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A PILOT-PLANT STUDY OF THE ELECTROLYTIC DISSOLUTION OF STAINLESS STEEL-CLAD FUEL

R. A. Brown

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BY 914/Kahn/amb
DATE 8/27/71

IDAHO NUCLEAR CORPORATION

NATIONAL REACTOR TESTING STATION
IDAHO FALLS, IDAHO 83401

Date Published—June 1971

PREPARED FOR
U.S. ATOMIC ENERGY COMMISSION

IDAHO OPERATIONS OFFICE
UNDER CONTRACT NO. AT(10-1)-1230

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A PILOT-PLANT STUDY OF THE ELECTROLYTIC
DISSOLUTION OF STAINLESS STEEL-CLAD FUEL

by

R. A. Brown

IDAHO NUCLEAR CORPORATION

A Jointly Owned Subsidiary of

AEROJET GENERAL CORPORATION
ALLIED CHEMICAL CORPORATION
PHILLIPS PETROLEUM COMPANY

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ABSTRACT

The electrolytic dissolution of simulated and actual stainless steel-clad fuel was demonstrated in a pilot-plant unit. The pilot-plant dissolver, using continuous feed and discharge of solvent nitric acid, was operated successfully for more than 2000 hours. The anodic dissolution process is a function of the current passing through the dissolver; control of the current and the acid feed rate is sufficient for control of product uranium concentration. EBR-II fuel elements, consisting of 91 stainless steel-clad uranium pins, were dissolved at predicted rates with a very small loss of uranium in the undissolved solids. No hazards were indicated from dissolution of the sodium used as a bonding agent in these fuel pins. The observed dissolution rate of fuel was used to predict the general operating behavior of the plant-scale dissolver.

SUMMARY

A continuous electrolytic dissolver, applying the solution-contact principle, was used for a pilot-plant study of important process variables in the dissolution of stainless steel-clad fuels. The anodic dissolution process was carried out in a niobium basket suspended in a titanium dissolver. Nitric acid was fed and discharged continuously from a recirculation loop which included the dissolver, a surge tank, and a heat exchanger.

An extensive study, providing more than 2000 hours of operating experience, was made using stainless steel pipe as simulated fuel. Five parameters were varied in a fractional factorial experiment to provide statistical estimates of their effects on dissolution rate and efficiency, formation of undissolved solids, and acid consumption. Of the parameters (dissolver current, temperature, acid concentration, fuel size, and gelatin concentration) considered, only the current had a significant effect on dissolution rate. Dissolution of stainless steel occurred at 0.60 grams per ampere-hour; undissolved solids were generated at 0.012 grams per ampere-hour; nitric acid (as hydrogen ion) was depleted at 0.037 moles per ampere-hour. Current utilization, a measure of electrical efficiency, was not affected by process variables.

Unirradiated EBR-II fuel elements were dissolved in a brief experimental run. The fuel dissolved rapidly and safely; sodium dissolution caused no fire or explosion hazard. Current utilization in the partially-loaded basket was 0.45 grams of stainless steel per ampere-hour; the total dissolution rate was 1.35 grams per ampere-hour. In a fully-loaded system, an overall dissolution rate of 1.8 grams per ampere-hour can be expected. Concentration-time profiles indicate that the fuel meat dissolves more rapidly than the steel cladding, thus eliminating any potential criticality hazard from undissolved uranium. Approximately three percent of the fuel charged remained undissolved, representing a 0.09% loss of the total uranium charged. Product hydrogen concentration was 0.4% in the diluted off-gas stream, well below the explosive limit of 4%. [15]

The two critical control variables for the system are current and acid feed rate. A mathematical model of the electrolytic dissolver recirculation loop was formulated and used as a basis for both analog and digital computer simulation of the plant dissolver unit now under construction. Both transient and steady-state operating conditions were simulated. On the basis of the predicted behavior, safe start-up conditions and operating policies were defined.

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I. INTRODUCTION

Electrolytic dissolution is a practical method for dissolving stainless steel-containing nuclear fuels. Stainless steel, which dissolves very slowly in nitric acid, will dissolve rapidly if made anodic in an electrochemical cell; the rate of solution is proportional to the current passed through the anodic surface.

The advantages of electrolytic dissolution over other aqueous processes, e.g., dissolution in sulfuric acid (SIR or Sulfex Process) or aqua regia (Darex Process), are:

1. Reduced corrosion
2. A nitrate dissolver product requiring no complexing before extractive processing in existing equipment
3. Compatibility with existing solvent extraction processes
4. Acceptable liquid and solid waste generation rates.

1. DEVELOPMENT HISTORY

The electrolytic dissolution of stainless steel-clad fuel was first studied by Pitzer^[1] at the Knolls Atomic Power Laboratory (KAPL) in 1951. Pitzer demonstrated the general feasibility of anodic dissolution. Laboratory-scale studies were also made by Martin and Waterman^[2] at the Atomic Energy Research Institute, Harwell, England in 1958. They reported uranium recoveries of >99.5 percent and rapid dissolution of stainless steel or stainless-UO₂ cermet over a wide range of nitric acid concentrations. Current efficiencies (experimental current utilization/theoretical current utilization) of 100 percent were achieved using 8M nitric acid and direct current while only 63 percent efficiency was obtained using alternating current.

H. E. Henry^[3] of Savannah River Laboratories (SRL) investigated electrolytic dissolution of stainless steel in 1959. He reported that dissolution rate was controlled by current density only; neither acid concentration, (2M to 10M HNO₃), salt concentration (0 to 0.9M Fe³⁺),

nor temperature (30° to 90°C) had any demonstrable influence on dissolution rates.

Clark's^[4] study at SRL established practical dissolution rates and low uranium or plutonium losses for all fuels except Zr-U alloys, which formed a ZrO₂ sludge resulting in a 50 percent uranium loss. This work, reported in 1961, also demonstrated that the valve metals, columbium and tantalum, retain their properties after exposure to 10⁹ rad.

A summary of electrolytic dissolution studies at SRL was made by Caracciolo and Kishbaugh^[5] in 1964. Their report describes the operation of a 5000-ampere dissolver using the "solution contact" principle. Unirradiated fuel assemblies of both stainless steel and Zircaloy were dissolved at rates of 0.50 and 0.25 grams per amp-hour, respectively, during a total operating period of 400 hours. Uranium dioxide dissolved as soon as it became exposed, and sodium-bonded fuels were dissolved safely at a temperature of 60°C.

The development of an electrolytic dissolution process at ICPP began with literature surveys and laboratory-scale experimentation in 1958^[6,7]. This work, justified by the anticipated need for reprocessing of APPR (Army Package Power Reactor) and HTRE (High Temperature Reactor Experiment) fuels, was directed at dissolution of fully enriched stainless steel and Nichrome-clad fuels. The electrochemistry of dissolution in nitric acid was established^[6,7,8,9] and the effects of electrolyte composition and temperature on dissolution rates were studied. A basic study of the physical properties of electrolytes was made, and materials of construction were evaluated for use in an electrolytic dissolver^[6,7]. The recommended material for anode construction was niobium, which also could serve as a cathode. Carpenter-20 cathodes were shown to have a limited life and were restricted to narrow operating ranges.

Dissolution of several dozen alloys (which were used as the anode in a small dissolver) was achieved at high current efficiencies. Significant amounts of undissolved solids, ranging from 1 to 7 percent of total anode weight loss, were observed during dissolution of APPR plate, stainless steel 304, 304L, 347, and Carpenter-20. The average amount of

residue formed during dissolution of APPR plate was 2.5 weight percent of the total anode weight loss; uranium losses ranged from 0.001 to 0.20 percent of uranium dissolved^[6,7]. The ICPP laboratory investigations also solved the problem of current distribution by providing a solution gap between fuel elements and the electrodes (the solution-contact principle).

Process problems remaining unsolved were the effects of fuel type and electrode geometry on dissolver current, off-gas formation, composition control, and continuous operation with undissolved solids in both the dissolver and extraction systems. Verification of the predicted effects of electrolyte composition, temperature, voltage, and current density on dissolution rate and sludge formation was also required. A small pilot-plant was designed and installed at ICPP in 1963 to answer these questions. A horizontal, 4-inch long, vee-shaped dissolver, employing the "solution-contact" principle, was chosen because of fuel element geometry and criticality considerations. The pilot-plant experimental program was used to both evolve the dissolver design and study the effects of process variables (e.g., fuel characteristics, dissolver loading, dissolvent composition, temperature, voltage, and undissolved solids) on dissolver operation.

Eighteen test runs were made in the pilot plant at an average dissolution rate and current density of 1.0 g/hr-cm² and 1.6 amp/cm², respectively, (based on anode area) with an average current utilization of 0.66 g/ampere-hr. The exploratory and developmental nature of this work, with the requisite wide range of operating conditions, precluded quantitative evaluation of the factors affecting the process. Qualitative conclusions were^[10]:

Fuel Composition	- No significant observation.
Fuel Size	- Higher current utilization with smaller elements.
Dissolver Loading	- Reduced current utilization when less than half-full. Elements shorter than the dissolver permit current by-passing. An insulating plate in the unused part of the dissolver solved this problem.

Dissolver Solution Composition	- No significant observation. (Laboratory studies show that conductivity passes through a maximum as acid concentration increases.)
Dissolvent Temperature	- No significant observation, although maximum current density was observed at an average (T_{in} , T_{out}) temperature of 49°C.
Voltage	- Current varies linearly over the range of 7 to 25 volts, with no apparent detrimental effects on the electrodes.
Undissolved Solids	- Approximately one percent of the fuel charged remained undissolved. Addition of 200 ppm gelatin prevented the coating of surfaces by sludge. Uranium associated with undissolved solids represented an average loss <0.002 percent of the uranium charged.
Gas Evolution	- Evolution of gas ranged from 0.004 scfm/amp-hr for stainless to 0.014 scfm/amp-hr for Nichrome. Gases evolved were nitrogen, oxygen, hydrogen, and oxides of nitrogen. Hydrogen concentration was held below the explosive limit by air dilution.
Corrosion	- No corrosion of the titanium vessel, niobium basket, or platinum anode was observed.

2. PRESENT SITUATION

In 1967, a larger dissolver system was installed in the Hot Pilot Plant at the ICPP. This unit, using a 24-inch long dissolver, (length being the only change in geometry from the preceding 4-inch dissolver) was designed to evaluate dissolver system improvements, determine the effect of operating parameters, and to obtain dissolution rate data for EBR-II fuel elements.

The Electrolytic Dissolver Pilot Plant, (EDPP) Figure 1, is essentially a continuous stirred tank reactor system, with a recycle loop for heat transfer. Nitric acid is fed and discharged continuously; fuel elements are fed on demand.

The major components of the EDPP are the:

Dissolver	- Titanium dissolver body which also serves as the cathode- Perforated niobium basket suspended in the dissolver- A niobium anode with a platinum sheet bonded to the surface facing the basket.
Surge Tank	- Stainless steel tank used to maintain constant acid volume in the dissolver- Immersed recirculation pump for acid
Heat Exchanger	- Stainless steel shell and tube counter-current heat exchanger for removal of heat generated in the dissolver
Automatic Control Systems	- Temperature control in dissolver by regulation of coolant flow rate to heat exchanger- Volume control by level control in surge tank, which in turn controls product discharge rate (fresh acid feed rate is fixed)
D. C. Rectifier	- Supplies current of 3000 amps (at up to 20 volts) operable with either current or voltage control

The 24-inch dissolver was installed to test design alternatives, operating procedures, and dissolution characteristics prior to final design, construction, and operation of the plant size (80" long) dissolver for EBR-II fuel processing.

INC-B-18740

FIGURE 1. The Electrolytic Dissolver Pilot Plant Schematic

3. FUTURE FUEL DISSOLUTION STUDIES

Future pilot-plant and laboratory studies will include dissolution of plutonium and alloyed fuels. Electrolytic dissolution of radioactive fuels will be studied in the Multicurie Cell in laboratory-scale apparatus. Fuels scheduled for dissolution in both the laboratory and pilot plant are EBR-II fuel sections, plutonium metal, and the alloys, uranium molybdenum, uranium-plutonium, and uranium-molybdenum-plutonium.

II. THE PILOT PLANT STUDY

Pilot-plant study of electrolytic dissolution was made in two stages, dissolution of simulated fuel elements followed by dissolution of EBR-II fuel elements (unirradiated). The use of simulated fuel, mainly stainless steel pipe, was necessary because of the limited supply of actual fuel (only six elements) and the prohibitively high cost* of fabrication of elements solely for pilot-plant testing.

1. SIMULATED FUEL DISSOLUTION

Electrolytic dissolution of stainless steel pipe provided data on the rate-controlling step in the process. The material dissolved was mainly Type 304 stainless pipe (Schedule 40). After preliminary operation and equipment testing were completed, a polyethylene blank was inserted in the dissolver basket, reducing the working volume of the dissolver and the effective anode area by half. The reduction was made to widen the current density range that could be studied using a 3000 ampere current, to reduce the consumption rate of stainless pipe, and to reduce the load on the heat exchanger, thus permitting operation at lower temperatures.

Two experimental programs were used for the study of the dissolution of stainless steel pipe. During the first period of EDPP operation, a thorough test of the system was required to define significant corrosion problems, evaluate equipment and control systems behavior, and to determine necessary design changes prior to the main experimental investigation. The EDPP was operated continuously for two ten-day periods. Operating conditions were to be held constant during this period to assure unbiased detection of significant problems and time-based trends. In an effort to maximize the return of information from

* Fabrication cost of EBR-II fuel, \$500 per Kg; at pilot-plant dissolution rates, (1.35 g/amp-hr and 3000 amp dissolving 4 Kg per hour) a ten-day operation would dissolve fuel costing \$480,000.

the operation, an evolutionary operation^[11] program was superimposed on the operating conditions.

1.1 Evolutionary Operation

Evolutionary operation (EVOP) is a type of experimental design which is usually used in a production (as opposed to research) environment. Operating conditions are subjected to a system of small perturbations about a standard set of conditions. While the change in the operating parameters is not large enough to cause significant variations in system responses (responses ideally vary within the normal operating limits), repeated cycles of the EVOP pattern (e.g., five conditions for a two-factor study, including a central standard point) will eventually detect those changes which are significant.

The EVOP program used for this part of the EDPP study involved the variation of nitric acid feed concentration and the size of the stainless steel pipe being dissolved. Table I lists the five sets of conditions.

TABLE I

EXPERIMENTAL CONDITIONS FOR EVOP PROGRAM

<u>Experiment</u>	<u>HNO₃ Conc.</u>	<u>Pipe Diameter</u>
1	8.0M	1.0 in.
2	7.5M	0.5 in.
3	7.5M	1.5 in.
4	8.5M	1.5 in.
5	8.5	0.5 in.

The block of five experiments was repeated for six complete cycles during the twenty-day operating period. Each condition was maintained for the time (approximately 12 hours) required to feed 800 liters of HNO₃. Thirty-two runs were made during the 365-hour operating period. Constant operating conditions were not maintained. Dissolver resistance doubled during each ten-day period, thus limiting the maximum attainable current in the dissolver. Acid feed rate control was poor; hourly rates

ranged from 44 to 103 liters/hour. The high variation in acid feed, combined with unsatisfactory conductivity control, caused stainless concentrations in the product solution to vary from 12.5 to 27.5 grams/liter.

The capability of the cyclic EVOP pattern to damp out time-based trends was shown by analysis of the dissolver current data. Table II lists average operating conditions calculated from 25 of the runs (five complete cycles). (Complete data for the EVOP cycles is shown in Appendix C.)

TABLE II

EVOP OPERATING CONDITIONS

<u>Variable</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Acid Concentration (moles/liter)	8.0M	7.5	7.5	8.5	8.5
Pipe Size (inches diam.)	1.0	0.5	1.5	1.5	0.5
Current (amperes)	2320	2380	2340	2360	2390
Resistance ($\times 10^3$)	6.1 Ω	6.5 Ω	6.5 Ω	6.6 Ω	6.6 Ω
Voltage (volts)	14.3	15.5	15.4	15.5	15.1
Dissolution Rate (gr/hour)	1117	1295	1200	1269	1234
Current Utilization (grams/amp-hour)	0.48	0.54	0.51	0.54	0.52

Listed at the bottom of Table II are the average current utilizations calculated from dissolution rate data for the five different operating conditions. No significant difference in current utilization was noted. If either feed acid concentration or pipe size has an effect on dissolution rate, the effect is too small to be detected at the operating levels chosen and with the experimental error levels observed.

The first phase of experimentation in the EDPP yielded the following practical and technical information:

1. No visible corrosion or erosion of the niobium anode or the titanium dissolver vessel.
2. Operating demonstration of the self-insulatory properties of the niobium anode.

3. Acid feed rate control unsatisfactory. (An electromagnetic flowmeter was subsequently installed.)
4. Significant sludge (undissolved stainless steel and silica) accumulation in dissolver and heat exchanger.
5. A 10% caustic wash successfully cleaned the system of sludge film and reduced dissolver resistance.
6. Concentration control by conductivity control system was unsatisfactory. (Fixed acid feed rate and constant current should provide adequate control.)
7. Neither acid concentration (7.5 - 9.5 M) nor pipe size (0.5 to 1.5 inches) caused any significant change in dissolution rate of stainless steel.
8. The average dissolution rate of stainless was 1.3 Kg/hr, which corresponds to a current utilization of 0.54 grams/amp-hr.

1.2 Factorial Experimentation

The electrolytic dissolution of stainless steel is a complex electrochemical process. Coupled with the chemical complexity is the heterogeneous nature of the dissolution process and fuel elements and the changing geometry of the dissolving fuel. Factorial experimentation^[12,13] was chosen as a means for objective evaluation of the effect of operating variables on the behavior of the system. The balanced nature of the designed experiment minimizes confounding (similar or identical patterns of variation of operating conditions), thus assuring independent estimates of effects. Useful estimates of normal operating variability may also be obtained from such analysis.

The experimental variables chosen for study in the pilot-plant program are shown in Table III:

TABLE III

IDENTITY OF CODED VARIABLES

Variable	Coding of Levels		
	-1	0	+1
X ₁ , Dissolver Temperature	115°F	130°F	145°F
X ₂ , Current	1050 Amps	1300 Amps	1550 Amps
X ₃ , Nitric Acid Concentration	7M	8M	9M
X ₄ , Pipe Diameter	1/2"	1"	1-1/2"
X ₅ , Gelatin Concentration	0 ppm	100 ppm	200 ppm

In addition to these five factors, the experimental design (half-factorial) gave independent estimates of the effects of all two-factor interactions (the effect of one variable on the response caused by another variable).

The responses measured were:

1. Dissolution rate of steel, gr/hr
2. Undissolved solids production, gr/hr
3. Acid consumption rate, moles/hr
4. Hydrogen concentration in off-gas, mole %.

Derived responses, used to evaluate effects on a normalized basis, were:

1. Current utilization gms/amp-hr
2. Undissolved solids (UDS) per 100 gms steel dissolved
3. Acid consumption per 100 gms steel dissolved
4. Hydrogen concentration in undiluted off-gas.

Nitrate ion concentrations were measured; however, dissolved gases made analyses unreliable.

Experimental data were analyzed using standard statistical techniques, including the t-test for differences between means. (A digital computer program [Appendix A] was prepared to make the t-test.) Results of the analysis follow in sections 1.21 - 1.27 inclusive.

1.21 Stainless Steel Dissolution Rate is directly proportional to the current passed through the fuel, with 0.60 grams dissolved per ampere hour.

1.22 Current Utilization is the measure of dissolution efficiency expressed as grams dissolved per ampere-hour. The theoretical current utilization for Type 304 stainless steel, calculated from Faraday's Law^[10], is 0.60 for a single-anode surface. Actual current utilization may differ from theoretical substantially because of current leaks, multiple-cell effects, and variations in fuel loading, settling, and geometry. At the 95% confidence level, only pipe size (X_4) affected current utilization. However, an uncontrolled variable, grain size of the steel, was confounded with the levels of pipe size used. Additional experiments indicated that grain size rather than pipe size may have been the controlling variable, with higher current utilization observed at smaller grain size. Experimental details are found in Table IV.

TABLE IV

CURRENT UTILIZATION DATA

<u>Grain Size, (Microns)</u>	<u>Pipe Diameter (inches)</u>	<u>Current Utilization gms/amp-hour</u>
2.5	2	0.70
4.6	1/2	0.65
5.0	1	0.60
5.5 and 9.0	1/4 (two lots)	0.59
8.0	1-1/2	0.56

Since neither fuel element dimensions nor grain size are subject to control in the dissolution process, no further study of this effect was made.

Current density, previously reported as affecting current utilization, did not show a significant effect at the 95% confidence level.

1.23 Undissolved Solids Generation Rate

The electrolytic dissolution of stainless steel leaves a residue of undissolved solids. The solids are mainly stainless steel particles, which result from grain-dropping from the dissolving surface. Niobium carbide, very slightly soluble, is also present in the residue. The generation rate of undissolved solids is directly proportional to dissolver current, producing approximately 0.012 grams per amp-hour or 2 grams per 100 grams of steel dissolved.

The rate of undissolved solids generation per unit weight of steel dissolved did not show any significant dependence on the experimental variables tested.

1.24 Nitric Acid Consumption Rate

The average hydrogen-ion consumption rate was 6.2 moles per 100 grams of stainless dissolved. While no relationship was determined between the hydrogen consumption and any of the five main variables, a highly significant (99% confidence level) effect was observed for the acid concentration - pipe size interaction. A higher dissolution rate (6.6 moles/100 gms steel) was observed at the low levels of the interaction than at high levels (5.76 moles/100 gms). No reason for this behavior has been developed. As with the possible relationship between current utilization and grain size (pipe size), our lack of control of fuel size makes additional study unnecessary.

The hydrogen depletion data may be used to predict the feed acid concentration required to maintain a given dissolver acid concentration. The steady-state material balance for the dissolver yields the following relationship:

$$PH_F - PH_O = 0.062eI$$

where

P = acid feed and exit rate, liters/hour

H = H^+ concentration for feed (F) and exit (O)

e = Average current utilization, g/amp-hour

I = Dissolver current, amps

The required hydrogen-ion concentration in the feed acid is:

$$H_F = 0.062 \frac{eI}{P} + H_O$$

1.25 Hydrogen Concentration in Off-Gas

The evolved hydrogen concentration in the product gas was in the two to three percent (by volume) range; after dilution, the hydrogen concentration was typically less than 0.1%. Hydrogen content showed extreme variability ranging from less than 0.01% to 0.76%. No relationship was observed between the measured values and experimental variables.

1.26 Variability

Experiments 17, 18, 19, 20, 38, and 39 were run at the same conditions to provide estimates of variability and of long-term changes in operating characteristics. (In a time sequence, they represent runs 1, 9, 17, 27, 33, and 34, respectively.)

The current utilization and dissolution rate show substantial variation from their mean values. The calculated standard deviation of the current utilization was 0.086; the 95% confidence limits for the mean are, therefore, ± 0.09 . The true value of the current utilization should (at 95% confidence) be in the range from 0.51 to 0.69 grams per ampere hour. (See Table VI.)

The confidence range represents a potential $\pm 15\%$ variation in the dissolution rate and the concentration of steel and uranium in product acid. The concentration variation during a run (once steady-state is reached) is much less than between runs. No adequate explanation

of the run-to-run variability can be provided. Changes in the position and packing of dissolving stainless pipe or in its microscopic (grain size, surface characteristics) properties could have caused the variations.

1.27 Summary of Results

The complete results of the stainless steel dissolution experiments are shown in Table V. Analysis of these data showed that the process is sensitive to only one variable, the dissolver current. Temperature, in the range from 115°F to 145°F, and nitric acid concentration (5.5 - 8.0M) had no apparent effect on the dissolution rate or other responses. No significant effects were observed for fuel size or gel concentration, the latter being studied for control of undissolved solids formation.

The main block of the factorial experimental program (experiments 1-20) was used to detect the main effects and their interactions.

Experiments 21 to 30 provided data for estimating the possible non-linear effects of the five variables studied. No non-linear effects were observed.

Experiments 31 to 37 were preliminary runs made with 3/4-inch tubing instead of 1/2-inch pipe. These results were not included in the statistical data analysis because of this difference.

Experiments 38 to 42 and 24-2 to 28-2 were used to repeat earlier dissolution conditions or, in runs 40 and 41, to extend the range of current density studied.

1.3 Dissolution of Stainless-Clad Uranium Rods

The brevity of the EBR-II fuel dissolution run limited the amount of data on the behavior of dissolving uranium. To augment these data, simulated fuel was prepared by inserting natural uranium rods (clad in 1/2-inch O.D. stainless steel tubing) into 1/2-inch schedule 80 stainless steel pipe. (Concentration data in Figure 2.)

TABLE V

FACTORIAL DESIGN DISSOLUTION DATA

Run	Expt	Temp X ₁	Current or Current Density X ₂	Acid X ₃	Pipe Size X ₄	Gel X ₅	Temp x C.D. X ₁ X ₂	Temp x Acid X ₁ X ₃	Temp x Size X ₁ X ₄	Temp x Gel X ₁ X ₅	C.D. x Acid X ₂ X ₃	C.D. x Size X ₂ X ₄	C.D. x Gel X ₂ X ₅	Acid x Size X ₃ X ₄	Acid x Gel X ₃ X ₅	Size x Gel X ₄ X ₅	Average Current Utilization g/amp-hr	Dissolution Rate g/hr	UDS Rate g/hr	UDS per 100 gms S.S. Dissolved %	H ⁺ Consumption moles/hr	H ⁺ per 100 gms S.S. Dissolved ($\frac{\text{mol}}{100\text{g}}$)	H ⁺ Concentration molar	NO ₃ Concentration molar	Hydrogen in Undiluted Off Gas mole%	Stainless Steel Concentration g/lit	Dissolver Resistance $\Omega \times 10^3$
23	1	-1	-1	-1	-1	+1	+1	+1	+1	+1	+1	+1	-1	+1	-1	-1	0.569	604	10.0	1.66	34.5	5.71	5.72	7.03	2.80	24.2	11.0
24	2	+1	-1	-1	-1	-1	-1	-1	-1	-1	+1	+1	+1	+1	+1	+1	0.684	720	13.3	1.84	37.2	5.17	5.55	6.86	1.40	27.6	11.0
25	3	-1	+1	-1	-1	-1	-1	+1	+1	+1	-1	-1	-1	+1	+1	+1	0.622	971	16.7	2.32	60.0	6.20	5.48	6.56		25.0	6.2
26	4	+1	+1	-1	-1	+1	+1	-1	-1	+1	-1	-1	+1	+1	-1	-1	0.669	1038	22.1	2.13	52.6	5.07	5.56	6.73		26.8	6.6
10	5	-1	-1	+1	-1	-1	+1	-1	+1	+1	-1	+1	+1	-1	-1	+1	0.564	584	16.8	2.88	40.4	7.00	7.38	8.67	1.00	22.6	8.2
11	6	+1	-1	+1	-1	+1	-1	+1	-1	+1	-1	+1	-1	-1	+1	-1	0.578	602	9.0	1.50	34.3	5.70	7.60	8.80		25.9	9.2
12	7	-1	+1	+1	-1	+1	-1	-1	-1	-1	+1	+1	-1	-1	+1	-1	0.752	1156	19.9	1.72	75.0	6.49	7.00	8.17	1.20	30.8	9.0
13	8	+1	+1	+1	-1	-1	+1	+1	-1	-1	+1	-1	-1	-1	-1	+1	0.770	1202	44.6	3.71	89.6	7.45	6.77	8.72	6.56	29.9	8.9
14	9	-1	-1	-1	+1	-1	+1	+1	-1	+1	+1	-1	+1	-1	+1	-1	0.546	576	15.7	2.72	43.8	7.60	5.44	6.90	3.75	20.0	12.0
18	10	+1	-1	-1	+1	+1	-1	-1	+1	+1	+1	-1	-1	-1	-1	+1	0.581	626	9.1	1.45	39.1	6.24	5.41	6.79	2.00	24.8	7.8
19	11	-1	+1	-1	+1	+1	-1	+1	-1	-1	-1	+1	+1	-1	-1	+1	0.655	1017	19.7	1.94	65.2	6.41	5.35	6.80		26.3	6.9
15	12	+1	+1	-1	+1	-1	+1	-1	+1	-1	-1	+1	-1	-1	+1	-1	0.572	893	12.4	1.39	53.2	5.96	5.52	6.88	3.75	24.5	10.3
20	13	-1	-1	+1	+1	+1	+1	-1	-1	-1	-1	-1	-1	+1	+1	+1	0.570	600	6.1	1.01	35.9	5.98	7.68	8.65	2.20	22.7	8.9
16	14	+1	-1	+1	+1	-1	-1	+1	+1	-1	-1	-1	+1	+1	-1	-1	0.474	502	3.7	0.74	28.4	5.66	7.92	8.89	5.10	19.0	10.1
21	15	-1	+1	+1	+1	-1	-1	-1	-1	+1	+1	-1	-1	-1	-1	-1	0.552	861	23.1	2.68	55.6	6.46	7.53	8.71	1.20	22.8	7.4
22	16	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	0.534	827	19.3	2.34	48.0	5.80	7.81	8.80		21.0	8.0
1	17	0	0	0	0	-1	0	0	0	0	0	0	0	0	0	0	0.508	648	22.6	3.49	47.4	7.31	6.45	7.51	1.88	20.4	8.4
9	18	0	0	0	0	-1	0	0	0	0	0	0	0	0	0	0	0.518	670	21.1	3.15	50.7	7.57	6.37	7.62	0.60	21.0	5.9
17	19	0	0	0	0	-1	0	0	0	0	0	0	0	0	0	0	0.645	844	10.4	1.23	55.1	6.53	6.20	7.70		26.9	6.0
27	20	0	0	0	0	-1	0	0	0	0	0	0	0	0	0	0	0.673	874	12.2	1.40	52.1	5.96	6.33	7.80	2.4	26.7	8.1
28	21	-2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.638	828	16.9	2.08	55.9	6.75	6.28	7.95	0.9	25.5	8.8
29	22	+2	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0.576	748	16.8	2.25	33.3	4.45	6.87	8.01	1.6	23.1	8.0
30	23	0	-2	0	0	0	0	0	0	0	0	0	0	0	0	0	0.440	345	6.2	1.79	17.9	5.18	7.13	8.05	TR	16.8	
31	24	0	+2	0	0	0	0	0	0	0	0	0	0	0	0	0	0.666	1207	42.4	3.51	78.0	6.45	6.30	7.98	TR	25.7	
32	25	0	0	-2	0	0	0	0	0	0	0	0	0	0	0	0	0.559	720	21.8	3.06	42.3	5.87	4.76	5.95	TR	22.2	10.1
35	26	0	0	+2	0	0	0	0	0	0	0	0	0	0	0	0	0.607	793	17.9	2.26	44.1	5.57	8.58	9.67	0.7	24.8	7.1
36	27	0	0	0	-1.5	0	0	0	0	0	0	0	0	0	0	0	0.534	692	30.3	4.38	34.8	5.02	6.92	7.72	1.2	21.9	7.9
37	28	0	0	0	+2	0	0	0	0	0	0	0	0	0	0	0	0.714	927	42.6	4.59	51.8	5.58	6.28	7.69	0.5	29.4	9.4
38	29	0	0	0	0	+2	0	0	0	0	0	0	0	0	0	0	0.683	889	27.4	3.08	49.1	5.52	6.46	7.70	0.6	27.9	8.9
39	30	0	0	0	0	+4	0	0	0	0	0	0	0	0	0	0	0.647	843	28.3	3.36	51.6	6.12	6.38	7.67	0.3	27.1	8.8
2-1	31	-1	-1	-1	-1	+1	+1	+1	+1	-1	+1	+1	-1	+1	-1	-1	0.556	600	14.5	2.42	47.0	7.83	5.11	6.55	1.88	24.5	11.7
3-2	32	+1	-1	-1	-1	-1	-1	-1	-1	-1	+1	+1	+1	+1	+1	+1	0.711	726	17.2	2.37	45.2	6.23	5.26	6.71	0.94	27.9	10.6
8-3	33	-1	+1	-1	-1	-1	-1	+1	+1	+1	-1	-1	-1	+1	+1	+1	0.811	1250	36.5	2.92	93.7	7.50	4.53	6.55	2.38	31.9	14.2
5-4	34	+1	+1	-1	-1+1	+1	-1	-1	+1	-1	-1	-1	+1	+1	-1	-1	0.906	1357	32.2	2.37	93.2	6.87	4.56	6.79	0.94	35.8	11.6
4-28	35	-1	+1	+1	-1	-1	-1	-1	+1	+1	+1	-1	-1	-1	-1	+1	0.908	1379	56.7	4.11	101.3	7.35	6.09	8.09	0.94	39.6	11.5
6-29	36	-1	-1	0	-1	-1	+1	0	+1	+1	0	+1	+1	0	0	+1	0.513	536	18.6	3.47	36.7	6.85	6.62	7.74	1.01	19.0	14.1
7-30	37	+1	-1	0	-1	+1	-1	0	-1	+1	0	+1	-1	0	0	-1	0.399	419	9.6	2.29	29.1	6.95	6.87	7.73	1.88	15.7	13.4
33	38	0	0	0	0	-1	0	0	0	0	0	0	0	0	0	0	0.650	843	16.2	1.92	56.9	6.75	6.20	7.69	TR	26.1	10.05
34	39	0	0	0	0	-1	0	0	0	0	0	0	0	0	0	0	0.603	782	16.2	2.07	44.0	5.62	6.71	7.86	TR	24.2	6.11
40	24-2	0	+2	0	0	0	0	0	0	0	0	0	0	0	0	0	0.608	1092	52.6	4.81	65.7	6.01	6.60	7.74	0.9	24.1	7.38
41	27-2	0	-2	0	0	0	0	0	0	0	0	0	0	0	0	0	0.530	438	9.1	2.07	15.8	3.60	7.20	8.07	1.9	22.2	8.28
42	29-2	0	0	0	-1.5	0	0	0	0	0	0	0	0	0	0	0	0.636	823	11.1	0.86	59.8	7.27	6.12	7.90	0.4	25.9	7.25
43	28-2	0	0	0	+2.0	0	0	0	0	0	0	0	0	0	0	0	0.685	887	17.6	1.98	59.7	6.73	6.10	8.00	0.8	28.3	8.60
44	40	0	+4	0	0	0	0	0	0	0	0	0	0	0	0	0	0.646	1491	52.0	3.49	128.4	8.50	6.42	7.97	1.6	18.4	6.43
45	41	+1	+6	0	0	0	0	0	0	0	0	0	0	0	0	0	0.596	1677	59.2	3.53	149.4	8.90	6.45	7.94	3.2	17.9	5.42

TABLE VI
STAINLESS STEEL DISSOLUTION DUPLICATE EXPERIMENTS

Experiment	Dissolution Rate grams/hour	Current Utilization grams/amp-hour	Undissolved Solids grams/hour	UDS per 100 g. Stainless grams/100 g. SS	Hydrogen Consumption moles/hour	H ⁺ per 100 g. Stainless moles/100 g. SS
17	648	0.508	22.6	3.49	47.4	7.31
18	670	0.518	21.1	3.15	50.7	7.57
19	844	0.645	10.4	1.23	55.1	6.53
20	874	0.673	12.2	1.40	52.1	5.96
38	843	0.650	16.2	1.92	56.9	6.75
39	782	0.603	16.2	2.07	44.0	5.62
Mean	777	0.598	16.5	2.21	51.0	6.62
95% Confidence Ranges	±101	±0.090	±6.8	±0.97	±5.0	±0.79

The dissolver was operated for 80 hours at 130°F, an acid concentration of approximately 8M and a current of 3000 amperes.

The concentration profiles for the run are listed below in Table VII and plotted in Figure 2. Contrary to results from the EBR-II fuel dissolution, the uranium did not dissolve rapidly after perforation of the stainless cladding; visual inspection of the fuel remaining in the basket in the latter parts of the run showed substantial amounts of uranium undissolved. The uranium and iron concentrations remained essentially constant after the start-up transient period.

Over a 32-hour period, uranium and iron concentrations were maintained at 9.7 ± 0.5 grams/liter and 8.6 ± 0.5 grams/liter, respectively. The excellent concentration control demonstrated during this run is more a measure of current and acid feed control than a predictor of actual fuel dissolution characteristics. The uranium pins dissolved were much larger than those in actual fuel (providing much less area for chemical dissolution) and were natural uranium rather than the uranium-fissium alloy in EBR-II fuel.

TABLE VII

CONCENTRATION PROFILES FOR SIMULATED FUEL DISSOLUTION

<u>Time(Hours)</u>	<u>Uranium (grams/liter)</u>	<u>Iron (grams/liter)</u>
0.0	0.00	0.00
0.5	0.02	5.1
2.5	0.51	6.9
4.5	3.7	6.6
6.5	6.1	6.6
8.5	10.2	5.7
10.5	11.1	5.6
12.5	9.5	6.1
14.5	9.4	6.2
16.5	9.2	6.6
18.5	9.1	6.2
20.5	10.0	6.3
22.5	10.0	6.3
24.5	9.7	6.2
26.5	10.0	5.9
28.5	9.9	5.8
30.5	9.3	6.0
32.5	8.9	6.3
34.5	9.4	5.9
36.5	10.0	6.1
38.5	10.0	6.0
40.5	9.4	5.9

FIGURE 2. Uranium and Iron Concentration Profiles for the Dissolution of Uranium and Steel Simulated Fuel

Analysis of dilute product gas (4 samples) showed an average hydrogen concentration of 0.3 mole percent, which is consistent with the previously obtained data (0.4%) from the EBR-II fuel dissolution.

1.4 Dissolution of Stainless Steel Rod in Aluminum Canisters

EBR-II fuel elements are packaged in aluminum canisters for storage in the ICPP basin. A trial dissolution of simulated fuel in aluminum containers was made in the pilot-plant dissolver. Both the canisters and the enclosed rods dissolved without difficulty. The packaging of EBR-II fuel should not have any significant effect on dissolver operation.

2. DISSOLUTION OF UNIRRADIATED EBR-II FUEL

2.1 EBR-II Fuel Description^[14]

Six EBR-II fuel assemblies were available for dissolution. The fuel used in this test contained natural and depleted uranium; fuel to be dissolved in the production unit will be 52.5% enriched. EBR-II fuel elements consist of 0.144-inch diameter pins, 13.5 inches long, contained in 0.18-inch diameter Type 304 stainless steel tubes. The tubes are approximately 18 inches long, including end fittings, and are spirally wrapped with 0.049-inch diameter wire. Each fuel element contains 65 grams of fuel meat, 0.7 grams of sodium, and 19 grams of stainless steel. The fuel meat is an alloy composed of 95 weight percent uranium and 5 percent fission. There are 91 fuel elements per fuel section, grouped in a hexagonal stainless steel tube. The fuel section is located in the center of a subassembly which includes blanket sections and end fixtures. A subassembly is 92 inches long and 2.29 inches across the flats.

The overall composition (wt%) of a subassembly with the blanket ends removed is:

Uranium	64.0%
Iron	22.6%
Chromium and Nickel	9.22%
Molybdenum	1.64%
Ruthenium	1.37%
Sodium	0.73%
Rhodium	0.21%
Palladium	0.14%
Zirconium	0.07%

2.2 Experimental Conditions

Steady-state operation of the dissolver was unattainable because of the limited amount (10^4 pounds) of unirradiated EBR-II fuel available for dissolution. The transient character of the run precluded any variation of experimental conditions. Six fuel elements were charged to the dissolver, three initially and the remaining three on demand. Operating conditions were held at the levels listed below, with continuous feed of fresh nitric acid and discharge of product solution.

Dissolver Exit Temperature:	150°F
Current:	3000 amps (1.2 amps/cm ²)
Feed Acid Concentration:	9.0 moles/liter
Exit Acid Concentration:	8.2 moles/liter
Acid Feed Rate:	130 liters/hour

2.3 Experimental Results

The EBR-II fuel dissolution was conducted in two steps, a fourteen-hour operating period, which was closely monitored and analyzed, and a thirty-six-hour heel-out period, during which residual fuel was completely dissolved. The run was interrupted five times for visual inspection of dissolving fuel assemblies. Fresh acid feed was started one hour after dissolution began, i.e., when solute concentrations had reached significant levels.

2.31 Visual Inspection of Dissolving Fuel

The recirculating electrolyte solution was withdrawn after 3, 5, 6, 8, and 13 hours of operation. Both the stainless steel hexagonal tube and the end support blocks dissolved readily, exposing the individual fuel elements for anodic dissolution.

Figures 3 and 4 show a partial fuel assembly, the support block, and hexagonal tube prior to dissolution. Figure 5 taken after 3 hours, shows approximately half of the hexagonal shell of the visible fuel assembly was dissolved, the individual elements had been opened, and uranium had been dissolved from the steel tubes. Two additional assemblies were charged at this point.

Figures 6 and 7 show the dissolution of the new fuel at hours 5 and 6, respectively. Figure 8 provides excellent detail of the fuel at 8 hours (5 hours after the second charge). Opened fuel elements, containing partially dissolved fuel meat and the steel pins forming the sodium reservoir (at the left), are visible.

Figures 9 and 10 show the fuel remaining after 13 hours of operation. Figure 11 shows an opened element with the fuel meat dissolved from the exposed area. Subsequent examination showed that the fuel meat and annular sodium had been leached one half-inch back from the opening.

Figure 12 shows arcing where a fuel element (at the sodium reservoir end) contacted the niobium basket.

2.32 Qualitative Observations of Dissolution

A visual record of the dissolution was made by use of closed circuit television and a TV tape recorder. The system operated smoothly, without any explosions or any unusual amount of gas evolution. During shutdowns, small pieces of sodium could be observed in the final stages of dissolution. Gas evolution at the end of the dissolver where the sodium reservoirs were located was more vigorous than elsewhere in the dissolver. Subsequent analysis of product solution showed that the sodium dissolved at a rate proportional to that of the uranium.

2.33 Product Solution Concentration Profiles

A complete listing of product solution analyses is given in Table VIII. Concentration-time profiles are plotted in Figure 13.

FIGURE 3. Partial EBR-II Fuel Assembly

FIGURE 4. Partial EBR-II Fuel Assembly

FIGURE 5. Dissolving Fuel After Three Hours of Dissolution

FIGURE 6. Additional Fuel Two Hours After Charge

FIGURE 7. Additional Fuel Three Hours After Charge

FIGURE 8. Additional Fuel Five Hours After Charge

FIGURE 9. Fuel Remaining Ten Hours After Charge

FIGURE 10. Detail of Fuel Remaining After Ten Hours of Dissolution

FIGURE 11. Close-up of a Dissolving Fuel Pin

FIGURE 12. Arcing Effect Where a Fuel Pin (at the Sodium Reservoir end) Contacted the Niobium Basket

TABLE VIII

PRODUCT SOLUTION CONCENTRATION PROFILES

Operating Hours	U g/l	Fe g/l	Na g/l	Mo g/l	Ru g/l	H ⁺ mol/l	%H ₂ in Undiluted Off-gas	Undissolved Solids g/l	Hydroclone Solids g/l	
									IN	OUT
0.00	0.00	0.00	0.00	0.00	0.00	9.00	0.00	0.00	0.00	0.00
0.50	0.16	2.42	0.036	0.02	0.002	8.56	1.34	0.21		
1.00	2.99	3.80								
1.5	8.90	3.52	0.16	0.255	0.017	8.38		0.28		
1.75	10.14						0.56			
2.00	10.75	3.40								
2.25	11.43								0.44	0.34
2.50	13.55									
2.75	14.12	3.50								
3.00	15.37	3.50					0.31			
3.75	15.86	3.80					1.18		0.60	0.55
4.50	14.90	4.22	0.21	0.436	0.073	8.20		0.63		
5.25	18.50									
5.50	18.40	5.21	0.27	0.518	0.058	8.08	1.04	0.75		
6.00	18.32	5.30					1.43			
6.75	17.00									
7.00	16.20									
7.50	15.80	6.22	0.25	0.456	0.077	8.16		0.79	0.68	0.61
7.75	15.50	6.10								
8.50	12.10	6.51	0.20	0.350	0.067	8.18		0.56	0.77	0.41
8.75	12.40	5.60					1.98	0.72		
9.50	12.20	5.90	0.19	0.357	0.057	8.20		0.46	0.57	0.53
9.75	11.60	5.80					3.07			
10.50	11.10	5.94	0.20	0.316	0.031	8.14		0.47	0.48	0.41
10.75	12.50	6.00					2.04			
11.50	13.50	5.98	0.23	0.354	0.065	8.16		0.63	0.74	0.65
11.75	13.40	5.80								
12.50	11.80	5.57	0.21	0.326	0.056	8.16		0.43	0.68	0.61
12.75	10.50	5.40								
13.50	8.30	5.29	0.18	0.247	0.046	8.34		0.33		
13.75	7.20	5.20						0.29		
14.00	6.40	5.00						0.26

FIGURE 13. Solute Concentration Profiles for the Dissolution of EBR-II Fuel in The Electrolytic Dissolver Pilot Plant

- a. Iron concentration, representing approximately 71% of the total stainless steel concentration, rose rapidly to 3.5 g/l during the first hour of operation and remained near that level for two hours. From the third to the seventh hour, iron concentration rose to a steady-state value of 6 grams/liter (~ 8.5 g/l stainless steel).

Iron concentration remained constant for about five hours, gradually dropping to 5.0 g/l during the last two hours of operation (possibly reflecting a decrease in current utilization due to a decrease in fuel inventory).

Steel concentration may be controlled within ± 0.5 g/l if a constant inventory of fuel is maintained. The current utilization during this run was 0.45 grams/ampere-hour. The fuel charge was maintained five inches below the liquid surface, resulting in current leakage directly from anode to cathode and thus, a lower current utilization than previously demonstrated. A similarly placed charge of stainless pipe was dissolved at 0.33 g/amp-hr as opposed to a typical value of 0.60 g/amp-hr.

- b. Uranium concentration, first detected after 30 minutes of operation, rose to a maximum level of 18.5 g/liter in a series of steps, subsequently declining in a similar manner to 6.5 g/liter at the end of the main operating period. The cyclic nature of the uranium concentration curve suggests six peaks, corresponding to the six fuel clusters opening by electrolytic dissolution, with subsequent rapid chemical dissolution of uranium.

Table IX lists the estimated times for uranium concentration maxima as a function of the position in the electrolytic dissolver. Based on the assumption that the maxima are attributable to dissolution of specific fuel elements, vertical position in the basket has a significant effect on the rate of electrolytic dissolution. The

effect observed is probably caused by current leakage, which is a direct function not of vertical position, but of an alternate current path through the electrolyte above the fuel elements. In this experiment, the dissolver was not fully loaded; an unobstructed current path existed between the anode and cathode.

TABLE IX

TIME DELAY BETWEEN CHARGING AND MAXIMUM
URANIUM CONCENTRATION AS A FUNCTION
OF INITIAL POSITION IN DISSOLVER

<u>Initial Position</u>	<u>Element Number</u>	<u>Estimated Time of U_{max}</u>
Bottom	1	2.75 hrs.
Middle	2	3.50 hrs.
Middle	4	3.50 hrs.
Middle	6	3.75 hrs.
Top	3	5.50 hrs.
Top	5	5.50 hrs.

The limited data from this run indicate that uranium concentration could fluctuate from 3 to 5 grams per liter in the pilot-plant scale of operation. In the plant system, the variations will be damped out by the larger acid volume.

- c. Molybdenum and sodium concentration profiles duplicate the uranium profile closely, indicating that both the annular sodium and the fuel meat dissolve simultaneously (i.e., the sodium is not leached out of the annulus preferentially). The close correspondence of sodium and uranium dissolution patterns suggest an effective system for monitoring uranium concentration, using a continuous instrumental analysis for the sodium.
- d. Ruthenium was only partially dissolved and contributes significantly to the undissolved solids; approximately 20% of the ruthenium dissolved.

- e. Hydrogen ion consumption was approximately 9.9 moles per 100 grams of stainless dissolved compared to 6.2 for straight stainless dissolution. The higher consumption rate for EBR-II fuel reflects acid consumption in the dissolution of uranium, sodium, etc.

2.34 Undissolved Solids

The dissolution of EBR-II fuel elements resulted in 3 weight percent of the fuel remaining undissolved. The undissolved solids contained 1.8 wt% uranium, representing a loss of 0.09 wt% of the total uranium charged.

The average suspended undissolved solids concentration was 0.47 grams/liter. During the "steady-state" portion of the run, (from 7.5 to 12.5 hours) solids concentration was 0.61 grams/liter. Analysis of these solids is shown in Table X.

TABLE X

UNDISSOLVED SOLIDS COMPOSITION, WEIGHT PERCENT

Operating Hours	Component				
	U	Fe	Ru	Mo	Si
8	1.88	7.70	26.7	7.66	
9	2.30	5.33	19.0		5.59
10	1.73	7.93	23.1	4.39	
11	1.10	12.15	25.0		4.13
12	2.39	11.01	19.4	4.77	
13	1.55	9.56	18.7		4.07

The major component of the undissolved solids is ruthenium, the least soluble metal of the fission solids. The undissolved solids from EBR-II dissolution are black and very finely divided; they settle from product solutions after two hours and are easily resuspended by a few swirls of a sample bottle.

2.35 Residual Solids

Three samples of solids were removed from the system after completion of the run.

1. Dark tar-like solids, removed from the niobium anode above the electrolyte level, contained 24 wt% uranium. Electrolyte which was splattered on the anode by vigorous gas evolution probably evaporated, leaving a residue similar in composition to the product solution.
2. Gray solids removed from the niobium anode were mainly iron and silicon and contained only 0.2 wt% uranium.
3. Gray solids trapped in the bottom of the hydroclone consisted almost entirely of stainless steel particles; only 0.06 wt% uranium was found in this material.

The analysis of each of these solids is shown in Table XI.

TABLE XI

RESIDUAL SOLIDS COMPOSITION FROM EDPP SYSTEM, WEIGHT PERCENT

	Component				
	U	Fe	Ru	Mo	Si
Tarry solids from dissolver top	23.60	7.53	0.12	0.23	0.12
Gray solids from dissolver anode	0.20	16.96	0.78	0.11	15.23
Hydroclone Solids	0.06	67.7	0.02	0.13	0.38

2.36 Gas Evolution

An average hydrogen concentration of 1.4 mole percent was present in the dissolver off-gas. The diluted off-gas contained only 0.4 mole percent hydrogen, an order of magnitude lower than the lower explosive limit of hydrogen in air.^[15]

III. PROJECTED PLANT OPERATING BEHAVIOR

Actual dissolution of irradiated EBR-II fuel in the plant dissolver will behave differently than pilot-unit operations. The higher acid feed rate and equipment volumes will change the dynamic behavior of the system. Potential criticality hazards require careful study so that safe operating conditions can be specified.

To meet this need, a mathematical model simulating the electrolytic dissolver plant was prepared. The major results of that simulation have been described in IN-1375, "Simulation of the Electrolytic Dissolver Plant for Nuclear Safety Review".^[16]

The summary of IN-1375 states,

"The general operating characteristics of the dissolver and recirculating acid loop are those of a well-mixed tank. The concentration variations among loop components may be neglected. Transient behavior is a function of the uranium dissolution rate, recirculating acid volume, and acid feed rate. At typical operating conditions (3000 amps dissolver current), approximately seven hours would be required to reach a new steady state. Short-term concentration changes should be less than 2 grams per liter. The uranium concentration cannot exceed:

$$\left[U_o + \frac{5620 N_e}{V_{ra}} \right]$$

U_o = Initial uranium concentration

N_e = Number of complete fuel sections
in dissolver

V_{ra} = Volume of recirculating acid

Simulated operations of the dissolver were conducted at uranium dissolution rates from 45 grams per minute (the mean uranium dissolution rate in the pilot-plant dissolution of EBR-II fuel) to 1000 grams per minute with 1 to 5 fuel sections charged. Maximum concentrations occurred (start-up

simulation) when the fuel charge had completely dissolved. Maxima were predicted for the expected operating range as a function of dissolution rate, fuel charge, and acid feed rate. Inherent safety factors were demonstrated for various fuel charges (e.g., if 3 fuel sections were charged and 5 liters of acid per minute were fed, a dissolution rate triple the expected rate could be tolerated with a 16 grams/liter concentration limit for total uranium).

Recommended operating procedures for plant start-up were defined:

1. Number of fuel sections charged $\leq 0.23 U_L$
 $(U_L = U_{235} \text{ concentration limit})$
2. Fuel charge rate $\leq 2800/Ie$ $\left(\begin{array}{l} I = \text{current, amperes} \\ e = \text{current utilization, grams/amp-hour} \end{array} \right)$
3. Acid feed rate = Ie/S $(S = \text{desired stainless steel concentration, grams/liter}).$

Subsequent study of system behavior was made to answer specific questions raised by the plant simulation. Various fuel charging sequences and transient operations were studied.

Dissolution of EBR-II Fuel at various assumed rates and with different fuel inventories was simulated. While the uranium concentration rose to new steady-state levels (dissolution rate/acid feed rate), no short-term deviations from new fuel element additions were predicted.

Fuel inventories of 3, 5, and 10 elements could, at most, raise the uranium concentration by 24, 40, and 80 grams per liter, respectively, if completely dissolved in the recirculating acid without fresh feed. If recirculation were stopped and the total uranium inventory dissolved in the dissolver body, local concentration rises of 170, 280, and 560 grams/liter are theoretically possible.

The nature of the dissolving fuel elements suggests that such concentration transients are extremely unlikely, if not impossible. Each fuel assembly is made up of 91 steel-jacketed fuel pins. The jackets must be dissolved electrolytically before uranium dissolution can start. Since the jackets are not generally dissolved until they are exposed (by dissolution of the interposed pins) to the cathode, the actual amount of uranium which could be dissolved during a shut-down is probably less than 20% of the total charge. The mode of EBR-II fuel dissolution should be viewed as that of 91N elements rather than N elements.

The problems associated with intermittent operation of the electrolytic dissolver were studied by simulation on a digital computer (using a special simulation language, CSMP-360; Appendix B).

During the time that acid recirculation is off, uranium could be dissolved from the fuel pins which had been opened electrolytically. Upon resumption of recirculation, the additional dissolved uranium would be distributed through the recycle loop (assumed to remain at 25 grams U/liter during shut-down). The concentration transient in the surge tank is of major interest because of potential criticality problems associated with its geometry. The objective of the simulation is to predict the range of uranium concentration maxima in the surge tank for the full range of uranium concentrations theoretically attainable in the dissolver during shut-down.

The uranium concentrations in the dissolver, surge tank, and heat exchanger are described by three simultaneous differential equations:

System Equations

$$C_{UD} = \frac{F}{V_D} (C_{UO} - C_{UD})$$

$$C_{UST} = \frac{F}{V_{ST}} (C_{UD} - C_{UST})$$

Where:

C_{UD} = U concentration in dissolver

F = Recycle rate

V_D = Volume of dissolver

C_{US} = U concentration in surge tank

V_{ST} = Volume of surge tank

System Equations

$$C_{UH} = \frac{F}{V_H} (C_{UST} - C_{UH})$$

$$V_H = 570 - V_{ST}$$

$$C_{UO} = C_{UH} (60F - P)/60F$$

Where:

C_{UH} = U concentration in heat exchanger

V_H = Volume in heat exchanger

C_{UO} = U concentration after dilution
by fresh acid

P = Fresh acid feed rate

The results of the simulation are listed in Table XII. During the brief transient period, the introduction of fresh acid did not cause significant concentration changes; over a longer period, fresh acid feed would effect a return to the original steady-state uranium concentration.

Figure 14 is a plot of typical uranium concentration transients for the three system elements (dissolver, surge tank, and heat exchanger). The concentration maximum in the surge tank occurs when $C_{UD} = C_{UST}$. The time at which the maximum occurs decreases as the acid recycle rate increases; the time increases with increasing surge tank volume.

Figure 15 shows the concentration maximum in the surge tank as a function of initial uranium concentration in the dissolver for three different surge-tank liquid volumes.

Figure 16 shows the relationship between the concentration maxima and surge-tank liquid volume for three initial U concentrations in the dissolver.

The assumption that all of the uranium in the dissolver would dissolve during a shut-down is extremely conservative. However, the assumption of perfect mixing in each of the components of the recycle loop is somewhat liberal. If the dissolver current is turned on when acid recirculation is resumed, the resultant mixing caused by gas evolution should cause the concentraion transients to behave essentially as predicted.

TABLE XII

PREDICTED MAXIMUM URANIUM CONCENTRATIONS IN SURGE TANK
DURING STARTUP AFTER A SHUTDOWN

Run Number	Assumed Initial Uranium Concen. in Dissolver (grams/ liter)	Acid Recirc. Rate (liters/ min)	Surge Tank Volume (liters)	Acid Feed Rate (liters/ hour)	Maximum Uranium Concen. in Surge Tank (grams/ liter)	Time of Maximum After Start-up (minutes)
1	75	57	120	0	44.4	2.3
2	125	57	120	0	63.8	2.3
3	75	114	120	0	44.4	1.1
4	125	114	120	0	63.8	1.1
5	75	57	120	50	44.4	2.3
6	75	57	250	0	38.2	3.4
7	50	57	120	0	34.7	2.3
8	100	57	120	0	54.1	2.3
9	150	57	120	0	73.5	2.3
10	175	57	120	0	83.2	2.3
11	200	57	120	0	92.9	2.3
12	75	57	150	0	42.4	2.6
13	75	57	180	0	40.8	2.9
14	75	57	210	0	39.5	3.1
15	100	57	250	0	44.7	3.4
16	200	57	250	0	71.1	3.4
17	300	57	250	0	97.4	3.4
18	400	57	250	0	123.7	3.4
19	300	57	120	0	131.8	2.3
20	400	57	120	0	170.6	2.3

FIGURE 14. Uranium Concentration Transients for Elements of the Recirculation System at 57 liters/min Recirculation, No Feed Acid, and a 120 Liter Surge Tank

FIGURE 15. Uranium Concentration Maxima in the Surge Tank as a Function of Initial Concentration in the Dissolver
 Acid Recirculation Rate = 57 L/Hr, Acid Feed Rate = Zero
 Initial U Conc. in Surge Tank and Heat Exchanger = 25 grams U/Liter

FIGURE 16. Maximum Uranium Concentrations in Surge Tank as a Function of Surge Tank Volume for Specified Initial Concentrations in the Dissolver

Where: Acid Recirculation = 57 L/Hr

Acid Feed Rate = 0

Initial Concentration in Surge Tank and
Heat Exchanger = 25 g/L

IV. CONCLUSIONS

1. EBR-II fuel can be dissolved rapidly and safely in the plant electrolytic dissolver system. The observed current utilization in the pilot-plant was 0.45 grams of stainless steel per ampere-hour. The overall fuel dissolution rate was 1.35 grams/amp-hr. Current utilization of 0.60 grams per ampere-hour (equivalent to 1.8 grams/amp-hr total) can be expected in a fully loaded dissolver.
2. After the fuel meat was exposed, the uranium dissolved more rapidly than the stainless cladding. No criticality hazard can be expected from the accumulation of undissolved uranium in the dissolver.
3. Uranium losses in the undissolved solids were less than 0.09% of the total uranium charged.
4. Hydrogen evolution presents no safety hazard; the hydrogen concentration in the diluted effluent (0.4%) is only one-tenth the explosive limit in air.
5. Sodium dissolution rates were proportional to those of uranium; no fire or explosive dissolution was observed.
6. Three percent of the total fuel charged remained undissolved. These finely divided, easily suspended particles were mainly stainless steel and fission products.
7. The two critical control variables for the system are dissolver current and the acid feed rate. The acid concentration of the product solution can be maintained anywhere between 6M and 9M (HNO_3) without affecting the dissolution rate. The dissolver exit temperature may be maintained between 115°F and 145°F without significant effect on the dissolution.
8. The addition of gelatin did not cause any significant change in the amount of undissolved solids produced. Earlier experimentation^[10]

had shown a reduction in the sludge coating of surfaces when gel concentrations of 100 to 200 PPM were used.

9. The use of a conductivity cell as a concentration monitor for uranium was not a success. Changes in uranium concentration cause very small changes in conductivity compared to other variables (stainless steel concentration, temperature). Alternate means for monitoring uranium concentration should be developed.
10. Theoretically possible uranium concentrations (predicted by computer simulation), which could occur during transient periods, should be limited by control of fuel charging sequences and acid feed rate; the use of a soluble poison (such as cadmium) is advisable.

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APPENDIX A

The following computer program was prepared to analyze the factorial experimental design used in this study. Use of the program requires specification of:

1. NIV Number of Independent Variables
2. NDV Number of Dependent Variables
3. NR Number of Responses
4. NLEV Number of Levels (of IV)
5. The matrix of factor levels
6. The matrix of responses

The output of the program lists the factor levels, associated response, the mean response for each factor level, and a value of the t-statistic for the difference between the means.

$$t = \frac{\bar{X}_1 - \bar{X}_2}{S_p \sqrt{\frac{1}{N_1} + \frac{1}{N_2}}}$$

The value of the t-statistic may then be compared to a table of critical t-values (provided for various confidence levels and degrees of freedom). If the calculated value exceeds the critical value for a given confidence level, the effect (response) is considered significant.

```

PROGRAM T
DIMENSION IX(20,40),Y(20,40),AVE(9),ABK(9),SDEV(9),SDFA(9)
READ 70, NIV,NDV,NR,NLEV
70 FORMAT (4I5)
DO55 I=1,NDV
55 READ 101,(Y(I,J),J=1,NR)
101 FORMAT (8F10.2)
DO10 I=1,NIV
10 READ 102,(IX(I,J),J=1,NR)
102 FORMAT (40I1)
DO9 I=1,NDV
DO9 J=1,NIV
PRINT 201,I,J,(Y(I,K),IX(J,K),K=1,NR)
201 FORMAT (1H1,20X,17H DATA CORRELATION/1H0,15X,2HY(12,1H),16X2HX(
1 I2,1H0 // (E23.5,I15))
PRINT 202
202 FORMAT (1H0/1H012X9HY-AVERAGE,9X,13HNO. OF POINTS/)
DO6 K=1,NLEV
SUMSQ=0.
SUM = 0.
AK = 0.
DO5 L=1,NR
IF(IX(J,L)-K)5,4,5
4 SUM=SUM+Y(I,L)
SUMSQ = Y(I,L)**2+SUMSQ
AK=AK+1.
5. CONTINUE
AVE(K) =SUM/AK
ABK(K)=AK
SDFA(K)=SUMSQ-SUM**2/AK
SDEV(K)=SQRTF(SDFA(K)/(AK-1.))
6 CONTINUE
NLEV1=NLEV-1
DO7 K=1,NLEV
IV=ABK(K)
7 PRINT 203,K,AVE(K),IV
203 FORMAT(3X5HLEVEL,I2,E14.5,I15)
PRINT 204
204 FORMAT (1H0/1H013X8HT-VALUES/)
DO8 K=1,NLEV1
NLEV2 =K+1
DO8 M=NLEV2,NLEV
SP= (SDFA(K)+SDFA(M))/(ABK(K)+ABK(M)-2.)
TV=ABSF(AVE(K)-AVE(M))/SQRTF(SP*(ABK(K)+ABK(M))/(ABK(K)*ABK(M)))
8 PRINT 205,K,M,TV
205 FORMAT(6X1HT2I1,E15.5)
9 CONTINUE
STOP
END

```

* Prepared by G. W. Hogg

APPENDIX B

The simulation program for the differential equations describing the uranium concentration in the recirculation loop requires only the Job Control Language information (which summons CSMP-360, a higher order programming language) and a FORTRAN statement of the system equations.

```

      ****CONTINUOUS SYSTEM MODELING PROGRAM****

      ***PROBLEM INPUT STATEMENTS***

      TITLE      ELECTROLYTIC DISSOLVER TRANSIENT
      INITIAL
                VH=570.-VST
RUN 1  PARAMETER CUDIC=75.
      PARAMETER F=57.
      PARAMETER VST=120.
      PARAMETER P=0.
      DYNAMIC
                CUO=CUH*((60.*F-P)/(60.*F+P))
                CUDDOT=(CUO-CUD)*F/130.
                CUSDOT=(CUD-CUS)*F/VST
                CUHDOT=(CUS-CUH)*F/VH
                CUD=INTGRL(CUDIC,CUDDOT)
                CUH=INTGRL(25.,CUHDOT)
                CUS=INTGRL(25.,CUSDOT)
      TIMER      FINTIM=15.,OUTDEL=0.1,PRDEL=0.1,DELT=0.01
      PRINT      CUS,CUD,CUH
      PRTPLT     CUS
      END
RUN 2  PARAMETER CUDIC=125.
CHANGE END
RUN 3  PARAMETER CUDIC=75.,F=114.
CHANGE END
RUN 4  PARAMETER CUDIC=125.
CHANGE END
RUN 5  PARAMETER CUDIC=75.,F=57.,VST=120.,P=0.8333
CHANGE END
RUN 6  PARAMETER VST=250.,P=0.0
CHANGE END
      STOP

```

APPENDIX C

EVOP DATA

Run	Acid Conc.	Pipe Size	Average Current, Units Amperes	Dissolver Resistance, Units, Ohms	Dissolution Rate, grams/hour	Current Utilization, g/amp-hr
1-1	0	0	3093	3.5	1285	0.42
1-2	-	-	2936	4.2	1477	0.50
1-3	-	+	2935	4.4	1207	0.41
1-4	+	+	2965	4.6	1640	0.55
1-5	+	-	2685	5.1	1455	0.54
2-1	0	0	2393	5.6	1138	0.48
2-2	-	-	2374	6.7	1358	0.57
2-3	-	+	2388	6.0	1220	0.51
2-4	+	+	2350	6.6	1160	0.49
2-5	+	-	2388	6.3	1345	0.56
3-1	0	0	2193	6.5	1228	0.56
3-2	-	-	2441	6.2	1148	0.47
3-3	-	+	2362	6.7	1275	0.54
3-4	+	+	2363	6.7	1366	0.58
3-5	+	-	2349	6.0	1245	0.53
4-1	0	0	2004	7.0	928	0.46
4-2	-	-	1921	7.8	977	0.51
4-3	-	+	1934	7.5	1173	0.61
4-4	+	+	1943	6.7	1124	0.58
4-5	+	-	1969	7.0	1062	0.54
SHUTDOWN AND CAUSTIC FLUSH TO CLEAN DISSOLVER SYSTEM						
5-1	0	0	2942	4.7	2003	0.68
5-2	-	-	2966	5.0	2048	0.69
5-3	-	+	2830	5.9	1795	0.55
5-4	+	+	2663	6.3	1825	0.44
5-5	+	-	2260	7.9	1189	0.53
6-1	0	0	2590	7.5	1615	0.62
6-2	-	-	2235	8.5	1503	0.67
6-3	-	+	2060	9.2	1125	0.55
6-4	+	+	2190	8.8	1057	0.48
6-5	+	-	2040	9.4	1062	0.52
7-1	0	0	1915	9.9	1005	0.50
7-2	-	-	1800	10.7	978	0.54

APPENDIX D

GLOSSARY OF TERMS

1. Valve Metal - will act as a cathode but not as anode because of a non-conducting oxide layer in the anodic state. This rectifying action will occur until a break-down potential, typical of the particular metal, is exceeded. A valve metal will act as a current path to another metal; the oxide has a high conductivity in metal-to-metal contact.
2. Soluble Neutron Poison - is a substance which, in a soluble form, will absorb neutrons unproductively, thus decreasing the reactivity of a system.
3. EBR-II Fuel - is the stainless steel-clad, sodium-bonded fuel used in the Experimental Breeder Reactor II.
4. Fuel Section - is the portion of a fuel subassembly that contains the fissile material.
5. Blanket Section - is the portion of a subassembly containing fertile material such as U-238.
6. Fission - is a synthesized mixture of molybdenum, ruthenium, rhodium, palladium, niobium, and zirconium which represents the equilibrium composition of the fission products of irradiated EBR-II fuel.
7. Fission Products - are the fission product isotopes of the above-named elements.
8. Hydroclone - a liquid-solid cyclone separator.
9. Current Efficiency - is the percentage of the theoretical current utilization which is achieved. (The quotient of actual current utilization and theoretical current utilization.)

10. Theoretical Current Utilization - is the mass of material dissolved by a current of one ampere in one hour as predicted by Faraday's Law.
11. Solution Contact - is the principle applied to the problem of current distribution across the dissolver. A small solution gap is maintained between the electrodes and the fuel elements, thus eliminating the non-uniform current distribution of metal-to-electrode contact systems.
12. Sodium-Bonded - EBR-II fuel consists of an enriched uranium-fissium alloy bonded to stainless steel 304 by sodium for heat transfer.
13. Dissolver Loading - the extent to which the dissolver is filled with dissolving fuel elements.
14. Current Density - is the current passing through the dissolver per unit area of electrode (anode).
15. Dissolution Rate - the rate of solution of fuel, expressed as weight dissolved per unit time or as weight dissolver per unit time per unit area of anode.
16. Sludge - is the finely-divided material which remains undissolved in the electrolytic dissolver, consisting of stainless steel, niobium carbide, and silica.
17. Fuel Volume Fraction - that fraction of a fuel element-water cell occupied by the metallic fuel element constituents.