

Assessment of a U Product Purity from Pyroprocessing Spent EBR-II Fuel

EBR-II 사용후핵연료의 건식처리공정에 의한 우라늄의 순도 평가

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Abstract

A comprehensive analysis has been conducted on the purity of the uranium product generated from a pyroprocessing of EBR-II spent fuel. The analysis results were compared to the low-level waste criteria for both ROK and USA under a collaborative program between INL and KAERI. It is found that the US LLW definition does not include the activity from any U isotopes, but the Korean one does. The analysis results show that Pu-239 is the only alpha emitting isotope other than U isotopes that exceed the limit in the EBR-II U product. Pu contamination of the product seems to be drastically reduced in a preliminary test of the modified cathode process, and the further development of the proposed technology may be possible to meet the US LLW criteria.

Key words : Pyroprocessing, EBR-II, purity, LLW criteria

요 약

EBR-II 사용후핵연료의 파이로건식처리공정에 의해 발생된 우라늄의 순도에 대한 포괄적인 분석을 수행하였다. 분석결과를 미국 아이다호 국립연구소 및 한국원자력연구원의 협력과제 하에서 한국과 미국의 저준위 폐기물 기준으로 비교하였다. 미국의 저준위 폐기물 기준은 우라늄 동위원소를 포함하지 않으나, 한국의 경우는 포함하는 것으로 조사되었다. 분석결과 EBR-II 우라늄 생성물 내에서 저준위 기준을 초과하는 유일한 알파 핵종은 우라늄 동위원소가 아니라 Pu-239였다. 생성물 내의 Pu 오염은 개량된 염증류공정을 통한 예비 실험 결과 획기적으로 줄일 수 있음을 알 수 있었으며, 보다 공정을 개선 시킨다면 제안된 기술을 이용하여 미국의 저준위 기준을 만족시킬 수 있을 것으로 판단된다.

중심단어 : 파이로건식처리공정, EBR-II, 순도, 저준위폐기물기준

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I. Introduction

In recent years, there has been a renewed interest in recycling spent nuclear fuel. Among the recycling technologies, a pyrometallurgical process has been accepted as one of the promising technologies to treat spent fuel and to reduce its volume [1~6]. Recently it has become one of the important requirements to recover long-lived nuclides, since the geological disposal of high level waste is encountering resistance to obtaining public acceptance. Also, the recovered uranium and transuranics (TRU) can be re-utilized as a fuel source. From this point of view, the recovering of long-lived nuclides is awakening to an interest in the fuel cycle process for the partitioning and transmutation of transuranic elements. For the sake of a spent nuclear fuel volume reduction, Korea Atomic Energy Research Institute (KAERI) is conducting a comprehensive study under a long-term plan for the nuclear technology development set up by the Korean government. The studies are focused on the development of a pyroprocessing, because it is accepted as a proliferation-resistant technology and well suited for the fuel cycle of a sodium cooled fast reactor with metal fuel.

The major experimental technologies for the pyroprocessing study include electroreduction, electrorefining, electrowinning and molten salt waste treatment. Thus far, thermodynamic studies and scale-up tests have been pursued in order to examine the applicability of electrolysis of molten salts to operations at a meaningful scale. However, those investigations have been conducted with simulated spent fuel without TRU, which is one of the main concerns in terms of the geological disposal or reuse of spent fuel. Over the long-term implementation of the nuclear fuel cycle, most of uranium and TRU should be used as fast reactor or burner fuel, while excess uranium may be stored for future use. In this case, the storage cost of the excess uranium would drastically be decreased if the activity of TRU in uranium is low enough to satisfy the low level waste (LLW) criteria.

1. Low-Level Waste Criteria

① Korean Requirements

Radioactive waste is divided into two classes under the Korean law. One is low and intermediate-level radioactive waste (LILW), and the other is high-level radioactive waste (HLW). HLW contains more than 3,700 Bq/g and 2 kW/m³ of alpha emitting radionuclides with half-lives greater than 20 years [7]. In other words, LILW contains less than 3,700 Bq/g or 2 kW/m³ of alpha emitting radionuclides with half-lives greater than 20 years. Figure 1 shows that LILW region based on this classification is very wide.

A LILW disposal repository is being constructed for completion by the end of 2011 in Korea. Regulation of a waste delivery to the disposal site was announced in June of 2005 as a part of the Korean law. The delivery of LILW to the repository requires that the concentration of each radionuclide does not exceed the ROK LILW limit in Table 1. Note that the waste delivery criterion is more restrictive than the general LILW criterion. Therefore, it is deduced that waste can be disqualified from being eligible for a disposal in the LILW repository by either having an activity level of 3,700 Bq/g or a heat loading of 2 kW/m³.

② U.S. Requirements

By contrast with Korea, the US LLW definition does not include the activity from any U isotopes. But it is stipulated that any material that originates from HEU must be downblended to no more than 0.9 % U-235. It is, thus, possible that the US LLW criteria might be achievable from pyroprocessing spent fuel. Table 1 shows how the product from a pyroprocessing of EBR-II

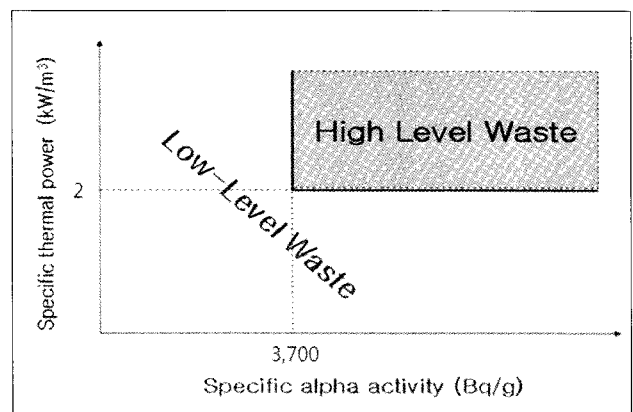


Fig. 1. Low level waste region in Korea

Table 1. ROK and US LLW Limits Compared to EBR-II Product

Radionuclide	ROK LLW Limit (Bq/g)	US LLW Limit (Bq/g)	EBR-II Driver U Product* (Bq/g)	EBR-II Blanket U Product (Bq/g)
H-3	1.11×10^6	NA	NA	NA
C-14	2.22×10^5	1.56×10^5	NA	NA
Co-60	3.70×10^7	NA	NA	NA
Ni-59	7.40×10^4	4.28×10^5	NA	NA
Ni-63	1.11×10^7	NA	NA	NA
Sr-90	7.40×10^4	NA	NA	NA
Nb-94	1.11×10^2	3.89×10^2	NA	NA
Tc-99	1.11×10^3	5.84×10^3	NA	Not Detected
I-129	3.70×10^1	1.56×10^2	NA	NA
Cs-137	1.11×10^6	NA	NA	NA
Alpha#	3.70×10^3	3.70×10^3	7.48×10^3	3.00×10^6

Includes uranium isotopes for ROK and excludes them for US.
 * Downblended to 0.9 % U-235 per Federal Register, Vol. 61, No. 151/40619 (1996).
 NA : not analyzed

spent fuel measures up to the ROK and US LLW limits. The EBR-II product activities included in this table are the maximum observed to date for each fuel type. They do not include the activity from U isotopes, so they really can only be compared to the US LLW limit. In the case of the EBR-II driver product, the limit is only missed by a factor of 2. For the EBR-II blanket product, the limit is missed by three orders of a magnitude.

Hence, the purpose of this paper is to introduce the analysis results of the uranium product generated from a pyroprocessing of metallic spent fuel. Idaho National Laboratory (INL) has reviewed the low-level waste criteria for both ROK and USA, reviewed historical uranium product data from processing Experimental Breeder Reactor-II (EBR-II) fuel and compared the historical data for the LLW criteria as a collaboration program with KAERI. A strategy for improving the uranium product purity has also been described.

II. Experimental

The Fuel Conditioning Facility (FCF) at INL treats spent metal fuel from EBR-II using an electro-metallurgical treatment. The processing equipment used in FCF is shown in Fig. 2. Driver fuel elements are chopped into segments 6.3 mm long at the element chopper, loaded into steel baskets, and immersed into the electrolyte of the Mark-IV electrorefiner (ER). The Mark-IV ER, which processes

primarily driver fuel, is maintained at 773 K and contains a molten LiCl-KCl-UCl₃ electrolyte floating above a liquid cadmium bath. Blanket fuel pins are chopped into segments 18.3 mm long at the blanket chopper, loaded into steel baskets, and immersed into the electrolyte of the Mark-V ER. The Mark-V ER, which processes blanket fuel, is also maintained at 773 K and contains only a molten LiCl-KCl-UCl₃ electrolyte. In both ERs, uranium is electrochemically transported to a steel cathode. The ER products, consisting of metal and salt, are sent to the high temperature vacuum furnace (commonly known as a cathode processor (CP)), to separate the salt from the metal using a vacuum distillation. The CP also consolidates the remaining metal and any additional depleted uranium (if necessary) to produce a homogeneous ingot.

If the CP ingots are from a blanket processing, a drill sample of the CP ingot is taken for an analysis. If the CP ingots are from a driver processing, those ingots are sent to another high temperature furnace called a casting furnace (CF) for a further processing. The CF consolidates the CP ingot with additional depleted uranium to produce a low enriched uranium (LEU) product. A pin sample is taken from the CF for an analysis. The drill samples and pin samples are analyzed to assess the uranium product purity.

III. Result and Discussion

1. Product Purity from Processing Spent EBR-II Fuel

Table 2 shows the averaged compositional data for

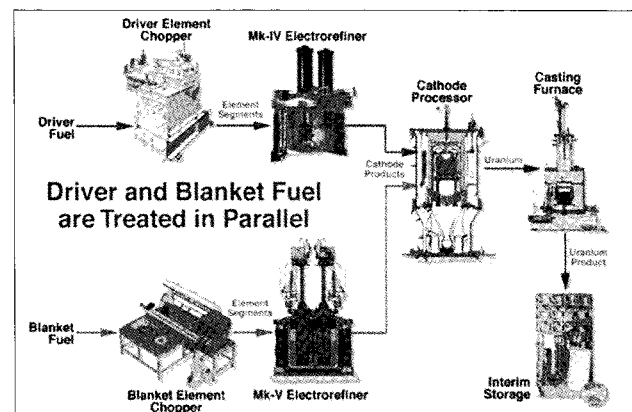


Fig. 2. Spent Fuel Treatment at Fuel Conditioning Facility

selected analytes from seventy-two blanket uranium products. Blanket uranium products are defined as those irradiated products from the Mk-V electrorefiner including blanket product collector (BPC) heel material since the start of operations in June 1998. Although forty-two analytes are routinely analyzed for, a limited sub-set is reported in this table due to their steady occurrence in the blanket products. Besides, the averaged data, the standard deviation data as well as the minimum and maximum values are also shown in this table. The data for the driver uranium products are included in Table 2 for a comparison and will be discussed in the following section. All of the selected analytes listed in Table 2 except the total uranium are results of chemical analyses. A total uranium quantity for the blanket and driver products are calculated by subtracting those impurities detected by analytical techniques from unity [8]. A total uranium quantity was determined directly by analytical techniques and compared to the calculated value for validation. Though the driver product was downblended with U-238 in the CF prior to sampling, the concentrations given in Table 2 have been adjusted to factor out the downblending. In other words, both the

blanket ingot and driver ingot numbers are representative of the concentrations coming out of the CP.

For the blanket uranium products, it is interesting to note that if the calculated values for the total uranium from the BPC heels and irregular batches (i.e. special experiments) are excluded from the average, the average of the total uranium would be 99.81 wt.% or 1884 ppm in purity. This value would meet the August 1994 fuel specifications for EBR-II fuel. This specification stated that the total C+N+O+Si contamination should be less than 2000 ppm, and the rare earth element contamination should be less than 5000 ppm.

The exceptions, most recently, are due to several batches of BPC heels and special experiments. The heel material has been slightly richer in impurities than normal blanket uranium products. Special experiments with copper chloride in the cathode processor have resulted in the total uranium values being as low as 96.7 wt.%. Of the seventy-two blanket batches analyzed in Table 2, only twenty-nine batches were sampled following the cathode processor operation. The other forty-three batches were sampled following the casting furnace operation. Since the sample size from the cathode processor ingot is not

Table 2. Selected Analytes from Blanket and Driver Uranium Products

Analyte #	Units	Driver Ingots				Blanket Ingots			
		Ave.	Std.Dev.	Min.	Max.	Ave.	Std.Dev.	Min.	Max.
U *	wt%	99.32	0.85	95.34	99.93	99.59	0.71	96.69	99.9
Zr	wt%	1.65	2.9	0.03	13.87	0.03	0.02	0.01	0.13
O	ppm	419	412	23	2981	196	116	40	749
C	ppm	734	630	38	5926	455	416	74	1803
Si	ppm	560	391	30	1716	158	192	50	1167
Fe	ppm	652	405	48	2688	1178	2396	263	16933
Cr	ppm	238	242	47	1227	150	227	33	1567
Ni	ppm	302	195	61	947	102	78	45	587
Mo	ppm	807	684	98	3173	196	460	55	2715
Tc	ppm	63	127	13	362	29	12	20	48
Sm	ppm	48	29	16	69	†	†	†	†
¹⁴⁴ Ce	ppb	1.8	1.9	0	8.5	0.1	0.1	0	0.5
¹⁵⁴ Eu	ppb	6.7	12.2	0.1	78.7	2.3	2	0.1	10.7
¹⁵⁵ Eu	ppb	13.1	28.5	0.1	217.3	3	3.9	0.2	18.2
¹⁰⁶ Ru	ppb	27	145	0	1224	25	31	1	135
¹²⁵ Sb	ppb	12	64	0	544	21	15	1	80
¹³⁷ Cs	ppb	150	267	0	1498	57	64	1	324
²³⁷ Np	ppm	91	147	10	628	4	6	0	91
²³⁹ Pu	ppm	48	45	6	225	282	291	34	1473
²⁴¹ Am	ppb	116	†	116	116	172.5	195.5	20.2	686.8

La, Pr, and Nd before their respective detection limit. / * Uranium = 1 - sum of impurities.

† No measurements above the detection limit. / ‡ Only one measurement above detection limit.

adequate for oxygen and carbon analyses, the data in Table 2 for oxygen and carbon are only from casting furnace samples.

The main source of zirconium in the driver uranium products is from carryover during electrofining operations from metallic fuel (U-10Zr), although a minor amount is from the zirconia coating used on the cathode processor crucible. The primary source of oxygen, carbon, and silicon is probably from the impurities in unirradiated original fuel or diluent DU, although alternative sources do exist during a processing (i.e. coatings, graphite, and glass molds). Iron, chromium, and nickel also derive from original fuel, DU, and from the cladding or steel components of the process. The most likely source for molybdenum in the driver products is from the DU used as a diluent although the cladding may have a small contribution. The isotopes are a result of fission during EBR-II operations and have not been normalized for decay over time.

Similar explanations for maximum values can be made for the driver uranium products as for the blanket products: carbon due to graphite, steel constituents (iron, chromium, and nickel) due to component dissolution, and gamma isotopes (Ru-106, Sb-125, and Cs-137) due to the start of processing.

As previously mentioned, driver uranium products require a dilution during a processing from a highly enriched (>20 wt. % U-235) to low enriched (<20 wt. % U-235) uranium. The dilution is performed at both the cathode processor and casting furnace operations with depleted uranium (DU) additions. It is partially for this reason that no sampling is performed on cathode processor ingots for blanket products. Samples are only taken following the casting furnace for the driver uranium products.

Figure 3(a) shows the activity of various alpha emitters in the product from the EBR-II driver fuel electrorefined in the Mark-IV electrorefiner. Any of the individual nuclides (U-234, U-235, U-238, U-236, and Pu-239) would by themselves cause the alpha activity limit to be exceeded. Similarly, it is shown in Figure 3(b) that the product from electrorefining the EBR-II blanket would also exceed the

alpha activity limit. Before completely dismissing the prospect that the pyroprocessing of spent fast reactor fuel can result in a product that can be disposed of as LLW, refer back to Figures 3(a) and (b) where it was shown that Pu-239 is the only alpha emitting isotope other than U isotopes that exceed the limit. If applicable technology can be developed or modified to reduce this Pu contamination of the product, then it may be possible to meet the US LLW criteria. And that set of criteria may ultimately be implemented in Korea.

2. Effect of Pu/U Ratio on Pu Contamination of Product

As shown in Table 1, alpha emitters are the primary impediment to the EBR-II product qualifying as LLW, and Pu-239 is the primary alpha emitter. It is, thus, potentially

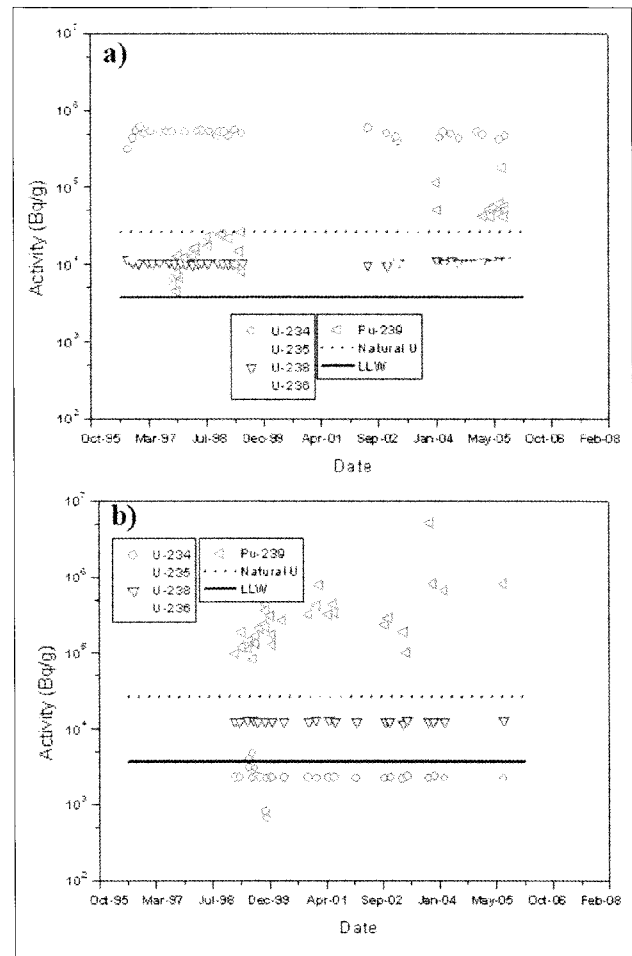


Fig. 3. Activity of alpha emitters in product from electrorefining EBR-II driver a) and blanket b).

valuable to investigate the factors that lead to high Pu-239 contamination levels. Understanding how the contamination occurs can naturally lead to inventing a technique or method to mitigate the problem. Figure 4(a) shows the Pu-239 concentration in the product for the blanket fuel as a function of the Pu:U ratio in the salt at the time of electrorefining the corresponding product batches. For the sake of using the data in Figure 4 to understand the underlying physical/chemical processes occurring in a pyroprocessing, a model was developed to calculate the Pu contamination as a function of the Pu:U ratio. The most important aspect of this model is that the U product has essentially no Pu contamination when it comes from the electrorefiner and that all such contaminations occur as a result of a cathode processor operation. In the cathode processor, salt and metal coexist at a high temperature until the salt evaporates and collects in the salt receiver. While the salt and metal are still in contact, the following chemical reaction can presumably occur and reach equilibrium.



The equilibrium constant K for this reaction can be written as follows, where a_i is the activity of reactant i .

$$K = \frac{a_{UCl_3} a_{Pu}}{a_U a_{PuCl_3}} \quad (2)$$

Then K can be determined from the free energy change of reaction (G_{rxn}), which has been cited to be 88 kJ/mole at 500 °C [9]. Note that the actual temperature in the cathode processor is much higher, but that thermodynamic properties such as the activity coefficients are not available at higher temperatures.

$$K = e^{-\frac{\Delta G_{rxn}}{RT}} = 1.13 \times 10^{-6} = \frac{\gamma_{UCl_3} \gamma_{Pu}}{\gamma_{PuCl_3}} \frac{x_{UCl_3} x_{Pu}}{x_{PuCl_3}} \quad (3)$$

Where, γ_i and x_i are activity and concentration of reactant i respectively.

An immanent assumption in the above equation (3) is that the activity of U is unity, since it is present in the

metal product at a 99+% concentration level. Different estimates have been made for the activity coefficients in various sources. There are, however, a lot of unavoidable errors in their values as well as those of the equilibrium constants. The equilibrium reaction between U and Pu occurs in the cathode processor over a range of temperatures up to around 1000 °C when the salt is separated from the metal via a distillation. All of the equilibrium constants are likely to have the dependence of temperature. Rather than try to estimate what effect the temperature has on the literature-based values of the constants, it was decided to simply group the activity coefficients into a single parameter and find the best fit for that parameter. The dotted line in Fig. 4(a) shows the modeled results. For this fit of the model, the value of the grouped parameters is given below,

$$\chi = \frac{\gamma_{UCl_3} \gamma_{Pu}}{\gamma_{PuCl_3}} = 0.0014 \quad (4)$$

Other assumptions for this calculation include 20 % salt and 80 % metal in the cathode processor and a total of 4 wt% actinides in the salt. The relative amount of salt and metal is important in determining at what point the curve starts to approach an asymptotic value. The model can be improved by including actual data for actinide concentrations in the salt and the ratio of salt to metal in each cathode processor batch. But this modeling exercise was only intended as a first order attempt to capture the effect of the Pu/U ratio in the salt on the Pu contamination in the U product.

The curve fit in Fig. 4(a) was not intended to yield a precise parameter that can be used for process calculations. Rather, it was intended to demonstrate that this basic model is consistent with the presented data. More process data and a computational optimization are necessary to derive a useful value for χ . Assuming the model and parameter estimation are roughly accurate, Pu contamination is expected to peak out at close to 2000 ppm even under conditions that support a U/TRU recovery in an liquid cadmium cathode (LCC). In Fig. 4(b), the Pu-239 contamination data as a function of the Pu/U ratio in the Mark-IV electrorefiner is plotted along

with the modeled results. As with the blanket product, it was assumed that the CP contained 20 % salt and 80 % metal, and the total actinide concentration was assumed to be 4 wt%. The same value of $\hat{\rho}$ calculated from the fit of the blanket data was used, and as seen in Fig. 4(b) does execute a good job of fitting the driver data. There is, thus, consistency between the driver and blanket product purity with respect to effect of the Pu/U ratio in the salt.

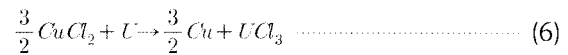
3. Strategies for an Improvement

In developing a strategy for improving the U product purity and meeting US LLW requirements, the focus should be on the Pu contamination. Over long fuel treatment campaigns, the Pu/U ratio in the electrorefiner salt will need to be at a level high enough to support a

group actinide extraction. This typically means that the Pu/U ratio should be 3 or higher. Without any change in electrorefiner or cathode processor operating parameters, the product is expected to contain in excess of 1200 ppm Pu. One approach to addressing this problem is to shift the equilibrium for the following reaction towards the products [10].



Experiments have been performed, which increase either directly or indirectly the amount of uranium trichloride in the salt in order to accomplish such a shift in the equilibrium. Those tests were successful in reducing the plutonium levels in the blanket uranium products but were discontinued due to the availability of uranium trichloride. An indirect method can also be used, which produces UCl₃ from the U metal in the product. Consider the reaction of cupric chloride with U.



This reaction was tested in a cathode processor. Cupric chloride was chosen as an oxidant based on its melting and boiling points, its cost, and the downstream effects on the uranium products. Results with cupric chloride on the reduction of plutonium in the uranium products are shown in Table 3. The quantity of the cupric chloride addition controls the final mole ratio of the plutonium trichloride to the uranium trichloride in the pre-cathode processor salt. The reaction time is defined as the start of the uranium trichloride vaporization to the end of the plutonium trichloride vaporization and can be controlled via cathode processor operating conditions. As can be seen in the data, the highest plutonium removals were

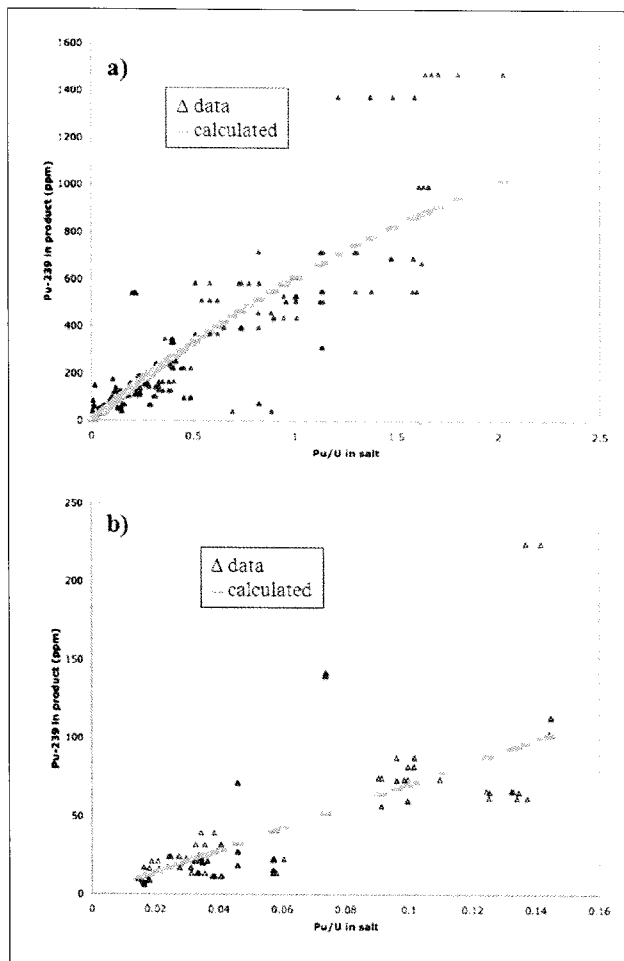


Fig. 4. Comparison of the model results with blanket (a) and driver (b) product Pu-239 contaminations as a function of Pu/U ratio in the Mark-V and Mark-IV electrorefiner, respectively.

Table 3. Results of three tests with cupric chloride for the reduction of plutonium

Test	Initial Mole Ratio PuCl ₃ :UCl ₃	Final Mole Ratio PuCl ₃ :UCl ₃	Reaction Time (h)	Plutonium Removed (%)
1	0.29	0.06	0.6	94
2	0.49	0.06	1.7	55

achieved by decreasing the reaction time. The plutonium reduction tests were performed early in the blanket processing program and have not been repeated with the present Pu:U ratios. Additional tests should be run that examine the utilization of multiple extraction steps, decreasing the final Pu:U ratio, and reducing the reaction time.

Based on a plutonium removal of approximately 90 %, at least two CP runs would be necessary to meet the US LLW requirements with the final ratio of Pu:U being reduced to less than 0.003. It is not known what effect a reduced reaction time would have on a plutonium removal rate. But if it can improve the Pu removal rate to 98 %, only one CP run would be needed to achieve the US LLW limit for Pu. This is a very promising result in that the excess uranium could be classified as a US LLW through an appropriate treatment with the pyroprocessing technology.

IV. Conclusions

An analysis has been conducted on the purity of the uranium product generated from a pyroprocessing of EBR-II spent fuel as a collaboration program between INL and KAERI. The analysis results show that Pu-239 is the only alpha emitting isotope other than the U isotopes that exceeds the LLW limit in the EBR-II U product. Pu contamination of the product as a metallic form is attributed to the reverse reaction between the PuCl_3 and U deposit during the cathode process. However, the Pu contamination is drastically decreased with a preliminary test for a modified cathode process by introducing additional CuCl_2 as a substituted substance for UCl_3 . Hence, a modification of the current cathode process for facilitating an evaporation of PuCl_3 , such as low temperature evaporation and multi step cathode processes, may be possible to meet the US LLW criteria.

Acknowledgments

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