MASTER

SERIES ELECTROLYTIC DISSOLVER FOR NUCLEAR FUELS
I. SCOPING STUDIES

M. R. Bomar

PHILLIPS
PETROLEUM
COMPANY

ATOMIC ENERGY DIVISION

NATIONAL REACTOR TESTING STATION
US ATOMIC ENERGY COMMISSION

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

PRICE \$.50

Available from the
Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Printed in USA

IDAHO CHEMICAL PROCESSING PLANT

IDO-14563
AEC Research and Development Report
Chemical Separations Processes for
Plutonium and Uranium
TID-4500, Ed. 16
Issued: November 15, 1961

SERIES ELECTROLYTIC DISSOLVER FOR NUCLEAR FUELS

I. SCOPING STUDIES

M. R. Bomar

Chemistry Section

PHILLIPS
PETROLEUM
COMPANY

Atomic Energy Division

Contract AT(10-1)-205

Idaho Operations Office

U. S. ATOMIC ENERGY COMMISSION

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

SERIES ELECTROLYTIC DISSOLVER FOR NUCLEAR FUELS

I. SCOPING STUDIES

M. R. Bomar

TABLE OF CONTENTS

	Page
ABSTRACT	1
I. SUMMARY	2
II. INTRODUCTION	3
III. EQUIPMENT DESIGN	5
A. Dissolvers	5
B. Power Supplies	7
C. Current Measurement	7
IV. EXPERIMENTS AND RESULTS	8
A. Test Procedure	8
B. Dissolution of Stainless Steel	8
C. Dissolution of Aluminum	9
D. Use of a Simulated Tantalum Container	10
E. Oxidation of Zircaloy-2	10
F. Effect of Alternating Current Voltage	11
V. CONCLUSIONS	12
VI. APPENDIX	14
VII. LITERATURE CITED	20

LIST OF TABLES

		Page
Table 1	SERIES DISSOLUTION OF STAINLESS STEEL USING DIRECT CURRENT PRELIMINARY TESTS	14
Table 2	SERIES DISSOLUTION OF 304 STAINLESS STEEL SEVENTEEN-HOUR DEMONSTRATION RUN	15
Table 3	SERIES DISSOLVER TESTS USING ALTERNATING CURRENT	16
Table 4	SERIES DISSOLUTION OF ALUMINUM TO ACID DEFICIENCY	17
Table 5	EFFECT OF TANTALUM DISSOLVER LINER ON THE SERIES DISSOLUTION OF ALUMINUM	18
Table 6	OXIDATION OF ZIRCALOY-2 IN SERIES DISSOLVER . . .	19

LIST OF FIGURES

		Page
Figure 1	PRINCIPLE OF SERIES ELECTROLYTIC DISSOLVER . . .	4
Figure 2	DISSOLVER DIAGRAMS	6

SERIES ELECTROLYTIC DISSOLVER FOR NUCLEAR FUELS

I. SCOPING STUDIES

M. R. Bomar

A B S T R A C T

A new dissolver design has been demonstrated on a laboratory scale for the electrolytic dissolution of nuclear fuels. Simplified electrodes are used which need not touch the fuel pieces, thus avoiding the usual problems of achieving adequate electrical contact between the fuel and the anode basket. Because a series of electrolytic cells are formed along the length of the dissolver, current efficiency is enormously increased; this technique allows for the use of higher voltage and much lower current than is presently considered essential in plant-scale electrolytic dissolution.

SERIES ELECTROLYTIC DISSOLVER FOR NUCLEAR FUELS

I. SCOPING STUDIES

M. R. Bomar

I. SUMMARY

The electrolytic process has long been recognized as one method for dissolving various nuclear fuels in nitric acid. In present dissolver designs, an inert basket acts as the anode and as a container for the fuel pieces. A major difficulty in such designs is the maintenance of good electrical contact between the basket and the fuel to be dissolved. In addition, the use of a relatively high current to achieve reasonable dissolving rates has been considered essential for a basket dissolver, requiring low-potential power sources of high current capacity.

A new electrolytic dissolution technique has been demonstrated on a laboratory scale at the Idaho Chemical Processing Plant which avoids these problems. Metal pieces to be processed are placed in a tubular dissolver into which dissolving solution can be continuously pumped. Current is then passed through the contents of the tube between electrodes placed at each end. The electrodes are immersed in the dissolver solution and need not be in physical contact with the dissolving pieces of metal. A series of electrolytic cells are formed within the dissolver, in which one end of each fuel piece acts as the cathode, reducing nitric acid, while the other end acts as the anode, going into solution. Dissolution rates of $2.5 \text{ mg}/(\text{cm}^2)(\text{min})$, with ten cells formed in series, have been demonstrated for stainless steel. Using the same technique, aluminum can be dissolved to a basic solution, and zirconium can be oxidized to ZrO_2 .

II. INTRODUCTION

A variety of metals used in the fabrication of nuclear fuel elements can be dissolved electrolytically in nitric acid⁽¹⁾. In previous work, this has been accomplished by placing the metal to be dissolved in a niobium or tantalum anode basket surrounded by a container serving as the cathode. Niobium and tantalum, though not noble metals, are at all times coated with an oxide layer that will not dissolve in nitric acid, and thus can be used for anode basket construction. Difficulty has frequently been encountered, however, in maintaining good electrical contact between the anode basket and the dissolving fuel pieces⁽²⁾. Vibration and other techniques have been used to circumvent this difficulty, with only moderate success. In addition, basket dissolvers require the use of high-current low-potential power sources, with their heavy bus-bars, water-cooled conductors, and high cost.

A new design for an electrolytic dissolver which avoids these problems has been investigated on a laboratory scale at the Idaho Chemical Processing Plant. The dissolver consists of a simple tube with an electrode at each end. The electrodes need not make physical contact with the dissolving metal, but serve merely to introduce current to the dissolver, which then passes through the solution in the vessel. The tube must be lined with an insulating material, which can consist of a natural oxide coating, if niobium or tantalum are used as the material of construction. Fuel pieces and nitric acid are introduced to the dissolver in a conventional manner. Dissolution is effected by applying a direct current voltage to the electrodes; the resulting current forms effectively a series of electrolytic cells between adjacent fuel pieces, which dissolve at the anodic end. The principle is illustrated in Figure 1.

Since in this series operation the current could participate more than once in an electrolysis reaction it was desirable to define a term expressing the degree of this multiple use of current. The current utilization factor was selected for this function and is defined by:

$$\text{Current utilization factor} = \frac{(\text{Grams of metal dissolved})}{(\text{Electrochemical equivalent of metal})(\text{Faradays})}$$

This factor is identical to the current efficiency when there is only one dissolving anode in the series, but represents the number of theoretical cells attained when there is more than one anode. Such a design eliminates the use of an anode basket, avoids the need for electrical contact between the fuel pieces and the dissolver, and greatly simplifies the power supply by using high voltage and low current instead of high current and low voltage.

FIGURE I
PRINCIPLE OF SERIES ELECTROLYTIC DISSOLVER

CPP-S-2036

III. EQUIPMENT DESIGN

A. Dissolvers

Four dissolvers of various designs were used in these tests. Two were modified by lining them with tantalum or inserting a tantalum strip. Figure 2 contains sketches of the dissolvers used. The modified dissolvers are shown separately to indicate the manner in which the tantalum was inserted.

Design No. 1 is an 18-mm glass U-tube with no provision for temperature control. This dissolver has ports at either end for inserting removable electrodes, and also has small side arms for metering in feed solution. Condensers were attached at either end. The metal was charged by removing both the condenser and the electrode from the desired end.

Design No. 2 is similar to No. 1 except that a piece of tantalum shim stock (approximately 1/2 inch wide by 10 mils thick) of a length nearly equal to the interelectrode distance was placed in the dissolver, to indicate whether or not it might be used as a material of construction for the dissolver wall. If the tantalum becomes sufficiently coated with oxide under the dissolution conditions so as to lose electrical contact with the dissolving metal pieces, it should not participate in the conductance of the electric current, and thus should be a suitable material for the vessel.

Design No. 3 is a jacketed 18-mm glass U-tube with ports for removable electrodes and separate heads for the condensers. The temperature in the dissolver body was controlled by passing cooling water through the jacket, but no provision was made for controlling the temperature at the electrodes.

Design No. 4 is a jacketed 18-mm glass U-tube with sealed-in-position platinum electrodes. This dissolver had separate heads for the condensers. Cylindrical electrodes were positioned in recesses in the dissolver wall. Feed solution was metered through side arms located at each end of the dissolver and pieces of metal were added to the dissolver

DISSOLVER
No. 1

DISSOLVER
No. 2

DISSOLVER
No. 3

DISSOLVER
No. 4

DISSOLVER
No. 5 & 6

No. 5
GLASS

No. 6
TANTALUM
SHIM STOCK

Figure 2
DISSOLVER DIAGRAMS

while it was in operation. The cooling water jacket covered the electrode area as well as the dissolver body.

Design No. 5 is a jacketed 20-mm glass straight-tube dissolver with sealed-in-position electrodes, consisting of two tantalum cylinders sealed in recesses at either end and a platinum cylinder sealed in a recess at the center of the dissolver tube. The dissolver had a removable liner made of 18-mm glass tubing, containing a number of approximately 1/8 inch holes at the areas in front of the electrodes. The vessel was positioned vertically and the dissolver solution was metered in at the bottom, overflowing at the top. This apparatus was intended to use either alternating or direct current and provide self-rectification for the former. With direct current, the tantalum electrodes were of necessity made cathodes; thus, when used with direct current, there was an anode in the center and a cathode at either end.

Design No. 6 was the same as No. 5 except that a liner made of tantalum shim stock was substituted for the glass liner. The perforated holes in front of the electrodes were in separate sections to reduce the direct polarizing effect of the electrodes on the tantalum liner.

B. Power Supplies

Two direct-current and two alternating-current power supplies were used. In low voltage direct current tests, use was made of an existing high-capacity filtered power supply which had a maximum output of 53 volts. In order to run tests at higher voltages, a somewhat simplified power supply was obtained, consisting of a 0-220 volt autotransformer connected to a full-wave rectifier.

Most of the alternating-current tests were run using an autotransformer providing two terminals, one of which was hot and the other ground. Use was also made of a 0-220 volt autotransformer which provided two hot wires and a ground wire.

C. Current Measurement

The total direct current passed during a particular experiment was measured with a copper coulometer and these values were used in the calculation of the current utilization factor.

IV. EXPERIMENTS AND RESULTS

A. Test Procedure

Eight-molar nitric acid was used as the dissolving reagent in all tests. In some of the preliminary work, an assortment of stainless steel scrap was used; however, in most of the reported experiments, one of the following metals served as fuel pieces: 304L stainless steel slugs approximately 1" x 1/2" x 3/16"; 6061 aluminum slugs approximately 1" x 1/2" x 1/4"; or Zircaloy-2 slugs approximately 1" x 1/2" x 3/16".

Considerable variations were made at times in the test procedure. Some generalized statements can be made, however. Tests were mostly carried out under continuous process conditions. The dissolver solution was metered into the dissolver with a Sigma pump at a uniform rate and drawn off as it reached the level of the outlet side arm. Because the flow rate was low and there was considerable agitation of the vessel contents, the composition of the effluent was approximately the same as that of the solution throughout the dissolver. Fresh metal was added whenever there was sufficient room for another piece. Samples and run data were usually collected for a particular period starting and ending with equilibrium conditions. The duration of these test periods as well as the length of time of operation prior to the specific test period are given in the tabulated data. The latter, defined as the equilibration time, represents the time required for manipulations prior to and between tests and, incidentally, is the period allowed for the dissolver to approach steady state or "equilibrium" operation.

Analytical work was performed by the Idaho Chemical Processing Plant Control Laboratory. In reporting the solution analyses the notation $\underline{N}^a$ or $\underline{N}^b$ represents the stoichiometric acidity or acid deficiency of solutions.

B. Dissolution of Stainless Steel

For the direct current dissolution of stainless steel, both the current and the current utilization are dependent on the applied voltage. The overall dissolution rate, which is a product of these variables, is approximately proportional to the applied voltage over the range studied.

The best dissolution rate for stainless steel was obtained with dissolver No. 4 using the unfiltered 0-220 volt direct current power supply. At 105 to 130 volts, and about 80°C, dissolution rates of 2.2 to 2.5 mg/(cm²)(min) were observed. Under these conditions, the current utilization factor was 9.60 to 10.70; 9.6 to 10.7 "theoretical cells" might be said to have formed in the dissolver. The solution flow rate was approximately 3 ml/min, which represents a turnover of 3 per cent of the dissolver volume/minute. The resulting product was approximately 1.9 molar in total stainless steel components and one molar in free acid, and free of sludge. In tests run at lower voltages and temperatures, there was an appreciable amount of finely divided black sludge in the dissolver product. This material has not been characterized. It was observed that as the voltage and current were increased, a point was reached where arcing took place between adjacent pieces of stainless steel. In the test described above, voltage was maintained at a level just below the point at which arcing would occur. The current density in the metal for these conditions was calculated to be from 4.7 to 5.8 amps/cm². Details of the stainless steel dissolution runs using direct current are shown in Tables 1 and 2 in the Appendix.

C. Dissolution of Aluminum

The few runs using aluminum as fuel were not sufficient to completely delineate the system's characteristics. An attempt was made in all of the aluminum experiments to produce basic aluminum nitrate. Under such conditions, it is probable that chemical reaction kinetics played an important role in the overall rate of reaction. The effect of voltage on the dissolution rate of aluminum was different and apparently more complex than that observed for stainless steel. Current increased with increasing voltage, but the current utilization decreased; the overall dissolution rate was not proportional to voltage for these runs, as it was in the case of stainless steel dissolution.

The highest dissolution rate observed for aluminum was approximately 0.9 mg/(cm²)(min), achieved in dissolver No. 4 at 110 volts direct

current. The solution produced in this test was 2.32 molar aluminum and 0.27 molar acid deficient. At this same composition, the rate of dissolution of aluminum in mercury-catalyzed nitric acid has been found to be about $4 \text{ mg}/(\text{cm}^2)(\text{min})$. More concentrated and more acid deficient solutions were produced in other experiments, but at lower dissolution rates. Tables 3 and 4 in the Appendix list details of aluminum dissolution tests.

During the course of aluminum dissolution to acid deficient conditions, two effects were noted: (1) brown fumes of NO_2 no longer were evolved as the system passed from acid to acid deficient conditions, and (2) solutions produced in the acid deficient region were darkened appreciably by small amounts of finely divided sludge. This sludge could be readily centrifuged, and would settle from the solution if allowed to stand overnight. Upon standing, a portion of the sludge appeared to dissolve, as evidenced by the solution becoming colored with the simultaneous evolution of gas bubbles.

D. Use of a Simulated Tantalum Container

In some of the tests, either a tantalum strip or a tantalum liner was placed in the dissolver to ascertain whether tantalum might be used as a container material. These experiments showed that tantalum could be used up to 35 volts direct current in the U-tube dissolver while dissolving stainless steel, and up to 50 volts direct current in the straight tube dissolver while dissolving aluminum. Higher voltages caused metal-to-tantalum conduction which defeated the desired series electrolytic dissolution effect. Shielding the tantalum from the direct polarizing effect of the electrodes was helpful in increasing the useful operating voltage. It was also noted that the insulating property of the oxide coating on the tantalum improved with length of run time.

E. Oxidation of Zircaloy-2

In a single test, it was shown that Zircaloy-2 could be oxidized in a series electrolytic dissolver. Since the electrolysis of zirconium is temperature dependent, the reaction was started by passing hot water through the dissolver jacket. As soon as the reaction was started, cooling

water was applied to maintain the system at the optimum temperature. The test could not be continued long enough to reach equilibrium, since no special provision had been made for the removal of the oxide sludge formed in the dissolver. Table 6 lists conditions for the experiment with Zircaloy-2.

F. Effect of Alternating Current Voltage

Previous investigators⁽³⁾ have studied the use of alternating current for dissolution of stainless steel in nitric acid, and found that the current efficiency was only 50 per cent as great as with direct current. In scoping studies, in which no quantitative data were taken, it was observed at the Idaho Chemical Processing Plant that when two pieces of stainless steel in 8-molar nitric acid are treated with alternating current, a thick oxide scale is formed that inhibits further flow of current. Use of alternating current has not been given much consideration partly for the above reason and also because it does not appear applicable to a basket dissolver. In order for alternating current to act on stainless steel at all times, one would have to have two baskets containing stainless steel and acting as separate electrodes. The series dissolver overcomes this latter objective.

The only tests run completely with alternating current were made on aluminum. It was observed that aluminum could be dissolved in this manner but that the rate was only 32 per cent as great as when approximately the same direct-current voltage was applied under comparable conditions.

Stainless steel was dissolved with alternating current but in this case one of the electrodes used was made of niobium and this gives at least partial rectification of the current. In this instance, the dissolution rate was 41 per cent of that for approximately the same direct-current voltage under similar conditions. This test was performed with dissolver number three, in which the cooling jacket did not cover the electrode areas; as a result, solution boiling occurred at the electrode areas.

Table 3 lists the information from runs using alternating current.

V. CONCLUSIONS

In the series electrolytic dissolver, stainless steel and aluminum can be dissolved in nitric acid without any metal to metal electrical contact. Zircaloy-2 can be oxidized to zirconium oxide by the same technique. The passage of electric current through a series of pieces of one of these metals in nitric acid results in the formation of a series of electrolytic cells. One end of each piece of metal dissolves electrolytically while the other end acts as a cathode. The most desirable condition is for the metal pieces to be separated, but contact between adjacent pieces does not prevent the reaction. This series effect multiplies the current utilization at the expense of higher voltages, thus allowing the use of a high voltage, low current power source.

When using this technique, material of the dissolver vessel must not conduct an appreciable quantity of current, thereby bypassing the metal pieces to be dissolved. The dissolver must therefore be either made of a non-conductive material or lined with a non-conductive material. Although niobium and tantalum are good conductors, they are at all times coated with an insulating oxide and are for that reason possible materials of construction for the dissolver vessel. The tests run with tantalum indicate that it can only be used below about 50 volts in dissolvers of the design considered in these experiments. However, modifications such as treatment to increase the thickness of the oxide coating, shielding, and perhaps even a separate segmented liner might be helpful in improving the useful voltage range.

At this stage in the development, the series dissolver appears to have the following advantages over the basket type electrolytic dissolver:

- 1) The anode basket to fuel "contact resistance" problem is entirely avoided.
- 2) A relatively high voltage-low current power supply is used. The advantages of the higher voltage power supply are more efficient transmission of power with smaller conductors and more uniform voltage regulation.

- 3) The electrodes can be designed to be relatively small and easily removable. A variety of materials can be used as the cathode and there are at least three possible choices for the anode. Since lower total currents are involved, arcing and resistance heating damage to the electrodes may be less.

On the other hand, certain fundamental problems associated with the series dissolver concept and not with the basket dissolver have not yet been explored. These are the definition of the principles to be used for geometrical scale-up, and the verification of the use of a conventional metal container or the demonstration of the feasibility of the use of a metal container lined with an electrical insulator.

The development of this dissolver concept will be continued in the laboratory with studies of the geometrical and electrical parameters related to scale-up, the design and placement of the inert electrodes, the electrochemical reactions at high current densities at both the inert and dissolving electrodes, and the relationship of the leakage currents through the metal container to the dissolution efficiency.

VI. APPENDIX

The following tables summarize the results of the tests for which data were taken:

Table 1

SERIES DISSOLUTION OF STAINLESS STEEL USING DIRECT CURRENT PRELIMINARY TESTS

Reagent: 8M nitric acid

DISSOLVER DIAGRAM NO.	2	1	2	3	4
VOLTS (DC)	10-25	20	30-35	43	43
CURRENT (AMPS)	1.8	1.7	1.0	0.98	1.2
ESTIMATED TEMPERATURE (°C)	60	60	60	25	25
EQUILIBRATION TIME (min)	0	0	0	25	0
LENGTH OF TEST (min)	680	120	530	890	630
FLOW RATE (ml/min)	(1)	(1)	0.61	1.07	1.2
DISSOLUTION RATE (mg/(cm ²)(min))	0.36	0.70	0.51	0.57	0.79
CURRENT UTILIZATION FACTOR	2.54 ⁽²⁾	4.85	4.56	7.00	8.32
SOLUTION ANALYSIS	Fe 0.48	Fe 0.35	Fe 1.09	Fe 0.86	Fe 0.96
(molarity of	Ni .007	Ni .005	Ni 0.18	Ni 0.14	Ni 0.18
specified component)	Cr .014	Cr .010	Cr 0.28	Cr 0.26	Cr 0.34
	Na ^a (3)5.35	Na ^a 6.11	Na ^a 2.20	Na ^a 3.17	Na ^a 2.08
			NO ₃ 6.85		NO ₃ 6.35

Note 1. 25 ml was added approximately every 15 minutes.

Note 2. For approximately the first half of the test, the tantalum strip shunted the current around all of the metal except the terminal piece at the cathode end. About midway in the run, the Ta strip lost contact with the terminal piece of metal and thereafter, all of the metal behaved uniformly.

Note 3. N^a refers to normality of acid in solution.

Table 2
 SERIES DISSOLUTION OF 304 STAINLESS STEEL
 SEVENTEEN-HOUR DEMONSTRATION RUN
 Reagent: 8M nitric acid
 Dissolver: No. 4

VOLTS (DC)	105-130	105-130	105-130	105-130
CURRENT (AMPS)	2.5	2.7	3.1	
TEMPERATURE (°C)	75-85	80-90	90	
EQUILIBRATION TIME (min)	170	420	885	
LENGTH OF TEST (min)	60	165	150	1035
FLOW RATE (ml/min)	3.0	3.03	3.33	3.1
DISSOLUTION RATE (mg/(cm ²)(min))	2.5	2.2	2.5	2.54
CURRENT UTILIZATION FACTOR	10.7	10.0	9.6	
SOLUTION ANALYSIS (molarity of specified component)	Fe 1.37 Ni 0.16 Cr 0.38 N ^a 0.96	Fe 1.38 Ni 0.16 Cr 0.30 N ^a 1.00 NO ₃ 6.20	Fe 1.31 Ni 0.15 Cr 0.30 N ^a 1.30 NO ₃ 6.55	

Note: The above tests are really segments of one long continuous run. To collect the data in an individual test, a coulometer was attached for the specified period of time and the effluent solution was collected, measured and analyzed. The last column applies to a composite for the entire test. Individual dissolution rates were based on solution analyses, and the overall rate was based on the total amount of stainless steel dissolved.

Table 3

SERIES DISSOLVER TESTS USING ALTERNATING CURRENT

Reagent: 8M nitric acid

ALLOY DISSOLVED	SS	6061 Al	6061 Al	6061 Al
DISSOLVER DIAGRAM NO.	3	4	4	4
VOLTS (AC)	100 ⁽¹⁾	110	110	110
CURRENT (AMPS)	2	2.2	2.1	1.5
TEMPERATURE (°C)	90	90	95	90
EQUILIBRATION TIME (min)	45	60	(2)	(2)
LENGTH OF TEST (min)	580	45	50	40
FLOW RATE (ml/min)	0.95	1.5	1.3	0.5
DISSOLUTION RATE ⁻ (mg/(cm ²)(min))	1.0	0.54	0.54	0.23
SOLUTION ANALYSIS (molarity of specified component)	Fe 0.86 Ni 0.18 Cr 0.35 N ^a 1.40	Al 1.88 N ^a 1.52	Al 2.14 N ^a 0.73	Al 2.37

Note 1. One electrode was made of niobium causing half-wave rectification.

Note 2. The three tests on aluminum were run in consecutive order. At the conclusion of a run, the flow rate was lowered and the next test started immediately.

Table 4

SERIES DISSOLUTION OF ALUMINUM TO ACID DEFICIENCY

Reagent: 8M nitric acid

Alloy: 6061 aluminum

Dissolver: No. 4

	<u>Experiment 4-A</u>			<u>Experiment 4-B</u>	
VOLTS (DC)	43	43	43	100-110	100 110
CURRENT (AMPS)	0.42	0.21	0.62	1.60	1.17
TEMPERATURE (°C)	87	85	82	70-90	60-90
EQUILIBRATION TIME (min)	60	120	240	60	300
LENGTH OF TEST (min)	60	120	180	240	287
FLOW RATE (ml/min)	0.83	0.58	0.33	2.1	1.4
DISSOLUTION RATE (mg/(cm ²)(min.))	0.44	0.40	0.20	0.92	0.72
CURRENT UTILIZATION FACTOR	30.6	47.4	8.1	15.8	15.5
SOLUTION ANALYSIS	Al 2.72	Al 3.54	Al 3.10	Al 2.32	Al 2.69
(molarity of	N ^b (I) 1.13	N ^b 2.35	N ^b 2.82	N ^b 0.27	N ^b 1.48
specified component)					

Note 1. N^b refers to acid deficiency or "normal basicity" of solution.

Table 5

EFFECT OF TANTALUM DISSOLVER LINER ON THE
SERIES DISSOLUTION OF ALUMINUM

Reagent: 8M nitric acid

Alloy: 6061 aluminum

Voltage: 53 volts DC

DISSOLVER DIAGRAM NO.	5 (glass tube)	6 (tantalum liner)
VOLTS (DC)	53	53
CURRENT (AMPS)	2.2	3.1
TEMPERATURE (°C)	75	70-80
EQUILIBRATION TIME (min)	180	180
LENGTH OF TEST (min)	180	180
FLOW RATE (ml/min)	0.83	0.75
DISSOLUTION RATE (mg/(cm ²)(min))	0.41	0.40
CURRENT UTILIZATION FACTOR	4.64	3.25
SOLUTION ANALYSTS (molarity of specified component)	Al 2.55 Nb 0.50	Al 2.79 Nb 1.10

Table 6

OXIDATION OF ZIRCALLOY-2 IN SERIES DISSOLVER

Reagent: 8M nitric acid

DISSOLVER DIAGRAM NO.	4
VOLTS (DC)	75
CURRENT (AMPS)	4.2
TEMPERATURE (°C)	65-75
EQUILIBRATION TIME (min)	0
LENGTH OF TEST (min)	180 ⁽¹⁾
FLOW RATE (ml/min)	0.7
DISSOLUTION RATE (mg/(cm ²)(min))	0.64
CURRENT UTILIZATION FACTOR	1.60
SOLUTION ANALYSIS (molarity of specified component)	Zr 0.102 Nb 7.76

Note 1. The test was terminated when the dissolver became plugged with ZrO₂.

VII. LITERATURE CITED

1. C. M. Slansky, et al, "Review of Research and Development at the Idaho Chemical Processing Plant on the Electrolytic Dissolution of Nuclear Fuel", IDO-14535, Feb. 3, 1961.
2. E. C. Pitzer, "Electrolytic Dissolution of Stainless Steel Clad Fuel Assemblies", KAPL-653.
3. E. S. Occhipinti, "Reprocessing of Power Reactor Fuels", fourth quarterly progress report 1958, DP-338.

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION