

**ANODIC PROCESS OF ELECTROREFINING  
SPENT NUCLEAR FUEL IN MOLTEN LICl-KCl-UCl<sub>3</sub>/CD SYSTEM**

by

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# **ANODIC PROCESS OF ELECTROREFINING SPENT NUCLEAR FUEL IN MOLTEN LiCl-KCl-UCl<sub>3</sub>/Cd SYSTEM**

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## **ABSTRACT**

This article summarizes the experimental results and engineering aspects regarding the anodic process for electrorefining 100 irradiated driver fuel assemblies, a demonstration project for the Department of Energy (DOE) to treat spent nuclear fuel. The focus is on the anode due to its unique geometry (fuel dissolution baskets loaded with chopped irradiated fuel segments), complex chemical compositions, highly demanding process goals and their significance to the entire spent fuel treatment process. Chemical analysis results of cladding hull samples were used as the key criteria to evaluate the effectiveness of the uranium dissolution and noble metal retention. Parametric study indicated that the diffusion of reactants in the porous fuel matrix was the rate-controlling step to the uranium dissolution from the chopped fuel segments. Anode resistance was the most effective parameter to assess the completeness of uranium dissolution and noble metal retention.

## **INTRODUCTION**

Electrorefining of spent nuclear fuels using a molten salt as the electrolyte shows promise for advanced nuclear engineering and spent fuel treatment because of its compactness, economy, radiation resistance, and compatibility with nonproliferation goals [1][2]. Argonne National Laboratory (ANL) has developed and demonstrated a pyrometallurgical process for the Department of Energy (DOE) to treat sodium bonded spent nuclear fuels [3]. This process has been used to treat irradiated fuel elements from the Experimental Breeder Reactor (EBR-II). One of the key steps in the demonstration was electrorefining spent driver fuel in a molten LiCl-KCl-UCl<sub>3</sub>/Cd system using a pilot scale electrorefiner (Mark-IV ER). The Mark-IV ER is located in the Fuel Conditioning Facility at ANL-West site in Idaho.

This article summarizes the experimental observations, results, and engineering aspects regarding the anodic process from electrorefining of 100 irradiated driver fuel assemblies (the demonstration project). The focus is on the anode due to its unique geometry (chopped spent fuel segments loaded in perforated steel baskets or uranium dissolved in a cadmium pool), complex chemical composition, highly demanding process goals and their significance to the entire spent fuel treatment process. The experience and data obtained from the demonstration project are important because the Department of Energy (DOE) has decided to apply this technology to treat the remaining spent EBR-II fuels [4].

## EQUIPMENT AND PROCESS DESCRIPTIONS

The Mark-IV ER and its support equipment are enclosed in a shielded argon hot cell, which is part of the Fuel Conditioning Facility. The major components of the ER are illustrated in Figure 1. The ER vessel is made of stainless steel (2.25 Cr-1 Mo) with an inside diameter of 1.0 m and height of 1.0 m. The vessel contains an approximately 10 cm thick bottom layer of molten cadmium and a 32 cm top layer of molten LiCl-KCl eutectic containing ~ 10wt% of  $UCl_3$ . The ER operating temperature is typically at 500 °C; however, some experiments were conducted at 450 °C. A cadmium stirrer, rotating at 18-22 rpm, is used to continually mix the cadmium. The salt is stirred/mixed during the electrorefining process by the rotating cathode and anode assemblies. Four ports (25.4 cm diameter) on the top of the ER are available for inserting anode and cathode assemblies.

The EBR-II driver fuel is a metallic uranium-zirconium alloy clad in stainless steel. Sodium metal was placed within each fuel element to serve as a thermal bond in the reactor. The spent driver fuel also contains fission products [5]. Prior to electrorefining, the fuel pins were removed from their respective assemblies, chopped into 0.64 cm long segments, and placed into rectangular, perforated, fuel dissolution baskets (FDBs)[6]. Four FDBs were then placed in a cruciform arrangement, loaded onto an electrode assembly, and inserted into the electrolyte of the ER as the anode (Figure 2). Each set of four FDBs contained the segments from two chopped spent fuel assemblies (122 fuel pins). The cathode was a mild steel mandrel, 6.67 cm in diameter, with an active length of ~ 23 cm in the electrolyte. The cadmium pool can be electrically configured as an anode as well.

Table I gives the major compositions of a typical anode load consisting of two chopped driver fuel assemblies. The fission product elements and impurities whose weights are less than 0.001 wt% are disregarded in the table. The electromotive forces (e.m.f.) for some major fuel components (metal elements and their most stable ions) in LiCl-KCl eutectic at 450°C are also listed in the table [7][8]. The metal and metal ion pairs, whose e.m.f. in the molten salt are more active than that of uranium and its trivalent ion, are defined as active metals in this study. Similarly, the metal and metal ion pairs, whose e.m.f. are more noble than that of the uranium and its trivalent ion, are defined as noble metals. It should be noted in Table I that zirconium, a major constituent of the fuel, is the most active among the noble metals.

The purpose of the electrorefining is to separate uranium from the other fuel components. Once the FDBs loaded with chopped fuel segments are inserted in the molten salt electrolyte, the bond sodium and active metal fission products chemically react and displace  $UCl_3$  from the molten salt. Uranium in the fuel segment is electrochemically dissolved from the FDBs and deposits onto the cathode. Zirconium and noble metal fission products are ideally retained in the cladding hulls. The overall process has been described in detail elsewhere [2].

The cladding hulls together with the undissolved fuel components are removed from the ER after electrorefining and become the majority component of the downstream metal waste form. The completion of uranium dissolution and noble metal retention affects not only the effectiveness of the electrorefining but the characteristics of the metal waste as

Table I. Reprehensive Spent Driver Fuel composition and e.m.f. [7]

Element	Weight, g	Wt%	Metal Ion	E <sup>o</sup> <sub>m</sub> (Ag) 450°C
U	8070.00	80.596	U(III)	-1.496
Zr	1080.00	10.805	Zr(IV)	-1.088
Na	216.00	2.160	Na(I)	-2.500
Mo	77.20	0.771	Mo(III)	0.119
Nd	93.10	0.930	Nd(III)	-2.093
Cs	77.30	0.773		
Ce	54.20	0.542	Ce(III)	-2.183
*Pu[8]	41.40	0.413	Pu(III)	-1.813
Ru	40.70	0.407	Ru(III)	0.615
Ba	33.00	0.330		
Si	28.80	0.288		
La	28.40	0.284	La(III)	-2.126
Pr	26.90	0.269		
Sr	21.70	0.217		
Tc	18.20	0.182		
Sm	17.70	0.177		
Y	12.60	0.126	Y(III)	-2.109
Te	11.20	0.112		
Rh	11.10	0.111	Rh(III)	0.526
Rb	9.45	0.094		
Pd	9.00	0.090	Pd(II)	0.513
Ti	7.73	0.077	Ti(II)	-1.010
I	4.82	0.048	I(I)	0.473
Np	4.06	0.041	Np(III)	-1.311
C	3.39	0.034		
Se	1.93	0.019		
Sn	1.50	0.015	Sn(II)	-0.355
O	1.38	0.014		
Pm	1.06	0.011		
Eu	1.06	0.011		
Cd	0.69	0.007	Cd(II)	-0.589
Br	0.65	0.007		
As	0.47	0.005		
Gd	0.45	0.005		
Ag	0.44	0.004	Ag(I)	0.000
Sb	0.35	0.004	Sb(III)	0.087
V	0.33	0.003	V(II)	-0.806
Cu	0.31	0.003		
Nb	0.16	0.002		
Total	10006.00	99.995		

well. Therefore maximizing the uranium dissolution and noble metal retention are major process goals for the anodic process during electrorefining of spent driver fuel. However, since the zirconium is the second largest component in the spent driver fuel (Table I) and the next easiest element to oxidize after uranium, attempting to maximizing uranium dissolution and zirconium retention is somewhat at cross-purposes and results in substantial challenges.

## **EXPERIMENTAL OBSERVATIONS, RESULTS, AND DISCUSSION**

### 3.1. Correlation of Uranium Dissolution with Zirconium Retention

The ER was operated under a controlled current mode. The cell voltage, current, anode voltage, and cathode voltage were recorded by a Data Requisition System during each electrorefining experiment. The anode and cathode voltages were measured versus an Ag/AgCl reference electrode. The open circuit potential is assumed to be zero in this article for simplicity.

It has been observed during the demonstration project that the anodic process predominantly controlled the overall electrorefining rate. Typical current and anodic voltage traces are shown in Figure. 3. It can be seen in Figure. 3 that the anodic voltage gradually increased as the uranium was electrochemically dissolved from the anode baskets. In order to retain other fuel components, the current had to be gradually reduced while the anode baskets were depleting.

Table II lists the representative experimental results of uranium/zirconium retention at different anodic voltages. The data shown in the table are the weight percents of elements retained within the FDBs after the electrorefining experiments. The percentages of uranium and zirconium retained were determined by the ratio of uranium or zirconium left in the cladding hulls in relation to their original quantities in the feed. The quantity of uranium or zirconium left in the cladding hulls was determined through special sampling and analytical procedures. Poor representations of the cladding samples were expected due to the difficulties in remotely “chipping out” representative samples.

The data given in Table II indicate that significant uranium dissolution could not be achieved without co-dissolving a certain amount of zirconium. The percent of zirconium co-dissolved was directly related with the maximum anodic voltage. Even when the anodic voltage was set below 0.4 V, which was the standard voltage difference between uranium and zirconium under the experimental conditions, poor results, 94 wt% uranium dissolution and 73 wt% zirconium retention, were obtained. It has been proven that the presence of  $UZr_2$  alloy in the fuel matrix was the predominant factor that contributed to the co-dissolution of zirconium with the uranium during these experiments. The details of the investigation have been reported in separate papers [6][9].

Table II. Retention of Uranium and Noble Metals after Electrorefining at Different Anode Voltages in Molten LiCl-KCl

Trial No.	Vanode, max	Vanode, avg	W%U	W% Zr	W% Mo	W% Ru	W% Ru106	W% Sb125
1	0.86	0.42	0.3	1.0	17.8	13.3	18.1	22.8
2	0.82	0.55	0.1	7.4	58.3	50.7	51.3	45.5
3	0.60	0.44	0.3	11.6	69.5	52.4	43.4	30.0
4	0.42	0.22	1.9	48.7	89.3	82.9	92.9	69.7
5	0.39	0.21	3.9	73.2	96.4	56.3	97.6	76.8
6	0.38	0.19	5.9	72.6	87.0	84.7	89.3	66.4
7	0.33	0.18	7.5	85.2	84.7	84.8	94.9	72.8

### 3.2. Correlation of Zr Retention with Noble Metal Fission Product Retention

In addition to zirconium retention, another important goal for the anodic process is to retain all the noble metal fission products within the fuel segments. This simplifies the downstream waste processes and prevents cathode products from being contaminated by the noble metal fission products. The dissolution results of noble metal fission products are also given in Table II. Due to the impossibility of analyzing each of the noble metal elements in the cladding, representative elements and isotopes such as Mo, total Ru, Ru-106, and Sb-125 were chosen to estimate the retention of noble metal fission products. The algorithm used to determine the percentage of noble metal retained was similar to those used for uranium and zirconium dissolution. The Ru-106 and Sb-125 levels in the feed have been decayed to the date of the  $\gamma$ -scan used to determine the isotopes left in the cladding hulls.

Table II indicates that the retention of noble metal fission products follow the same trend as that of zirconium retention. When majority of zirconium was retained, e.g. over 50 percent, the noble metal fission products were well retained. When the majority of zirconium was dissolved, the retention of noble metal fission products was poor.

It can be seen from the Table I that the standard voltage difference between uranium and noble metal fission products such as Sb, Mo, and Ru are 1.58 V, 1.59 V, and 2.11 V, respectively. Thermodynamically, these noble metals could not be oxidized within the applied voltage range. Although the noble metals may physically escape from the fuel segments due to agitation and small particle size, 10  $\mu\text{m}$  [9], the physical loss should not correlate with the percentage of zirconium dissolved because zirconium dissolution was an electrochemical process.

The spent driver fuel contains ~ 11 wt% of zirconium metal in the fuel segments (Table I), which is equivalent to ~ 26 volume %. Therefore, when the ER was being operated at high anodic voltages, more zirconium dissolved with the uranium. The matrix of residuals remaining in the fuel segments was fractured upon the loss of zirconium, which caused more ruthenium and other noble metal fission products to escape from the fuel segments. When ER was being operated at low anodic voltages, the residual matrix fractured less, and more noble metal fission products could be retained.

### 3.3 Anode Resistance and Its Impact on the Electrorefining Rate

It has been reported that the redox reaction of  $U^{3+}/U^0$  in molten LiCl-KCl is reversible [10][11]. Therefore, the electrochemical process at the anode in the ER is likely limited by the mass transfer of the reactants. Dissolving uranium metal exclusively from a zirconium-rich matrix would render the matrix porous. The uranium interior to the matrix would become less and less accessible to the electrolyte as more uranium is electrochemically dissolved from it. As a consequence, the anodic resistance increases. The current must be reduced in order to compensate for the increasing resistance within the voltage limitation. Thus, the electrorefining rate becomes limited.

There were at least two mass transfer processes occurring at the anode, which could limit the overall uranium electrorefining rate. One such process was the diffusion of reactants through the metal fuel matrix inside the fuel segments. It is referred to as “internal diffusion” or the mass transfer process inside the fuel segments in this article. The other process was the diffusion of the reactants from the fuel segment surface to the bulk solution, which included the diffusion of the reactants through the basket. It is referred to as “external diffusion” or the mass transfer process outside the fuel segments. To identify the significance of the two diffusion processes, the following experiments were conducted and the results are given in Figure 4.

Two electrorefining experiments were performed with the length of the fuel segments set at 0.64 cm. The ER was operated at otherwise the same conditions but the FDBs were rotating at 25 rpm and 75 rpm, respectively. The two nearly overlapped curves in Figure 4 represent the anodic resistances recorded for the two experiments. If the “external diffusion” were the rate-limiting step, increasing in the rotating speed of the FDBs would improve the mass transfer process. As a result, the anodic resistance was expected to decrease. The two almost identical curves of the anodic resistance shown in Figure 4 indicate that the anodic process was not limited by the mass transfer outside the fuel segments.

The third curve in Figure 4 represents the anodic resistance when the FDBs were rotating at 25 rpm but with the two FDBs were filled with chopped fuel segments of 0.64 cm in length and the other two loaded with segments of 1.9 cm in length. Although this experiment ceased at ~ 2445 ampere-hours due to a mechanical failure, its average anodic resistance was 25% higher than the previous two during the 2445 ampere-hours. These experimental results have demonstrated that the diffusion of electrochemical reactants inside the fuel segments or the “internal diffusion” was the rate-limiting step for the electrochemical dissolution of spent driver fuel.

### 3.4. Impact of Periodic Interrupting Current on the Anodic Process

Periodically changing current or voltage techniques have been utilized in the electroplating industry to improve the resulting quality of deposits, especially for mass transfer controlled electrochemical processes. [12-15]. Reports regarding using the periodically changing current technique to improve the anodic performance are rare. Three experiments were carried out at the Mk-IV ER in which a periodic interrupted current was applied to the

anode. The experiments were performed in an attempt to improve the performance of both the anode and cathode. Only the results of the anode process are presented in the article.

Table III. Experimental Conditions of Periodic Interrupting Current

Anode	Cathode	Current Duration
FDBs	Steel Mandrel	6 seconds
Cadmium Pool	Steel Mandrel	2 Seconds

The experimental conditions and polarities are given in Table III. The interrupting current was generated by the following means: an external current was applied from the FDBs (anode) to a steel mandrel cathode for 6 seconds, then the FDBs were electrically disconnected. The cadmium pool was connected as the anode for 2 seconds. After this cycle, the current was switched to the FDBs again for 6 seconds, and so on. Given such a configuration, the actual current pattern that passed through the FDBs was serial square waves of 6 seconds on, 2 seconds off. That is, the diffusion process inside the fuel segments had a 2-second relaxation after each 6-second electrochemical dissolution. It was observed that the average anodic resistance of the tests was consistently lower in comparison to those conducted with continuous currents. The anodic dissolution results from the interrupting current experiments are shown in Table IV.

It is interesting to note from Table IV that the major effect of the periodically interrupted current on the anode was an improvement of zirconium and noble metal retention. For all three experiments, 100% of the zirconium and almost all of the noble metal fission products were retained with average uranium dissolution of 93.7%. The interrupting current experiments have proven that the internal diffusion control model for the anodic process was valid. The uranium presented in the form of  $UZr_2$  was difficult to dissolve. In fact, the amount of undissolved uranium corresponded with all the uranium in the  $UZr_2$  phase. The disadvantage of the periodic interrupting current technique was that it prolonged the electrochemical dissolution time.

Table IV. Dissolution Results of Periodic Interrupting Current Experiments

Trial No.	W% U	W% Zr	W% Mo	W% Ru	W% Ru106	W% Sb125
1	5.9	100.0	95.6	88.4	88.9	64.0
2	6.2	100.0	99.9	85.6	93.2	72.2
3	6.9	100.0	85.8	85.8	100.0	79.6

### 3.5. Electrochemical Dissolution of Spent Driver Fuel under Fixed Operating Conditions

Based upon the experimental results presented above, it was decided that the anodic process must achieve >96 wt% uranium dissolution even at the expense of some noble metal retention in order to meet the established goals. Apparently, the most crucial operating parameter with regard to uranium dissolution and noble retention was the anode cut-off

voltage or the anodic resistance. The other parameters, such as the anode rpm, were of minimal influence. Therefore, the fixed operating conditions for the anodic process were determined; maximizing the cell current while keeping the anodic voltage below 0.4 V until the anodic resistance reached 30 m-ohm. Table V summarizes the electrochemical dissolution results after processing 22 batches of spent driver fuel under the fixed operating conditions. An average uranium dissolution of > 96 wt% was achieved while retaining over 80 wt% of zirconium and majority noble metal fission products. The results of two representative active metal fission products, cerium and neodymium, are also summarized in the table and are discussed within the next section.

### 3.6. Dissolution Behaviors of Active Metal Fission Products

As mentioned before, the active metal fission products will chemically dissolve into the molten salt electrolyte prior to electrorefining because they are thermodynamically more active than the uranium in the system. But the results given in Table V reveal that the active metal fission products, such as Ce and Nd, were not completely dissolved even after >96% uranium had been electrochemically dissolved. Figure 5 plots the percentage of U, Ce, and Nd retained with the cladding hulls for the 22 experiments under the fixed operating conditions. It is very interesting to note that the percent of Ce or Nd retained follows the same pattern as the percent of uranium retained. The amount of Ce and Nd, which remained within the cladding hulls, were probably not a result of adhering electrolyte because the concentrations of Ce and Nd in the electrolyte were increased as more batches of the spent fuel were processed. The correct mechanism for the co-retention of Ce or Nd with uranium is currently under study.

## CONCLUSIONS

For the electrorefining process of spent drive fuel in a molten  $\text{LiCl-KCl-UCl}_2$  system, significant uranium dissolution could not be achieved without co-dissolution of some zirconium.

The mass transfer of electrochemical reactants inside the fuel matrix, or the mass transfer resistance, was the rate-limiting step for the overall electrorefining process.

Anode resistance was the most effective parameter to assess the completeness of uranium dissolution and noble metal retention.

The interrupting current technique could improve the effectiveness of zirconium and noble metal retention at the anode but prolonged electrochemical dissolution time.

Table IV. Electrochemical Dissolution Results of Spent Driver Fuel under Fixed Operating Conditions

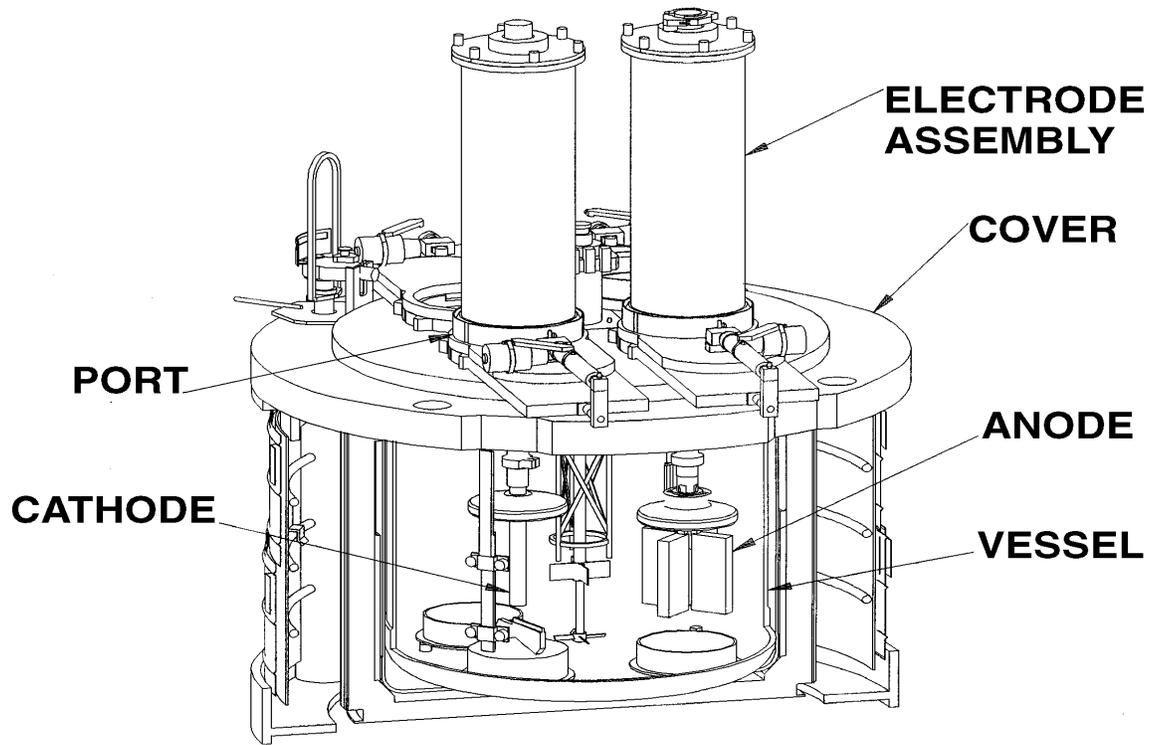
Trail No.	U	Zr	Mo	Total Ru	Ru-106	Sb-125	Total Ce	Total Nd
1	4.3	64.0	88.9	78.2	65.9	52.3	3.9	2.0
2	3.9	64.4	85.8	79.4	67.7	50.6	3.1	1.7
3	3.5	56.3	76.7	70.3	58.5	44.6	3.5	2.7
4	4.1	61.8	81.0	70.0	64.5	51.1	4.4	2.5
5	3.9	57.8	89.0	79.1	78.8	54.6	3.0	2.1
6	3.4	50.6	78.8	67.4	73.8	53.7	2.8	1.9
7	5.0	81.3	94.3	86.4	92.5	73.6	3.6	2.5
8	5.8	89.5	100.0	96.5	95.5	79.1	4.7	3.7
9	4.7	88.4	92.7	86.8	70.6	53.9	4.4	2.6
10	4.7	90.7	93.7	89.4	78.5	60.2	4.4	2.1
11	4.1	93.6	92.9	86.9	76.1	65.3	3.5	1.9
12	3.1	98.6	92.9	87.1	75.3	63.0	2.5	0.8
13	3.5	80.3	91.2	86.5	77.5	62.0	3.3	1.6
14	4.0	83.5	100.5	95.1	98.9	64.5	3.6	1.9
15	4.5	82.0	87.7	85.7	93.7	73.9	3.2	1.9
16	3.1	95.2	78.6	73.7	100.0	66.0	2.1	0.7
17	4.7	93.2	97.7	92.2	87.3	66.8	3.9	2.3
18	4.1	96.5	98.0	92.1	69.0	61.4	3.9	2.5
19	4.6	83.5	102.1	89.5	102.0	78.3	2.0	1.3
20	5.2	86.6	99.5	92.0	98.6	72.0	3.6	2.5
21	4.5	85.8	105.7	100.3	95.5	72.8	5.7	2.6
22	4.3	83.1	101.1	94.7	89.5	67.7	4.4	2.9
<b>Average</b>	4.2	80.3	92.2	85.4	82.3	63.0	3.6	2.1
<b>STDV</b>	0.7	14.1	8.1	8.9	13.1	9.4	0.8	0.7

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16.

Figure 1. Schematic of Mk-IV electrorefiner



Figure 2. Fuel dissolution baskets (FDBs) in their handling tray.

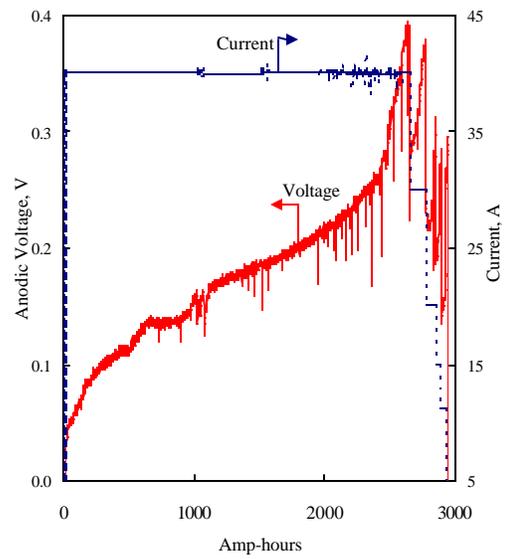


Figure 3. Current and Anode voltage traces.

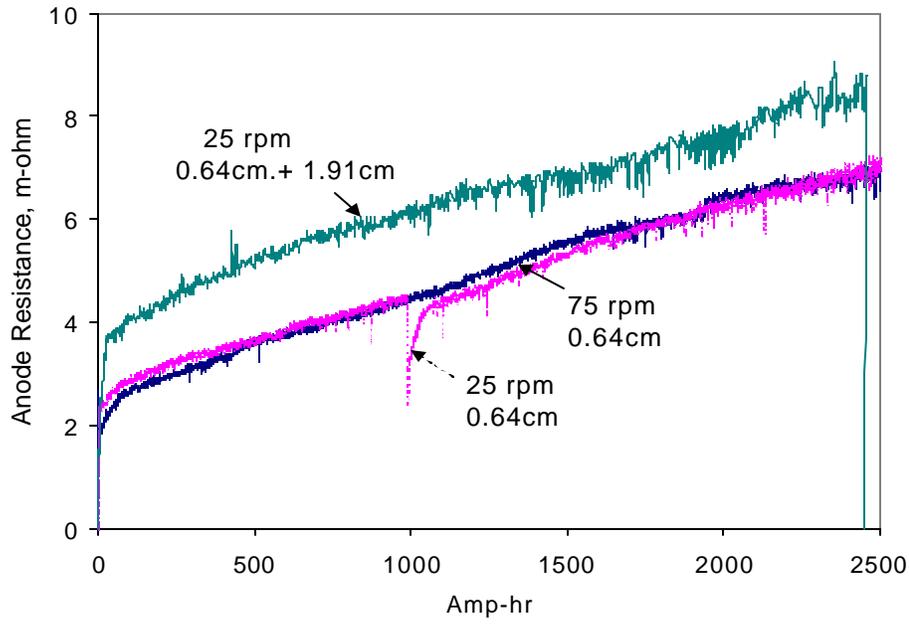


Figure 4. Anodic resistance at different rpm and chopping length.

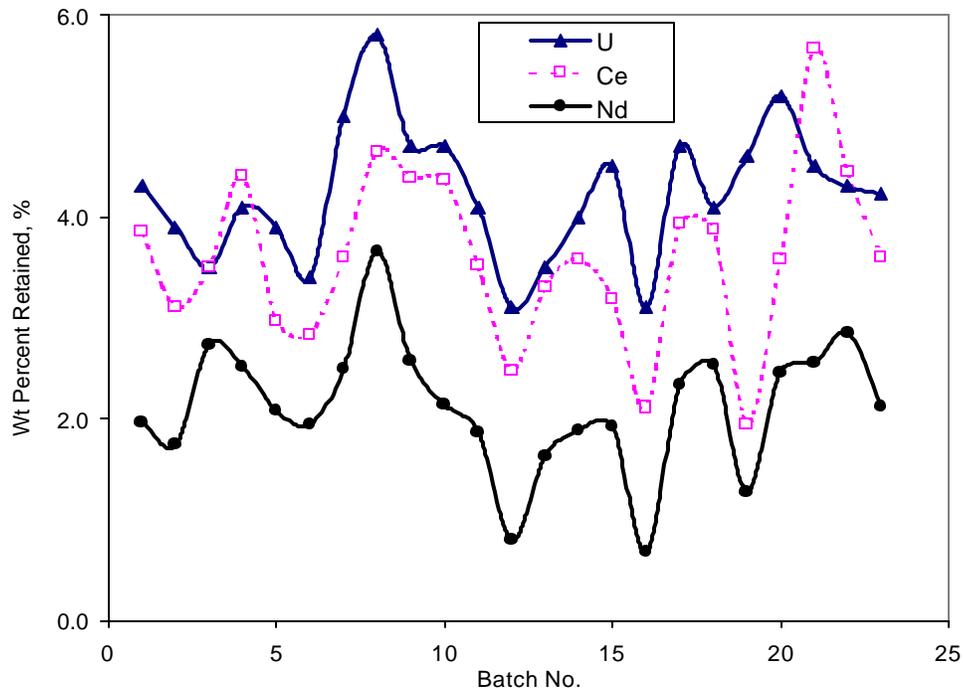


Figure 5. Percentage of U, Ce, and Nd retained with claddings after electrorefining under fixed operating conditions.