RUTHENIUM REMOVAL FROM INTERMEDIATE LEVEL RADIOACTIVE
WASTES BY ELECTRODIALYSIS METHODS

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TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	<u>1</u>
SUMMARY	<u>2</u>
ELECTRODIALYSIS PROCESSES	<u>2</u>
EQUIPMENT AND PROCEDURES	<u>3</u>
Radioactive Wastes Investigated	<u>3</u>
Electrodialysis Units	<u>4</u>
Operational Equipment and Procedures	<u>7</u>
Analytical Methods	<u>7</u>
RESULTS AND DISCUSSION	<u>7</u>
Ruthenium Removal by Electrodialysis	<u>7</u>
Ruthenium Removal by Packed Bed Electrodeionization	<u>13</u>
Acid Condensate Treatment	<u>13</u>
Alkaline Condensate Treatment	<u>21</u>
CONCLUSION	<u>23</u>
REFERENCES	<u>24-25</u>

RUTHENIUM REMOVAL FROM INTERMEDIATE LEVEL RADIOACTIVE WASTES BY ELECTRODIALYSIS METHODS

L. F. Coleman and J. R. Eliason

INTRODUCTION

Ruthenium-106, due to its appreciable fission yield, one year half-life, and complex solution behavior is present in intermediate level wastes (e.g., condensates) from nuclear fuel reprocessing plants. It exists in these wastes in a number of chemical forms dependent on origin, solution composition, temperature and age. The presence of cationic, anionic and uncharged ruthenium in process condensates poses special waste treatment problems. The processes which have been developed to remove the other major, radiologically-significant radio-nuclides lack the capability to remove ruthenium effectively. Re-evaporation is an effective means for producing a condensate adequately decontaminated with respect to ruthenium in many cases, but there remains an economic incentive for finding more efficient treatment methods.

A scouting study of methods for removing ruthenium from intermediate level condensates showed electrodialysis processes to have possible economic advantages over re-evaporation. Walters⁽¹⁾, Burns⁽²⁾, Ito⁽³⁾ and Krawczynski⁽⁴⁾ have discussed the economics of such treatment methods for radioactive wastes. In contrast to evaporation, electrodialysis processes embody the concept that for certain electrolyte concentrations it is more economical to remove the dissolved electrolyte from the system than to remove the bulk liquid from the acids, bases, or salts.

This report presents information on ruthenium removal from actual process wastes by electrodialysis methods.

SUMMARY

Electrodialysis was demonstrated to be capable of removing ruthenium from nitric acid wastes, but the economics for removing greater than 90 percent of the ruthenium activity by only an electrodialysis step are not considered favorable.

Electrodeionization through packed resin beds (a special application of electrodialysis) was shown to be a feasible and economical method for removing ruthenium from acid and alkaline process condensates containing on the order of 500 ppm dissolved solids. Laboratory results show that 95 percent of the ruthenium can be removed from acidic and alkaline condensates at up to fiftyfold volume reductions.

ELECTRODIALYSIS PROCESSES

Electrodialysis refers to ion transport through permeable membranes under the influence of an applied electric field. Historically, the useful application of electrodialysis was investigated for deionizing water by Billiter⁽⁵⁾ in 1931, and the early theory is traced to Teorell⁽⁶⁾ and Sievers and Meyers⁽⁷⁾ in 1935-36. The main technological breakthrough came with the commercial availability of permselective membranes in the early 1950's⁽⁸⁻⁹⁾. A general description of electrodialysis processes, process variables, and operating conditions can be found in a number of books, reports and reviews⁽¹⁰⁻¹⁸⁾.

The electrodialysis process is economically competitive with ion exchange or evaporation processes for solutions containing about 500 to 30,000 ppm dissolved solids in terms of water reclamation. For radioactive waste treatment, the economics are favorable in the 500 to 10,000 ppm concentration range and particularly so in the 1,000 to 3,000 ppm range⁽³⁾. At low electrolyte concentrations (less than about 500 ppm) electrodialysis operating efficiency falls rapidly due to high

solution resistances and membrane polarization. Strongly ionized resins, when placed in the electrodialysis compartments, can overcome this increased resistance problem by serving as an ion conduction medium. This technique is herein referred to as "electrodeionization." Membrane polarization, in this case, leads to the electrolytic dissociation of water whereby the hydrogen and hydroxyl ions produced serve to regenerate the resin beds. A discussion of packed bed electrodeionization is given in articles by Sammons⁽¹⁹⁾, Gittens⁽²⁰⁾, and Glueckauf⁽²¹⁾.

EQUIPMENT AND PROCEDURES

Radioactive Wastes Investigated

Acid and alkaline intermediate level condensates were used in the investigation. The alkaline condensate originates from the boiling high level waste stored at Hanford⁽²²⁾. The acid condensate originates as the overheads from the acid fractionator system of the Purex acid recovery process. Concentrations of the primary radionuclides and electrolytes are given in Table I.

TABLE I

Composition of Process Condensates

Radionuclides (Concentrations in $\mu\text{Ci/ml}$)

<u>Waste</u>	<u>CePr¹⁴⁴</u>	<u>Sr⁹⁰</u>	<u>Ru¹⁰⁶</u>	<u>Cs¹³⁷</u>	<u>ZrNb⁹⁵</u>	<u>Nonradioactive constituents</u>
Acid condensate	10^{-3}	10^{-7}	10^{-2}	10^{-4}	10^{-3}	$1-3 \times 10^{-2} \text{M HNO}_3$
Alkaline condensate	10^{-3}	10^{-4}	10^{-3}	10^{-2}	10^{-3}	$2-12 \times 10^{-3} \text{M NH}_3$ 30-200 ppm butyl phosphates.

Electrodialysis Units

An Ionics^{*} design laboratory-scale electrodialysis unit having 20 concentration-dilution cell pairs was the largest unit used. This unit had 220 cm² of effective membrane transfer area per cell pair and a rated capacity of 50 percent salt removal per pass for 1630 ppm sodium chloride solution at 50 U.S. gallons per hour. A number of three-cell electrodialysis units, made of plastic, were used. These units had 36 cm² of membrane transfer area for the normal electrodialysis experiments and 7 cm² for the packed bed electrodeionization experiments. Dilution channel widths were 0.63 and 1.9 cm while anolyte and catholyte compartment widths were 1.9 cm. Resin packed compartments contained 13.5 cm³ of mixed bed exchangers (60/40 volume mixture of Dowex-1 and 50W) for 1.9 cm width compartments and 4.5 cm³ for 0.63 cm width compartments. Ionics type CR 61 and AR 111 permselective membranes were used in all cases.

Figure 1 shows a simplified electrodialysis unit operated in a concentration-dilution mode as would apply to radioactive waste decontamination. Processes other than counterion transport occur in actual electrodialysis operation; coion transport, back diffusion, osmosis, hydrogen and hydroxyl ion transport. The extent to which these unfavorable processes occur is dependent on cell design, membrane properties and operating conditions. Figure 2 illustrates a packed bed electrodeionization process and the means of regeneration. Electrodeionization depends on polarization sufficient to electrolytically dissociate water to effect regeneration of membranes and resins, whereas polarization is an unwanted effect for normal electrodialysis.

^{*}Trademark Ionics Incorporated, Cambridge, Massachusetts.

A = Anion Permselective Membrane
 C = Cation Permselective Membrane
 Dilution + Concentration
 Compartment = Cell Pair

(a) Typical Concentration - Dilution Electrodialysis Unit
 (b) Processes Which can Occur During Electrodialysis

FIGURE 1

FIGURE 2

Simplified Illustration of Packed Bed Electrodeionization

Operational Equipment and Procedures

Centrifugal pumps, 1/8th hp with all wetted parts of stainless steel, were used in conjunction with the Ionics unit. Flows through the dilution, concentration and electrode compartments were metered, and pressures were measured on inlet and outlet sides of the unit. Effluents from dilution and concentration streams were cooled and passed through in-line electrical conductivity cells and through a beta monitor cell.

The plastic electrodialysis cells were fed by gravity or peristaltic pumps. Solution effluents from the three-cell units were collected in fraction collectors and volumes and flow rates obtained. Continuous operation experiments were performed, either by varying the relative flow rates through the concentration-dilution cells to give a net volume reduction or by recirculation of a single batch of concentrated waste. Batch experiments were also performed in which the dilution effluent was continually recirculated until the desired degree of de-ionization was obtained.

Analytical Methods

Radiochemical concentrations were determined by gamma energy analysis of spectra obtained with a 400 channel analyzer. Ruthenium was extracted by the method of Kiba, et al.⁽²³⁾, which involves oxidation in alkaline hypochlorite followed by pyridine extraction.

RESULTS AND DISCUSSION

Ruthenium Removal by Electrodialysis

An Ionics design electrodialysis unit was used to evaluate bulk electrolyte removal from nitric acid solutions. The proposed flow diagram for treatment

was analogous to that of Ito⁽³⁾ and is illustrated in Figure 3. Data for the first electrodialysis stage are given in Table II. The system performs well and operating difficulties are not expected for normal electrodialysis treatment. Electrodialysis power cost of 11¢/1,000 gallons for a volume reduction of 10 is higher than suggested by Ito⁽³⁾ and could be improved by scale up, counter current flow of concentration and dilution streams and overall optimization of variables.

TABLE II

Electrodialysis of 0.024 N Nitric Acid*

<u>Flow Rate</u> <u>(ml/min)</u>	<u>Volume Reduction</u>	<u>Current Density</u> <u>(ma/cm²)</u>	<u>% HNO₃</u> <u>removed</u>	<u>Power Use</u> <u>Kwh/1000 gal.</u>
500	10	11	89	11
1000	10	13	84	7
1500	10	18	81	6
2000	10	9	57	1
2000	10	13	60	2
2000	10	18	68	4
2000	10	23	74	8

* 20 concentration-dilution cell pairs - 220 cm² membrane/cell pair.

Electrodialysis of process condensate was initiated with three cell units; the flow pattern for the batch operation used is shown in Figure 4. Ruthenium removal relative to nitrate removal was observed to be slow as shown in Table III.

TABLE III

Relative Nitrate-Ruthenium Decontamination Factors*

<u>NO₃⁻ DF</u>	<u>Ru DF</u>
10	2.2 - 4.2
20	2.9 - 4.8
50	3.7 - 5.6
100	5.0 - 6.5
1000	11.5 - 15.0

* 1-3 day aged nitric acid condensate - 0.01 - 0.02N HNO₃

FIGURE 3

Two Stage Electrodialysis Followed by Ion Exchange

FIGURE 4

Flow Pattern for Batch Electrodesorption Operation

Scale up to a seven-cell unit and to a 20-concentration dilution cell pair unit gave similar results (Table IV).

TABLE IV

Electrodialysis Cell Comparison

<u>Seven Cell Unit</u>		<u>Twenty Cell Pair Unit</u>	
110 cm ² effective membrane		4400 cm ² effective membrane	
<u>% In solution</u>		<u>% In solution</u>	
<u>Nitrate</u>	<u>Ruthenium</u>	<u>Nitrate</u>	<u>Ruthenium</u>
70	87	70	88
50	77	50	78
30	63	30	66
10	42	10	44

Fractional removal of ruthenium versus time was obtained by batch treatment at constant operating voltage. Measurements of ruthenium removal were based on ruthenium depletion of the feed solution rather than on amounts transferred to the anolyte and catholyte compartments to avoid membrane equilibration effects. Typical results, Figure 5, show two distinct removal rates. The initial fast removal step is believed to be one of ion transport and would include the removal of highly ionized cationic and anionic ruthenium forms present. The slower rate, which appears to be first order, could include a number of removal mechanisms.

It was observed that the ionic fraction of the ruthenium in the fresh waste changed from anionic to cationic on aging. One explanation for the observed changes would be the presence of highly nitrated, anionic nitrate-nitrosyl ruthenium species in the fresh waste which could hydrolyze in the dilute acid system to form cationic and neutral complexes. The slow removal process had an associated half-life of 60-80 minutes and the change in its rate with aging points to more than a singular first order removal mechanism.

FIGURE 5

Batch Electrodialysis of Acid Condensate at Constant Voltage

Sorption experiments following partial deionization showed improved removal over that of the raw waste. Sorbent removal rates were slow and again the mechanisms may involve chemical and physical adsorption as well as dissociation or re-equilibration of the various possible ruthenium forms. Nitrate based resins removed less ruthenium than hydroxide based resins which reflects a poor ruthenium selectivity over that of nitrate or solution equilibria involving nitrate ion. Nitric acid addition to previously partially deionized nitric acid process solutions gave similar sorption results to that of the raw waste. This suggests, that for this acid condensate, poor ruthenium removal on ion exchange media is largely a function of unfavorable equilibria and slow reaction rates, rather than the presence of completely insoluble, unremovable, unionized ruthenium compounds.

Ruthenium removal on a mixed bed resin column following electrodialysis is shown in Figure 6. The overall decontamination factor of the electrodialysis plus ion exchange treatment for fresh acid condensate was 11. This process, therefore, has limited utility for intermediate level waste treatment where ruthenium decontamination factors of 20 to greater than 100 are often required.

Ruthenium Removal by Packed Bed Electrodeionization

Acid Condensate Treatment

A tentative flow sheet for packed bed electrodeionization of radioactive condensates is shown in Figure 7. The primary features of such a process are a high degree of ruthenium and electrolyte removal at practical volume reductions. Ninety-five percent ruthenium removal at fiftyfold volume reductions was readily obtained for acid and alkaline condensates investigated.

FIGURE 6

Mixed Bed Ion Exchange Performance

ARCEE RICHARD, WASH.

FIGURE 7

Packed Bed Electrodeionization Flowsheet

Ruthenium breakthrough results from an electrodeionization experiment are shown in Figure 8. Also shown is the ruthenium breakthrough curve obtained from an equal volume, mixed bed ion exchange experiment with the same condensate. Ruthenium breakthrough approaches 70 percent by ion exchange only at a flow rate of 2 ml/min (0.2 bed volumes/min). Electrodeionization showed signs of greater than 10 percent breakthrough at flows of 9, 7, and 2.7 ml/min. At a flow of 0.7 ml/min (33 percent current efficiency) ruthenium breakthrough dropped to less than one percent. When the flow was increased to 6 ml/min the system rapidly approached the ion exchange column breakthrough level. Power was also terminated to allow the system to assume the behavior of the ion exchange column. A return of applied potential brought the electrodeionization system to one percent breakthrough at 80 percent current efficiency, while another power off-on cycle showed the same behavior. Subsequent experiments up to 2600 bed volumes throughput showed 98 percent ruthenium removal was obtainable to this point at 50 percent current efficiency.

Mechanisms for the observed ruthenium removal were not established. Some of the probable mechanisms are: More favorable ion exchange equilibria, electrolytic dissociation of mononuclear and polynuclear complexes and induced chemical and physical adsorption.

One of the problems in interpreting the results is the question of ruthenium equilibria within the overall system since it was observed that 40-50 percent of the ruthenium continually loaded on the dilution compartment membranes and resins and that solution age was also a variable. Further investigation of the equilibrium ruthenium saturation problem showed that greater than 99 percent ruthenium

FIGURE 8

Comparison of Packed Bed Electrodeionization and Mixed Bed Ion Exchange Ruthenium Decontamination

removal could be obtained after 10,000 bed volumes of aged (90-day) acid condensate had been processed. Aged condensate was purposely used to minimize variations in the ruthenium composition of the feed.

Figure 9 shows the results of an experiment where 8,000 bed volumes of 0.02N acid condensate were passed through the unit to saturate the system. Up to the 8,000 bed volume throughput point, the unit had been operated at an average flow rate of 1.2 ml/min and 7 ma/cm², at which conditions 28 percent breakthrough occurred. At flow rates of 0.1 and 0.5 ml/min and the same current density, ruthenium removal increased to 99.9 and 80 percent, respectively, and at a flow rate of 0.5 ml/min and a current density of 10 ma/cm² ruthenium removal was 99 percent. After 8 days' operations at the latter conditions, ruthenium removal decreased to 90 percent with an accompanying hydrogen ion concentration increase in the effluent. A severe external leak necessitated termination of the experiment, and it is assumed that the observed breakthrough at this point was due to mechanical failure rather than a ruthenium saturation effect.

Mixed bed ion exchange treatment of the aged condensate referred to in Figure 9, at a flow rate of 0.5 ml/min through a replicated packed resin bed, reached ruthenium breakthroughs of 38 and 50 percent after 10 and 42 bed volumes throughput, respectively.

For the experiment illustrated in Figure 9, analyses of the anolyte and catholyte streams showed 40-50 percent of the feed ruthenium continually loaded on the dilution compartment resins and membranes. The ruthenium which transferred through the membranes was 70 percent cationic and 30 percent anionic. This is of interest in comparison to the sorbent data of Table V, which shows

FIGURE 9

Packed Bed Electrodeionization of Aged Acid Condensate

less than one percent of the original ruthenium activity to be anionic for the aged condensate.

TABLE V

Sorption Data for Acid Process Condensate

Conditions: 200 x 400 mesh resins
1 gram resin/100 ml solution
1 hour equilibration at 22°C

<u>Sample</u>	<u>% Ruthenium removed</u>	
	Anion Resin	Cation Resin
Feed to electrodeionization Unit 0.024 <u>N</u> HNO ₃	< 1	59
Effluent after 10,000 bed volumes of waste processed, 1% ruthenium breakthrough	29	29
Effluent after 11,000 bed volumes of waste processed, 10% ruthenium breakthrough	21	33

The observed transfer of approximately 17 percent of the feed ruthenium to the anolyte compartment in the forementioned experiment shows that anionic ruthenium complexes are being formed during electrodeionization. This effect could be explained on the basis of equilibrium changes and electrolytic dissociation of uncharged ruthenium complexes.

Electrodeionization costs for acid condensate treatment are speculative. Based on the data of Figure 3, the cost to obtain 99 percent ruthenium removal would be \$2.60/1,000 gallons for electrodeionization power alone. The economics would be grossly improved with a lower electrolyte concentration, narrower channel widths and a multicompartment unit where electrode power losses are averaged over a large number of cell pairs. It is not unreasonable that the power cost could be reduced to less than \$1/1,000 gallons when processing condensate wastes

with a large scale unit. Power costs for treating 0.01N nitric acid condensate at 2 percent ruthenium breakthrough and a total of 2600 bed volumes throughput were \$0.6/1,000 gallons. At power costs of \$1/1,000 gallons, a membrane replacement cost of about \$10/1,000 gallons could be tolerated and still be economically competitive with evaporation costs of \$18/1,000 gallons⁽²⁴⁾.

Alkaline Condensate Treatment

Electrodeionization of alkaline condensate gave 95 percent ruthenium removal on processing 21,000 bed volumes of waste through the three-cell electrodeionization unit. Zirconium and cerium were removed as effectively as ruthenium, and cesium was at one percent breakthrough after processing 21,000 bed volumes of waste at a volume reduction factor of 47.

Anolyte and catholyte streams were combined and recirculated to give the volume reduction factor and no attempt was made to measure the relative transfer of anionic and cationic species. Distribution of radionuclides accumulated during the processing of the alkaline condensate is given in Table VI.

TABLE VI

Distribution of Radionuclides Within A 3-Cell Electrodeionization System

Conditions: Bed volumes - 4.5 cm³ 60/40 mixture of Dowex 1 and Dowex 50W
 Flow rate - 2 ml/min
 Condensate - pH 10 - 3 x 10⁻³ M NH₃
 Volume reduction - 47
 Throughput - 21,000 bed volumes

Component	Fractional Distribution of Input Activity			
	CePr ¹⁴⁴	Ru ¹⁰³⁻¹⁰⁶	Cs ¹³⁷	ZrNb ⁹⁵
Dilution resin	0.70	0.55	0.15	0.85
Anolyte resin	0.08	0.22	0.05	0.07
Catholyte resin	0.21	0.15	0.32	0.06
Dilution waste	< 0.05	0.05	0.01	< 0.05
Concentrate waste	< 0.09	0.09	0.48	< 0.05

The results in Table VI show that a large fraction of the cerium, zirconium and ruthenium remain within the dilution compartment. Chemical elution of the individual resin filled compartments with sodium hydroxide followed by sulfuric acid readily removed the cesium and cerium; however, zirconium and ruthenium were not removed to a high degree as shown in Table VII. The inability to remove the zirconium and ruthenium completely is not expected to influence subsequent resin loading for this carrier-free system. This was demonstrated in the case of ruthenium where 6,000 bed volumes of waste were processed followed by chemical regeneration and successive treatment of 6,000 bed volumes of waste. At the same operating conditions similar breakthrough results were obtained. It has also been observed that anion resin columns operate successfully following chemical regeneration although ruthenium cannot be completely eluted.

TABLE VII

Elution of Radionuclides From A Three-Cell Electrodeionication Unit*

Eluting Agents: 6 Bed volumes of 6N NaOH 5 Bed volumes of water 6 Bed volumes of 6N H ₂ SO ₄				
Compartment	Fraction Eluted			
	CePr ¹⁴⁴	Ru ¹⁰³⁻¹⁰⁶	Cs ¹³⁷	ZrNb ⁹⁵
Anolyte resin	0.6	0.3	0.99	0.7
Catholyte resin	0.8	0.3	0.99	0.7
Dilute resin	0.9	0.2	0.99	0.6

*After processing 21,000 bed volumes of alkaline condensate.

Electrodeionized effluent at 5 percent ruthenium breakthrough, accumulated for 28 days, was passed through a 200 x 400 mesh cation resin column at a flow rate of 32 bed volumes/minute. A constant ruthenium breakthrough of 50 percent

was maintained to 10,000 bed volumes. Immediate anion column treatment of the cation column effluent at the same flow rate removed 22 percent of the residual ruthenium out to 10,000 bed volumes throughput.

Electrodeionization power costs for alkaline condensate treatment at 95 percent ruthenium removal were \$1/1,000 gallons based on the three-cell unit data. As stated in regard to acid condensate treatment, multicompartment units and narrower compartment widths would improve the overall operating efficiency.

CONCLUSION

The use of packed bed electrodeionization to remove ruthenium from aged acid and alkaline condensates has been demonstrated on a laboratory scale. Adequate decontamination factors and volume reductions have been achieved and competitive economics indicated. Membrane and resin lives have not been evaluated, nor have the ruthenium removal mechanisms been established.

Future development should include: 1) determination of the mechanisms for ruthenium removal; 2) evaluation of lifetimes of membranes and resins under the prescribed operating conditions; 3) evaluation of cyclic operation employing chemical as well as electrochemical regeneration of membranes and resins; 4) cost evaluation for maintaining a fixed level of ruthenium decontamination.

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