiation damage depends on its microstructure, it is also essential that the most resistant microstructure be obtainable by remote fabrication.

It is not known which of the fission-product elements may impair the structure of the metal and its resistance to radiation damage, which ones will have no effect, and which ones may actually improve the metal. It would be expected, however, that the presence of a nonmetal atom like xenon or a large atom like cesium would tend to impair rather than to improve. Thus the rare gases, the alkali and alkaline earth elements, may be the most important elements to remove from the standpoint of metallic structure. These, as it has been noted, are the particular elements that are removed by volatility in pyrometallurgical processing. Experiments not reported here (*2*) show that the decontamination factor for cesium and strontium in a single liquid-metal extraction step is of the order of 500 to 10,000 at the highest fission-product concentrations in which the process was tested, a level which corresponded to a burn-up of about 0.03%. Since these decontamination factors were considerably higher at high concentrations than at low, it would be logical to assume even better decontamination factors if the experiments could be run at a level corresponding to 2% burn-up.

Those elements which are removed specifically by extraction rather than volatilization, particularly the rare earths, might or might not be harmful to the metallic properties. At the present time too little is known about this to permit even a guess. Those elements which are not removed, such as ruthenium and molybdenum, might at low concentrations actually improve the metal. At least there is no information either of a practical or theoretical nature which would indicate that they have a large adverse effect on the metal. The radii of these atoms are such that they might fit into the uranium lattice without producing excessive distortion.

Thus, although the evidence is still meager it appears that pyrometallurgical processing would remove those fission-product impurities most likely to distort the uranium lattice by their presence. Those elements which it does not remove would be expected to have much less deleterious effect and possibly even to be beneficial.

As far as poisoning by neutron absorption is concerned, in a thermal reactor a very large part of this is in Xe^{135}, which is transitory and of minor importance in a processing cycle. Except xenon, most of the poisoning is in the rare earths, samarium, gadolinium, neodymium, and europium, and in iodine. Of these elements the rare earths are extracted to a large extent in the liquid-metal process and the iodine would in all probability be lost by decomposition of uranium iodides at the temperature of the extraction. The next group of fission products in order of decreasing importance as poisons in a thermal reactor are cadmium, strontium, cesium, rhodium, and krypton. By this time the contribution of each element to the poisoning is small. The authors have no information about cadmium and rhodium, but the rest of this group is completely removed by volatility.

In a fast reactor the problem of fission-product poisoning is minor and is a much less urgent reason for reprocessing than it is in a thermal reactor. The major contributors to poisoning in this case are elements which have been found to be fairly difficult to remove, such as zirconium, ruthenium, and molybdenum. However, enough of the zirconium is removed along with those elements which contribute less but still appreciably to the poisoning effect so that this effect is reduced to approximately half of its original value by a single extraction step.

It is clear that while a metal is in the molten condition additional fissionable material could be added and distributed uniformly through the metal.

Thus pyrometallurgical processing, though in its very early development stages compared with more conventional types, seems to have promise of accomplishing the goals of a processing cycle at potentially less cost. To achieve the final development of such a process will require a prolonged effort on the part of metallurgists, chemical engineers, and chemists. Effort in this laboratory is continuing with experiments on scaling up and on the development of continuous extractors to operate at this temperature.

The results with the artificial fissium mixtures are not very satisfying owing to the lack of agreement in the results. Unless some improvements are made in this agreement, it may be necessary to go back to real fuels of lower burn-up and to extrapolate to the expected burn-up or to wait until fuels of high burn-up are available and have decayed to a level of radioactivity which will permit laboratory handling. Other techniques of making fissium may be more successful and will also be tried.

Before closing, it would be well to point out that this type of processing is also uniquely adapted to use with a liquid-metal fuel reactor. If the other problems which are peculiar to the use of liquid-metal fuels are solved, it is probable that there will be a strong future for pyrometallurgical processing in the reprocessing cycle of a liquid-metal reactor.

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Use of Zirconium in Liquid-sodium Systems

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The attractive nuclear properties of zirconium make it a highly desirable core material for sodium-cooled reactors. The elevated temperature strength while low is sufficient for certain applications. Development of higher strength alloys is underway. Sodium in itself is completely compatible with zirconium; however, the nonmetallic contaminants, namely oxygen, hydrogen, and nitrogen, can effect serious damage. The primary problem in the use of zirconium in a sodium system, then, lies in controlling these impurities in the sodium.

In surveying the field of materials possessing potentialities in nuclear reactor construction, one is immediately attracted to zirconium because of its low capture cross section for thermal neutrons. The fact that during the initial period of reactor design and construction metallic zirconium was essentially a laboratory curiosity resulted in the use of aluminum as the primary metallic nonfuel core material. However, with the advent of the submarine reactor and the necessity for higher operating temperatures for feasible power production, the limitations of aluminum were exceeded. Neutron economics then demanded that the application of zirconium be completely investigated. The success of this investigation and the subsequent development program is amply demonstrated by a survey of the literature.

As the primary objective of the zirconium development program was the Submarine Thermal Reactor, or S.T.R., it was natural that water-corrosion resistance was of the utmost importance. As a consequence, until very recently essentially all processing and fabrication developments have been directed toward eliminating those impurities found to be detrimental with respect to corrosion. The fundamental objective of the alloy-development program also was the compensation for those deleterious impurities which cannot conveniently be removed.

The current interest in nuclear station-