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SUBJECT: Darex Process: Batch Dissolution and Chloride
Removal Steps for Yankee Atomic Fuel

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FROM: J. H. Goode
J. R. Flanary

NOTICE

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ABSTRACT

In laboratory scale experiments dilution of a Darex dissolver product from the integral dissolution of Yankee Atomic fuel specimens with at least an equal volume of 15.8 M HNO_3 , followed by distillation, produced a solvent extraction feed containing less than 50 ppm of chloride. Properly designed chloride stripping and nitric acid rectification columns are capable of conserving acids for recycle to the dissolver and chloride removal still. In initial experiments about 87% of the nitric acid and 36% of the hydrochloric acid were recovered.

1.0 INTRODUCTION

Conversion of Yankee Atomic fuel pins to an aqueous nitrate solution by a batch-operated Darex system assumes integral dissolution of the 304L stainless steel cladding and the sintered UO_2 pellets of the core in dilute aqua regia, followed by distillation to remove chloride and water. Nitric acid is also removed in excess of that required for solvent extraction. The reagents removed by distillation should be recycled to achieve economical operation. Clark¹⁶ found that the addition of a 30% excess of oxygen over that required to oxidize NO to NO_2 in the dissolver off gas gave nitrogen and chloride recoveries of greater than 95% when adequate condensing surface was available.

Since a large capital investment in processing equipment is not desired at this time, the work presented in this report was undertaken to determine the simplest batch equipment required for (1) chloride removal by evaporation without stripping or rectification equipment and (2) chloride removal by a simple batch dissolver-stripper-rectifier system where maximum conservation of nitric acid is obtained. A third study is now underway in modified equipment to determine maximum conservation of both nitric and hydrochloric acids. These studies will produce the data required to compare the value of the acid recovered with the cost of the extra capital equipment required for acid recovery.

This report represents an interim summary of work on a laboratory scale and does not represent a complete evaluation. A final ORNL report will be issued later to summarize all batch Darex dissolution and feed preparation work.

Appendixes I and II show reactor and fuel element data and a proposed Purex-type of solvent extraction flowsheet for the recovery of uranium and plutonium from the Darex product solutions prepared from spent Yankee Atomic fuel.

2.0 SUMMARY

Batch dissolution of stainless steel samples in dilute aqua regia with a simple updraft condenser showed a hydrogen ion consumption of 4 mols per

mol of stainless steel and a chloride consumption of 0.2 mols per mol. A minimum concentration of 2 M HCl-5 M HNO₃ was required in these experiments for complete dissolution of the metal when a stainless steel loading of 75 g/l was desired in the dissolver product. Design criteria were later changed to stainless steel and uranium loadings of 33 and 120 g/l, respectively. Dissolution is satisfactory using 1.5 M HCl-5 M HNO₃ when the acid and chloride values are conserved with an adequate downdraft condenser on the dissolver.

Simple batch buildown of the dissolver products from high stainless steel-uranium loadings (75 g/l SS and 180 g/l U) and low stainless steel-uranium loadings (33 g/l SS and 120 g/l U) removed only 50% of the chloride from the solutions. Satisfactory chloride and acid removal was obtained by evaporating the dissolver solution to 1/2 volume, adding a volume of 15.8 M HNO₃ equal to the original dissolver solution volume, and evaporation to 126°C. Dilution of the product with water produced a solvent extraction feed containing less than 50 ppm of chloride.

Preliminary experiments with a stripper-rectifier column attached to the dissolver indicated that satisfactory chloride removal and acid recoveries of greater than 87% of the nitric acid and 36% of the hydrochloric acid can be achieved in simple batch equipment. The overhead condensate containing dilute, i.e., 0.75 M HCl and 6 M HNO₃, acids was butted with nitric and hydrochloric acids and recycled to the dissolver. The concentrated nitric acid recovered in the rectifier bottoms was reused in the next batch evaporation-distillation. A loop-type batch dissolution-feed preparation flowsheet is proposed.

3.0 BATCH DISSOLUTION EXPERIMENTS

Experiments were made to simulate dissolution of the stainless steel cladding of Yankee Atomic fuel elements. Specimens of 304L stainless steel were dissolved in a heated Pyrex flask equipped with an updraft reflux condenser which was vented to atmosphere. The volumes of dilute aqua regia used were calculated to yield a 50 g/l solution.* It was found that passivation of the stainless steel occurred after 70-80% had dissolved when the initial dissolvents were 5 M HNO₃ and from 1.5 to 1.7 M HCl. The reaction went to completion when 2 M HCl-5 M HNO₃ was used. Analysis of the dissolvent showed that about 4 mols of acid, i.e., hydrogen ion, per mole of stainless steel was consumed during the dissolution in this equipment with a loss from the system of 0.2 mole of chloride per mole of stainless steel dissolved (Table 3.1). Other data on the dissolution of stainless steel have been presented by Hyman,^{9,43} Kitts,³⁴ Perona,⁸ and others.^{1-18, 20-22, 25-43}

*Design criteria later changed to 33 g/l stainless steel.

Table 3.1 Dissolution of 304L Stainless Steel in Aqua Regia

Time: 20 min
 SS wt: ca. 12 g

Temp: 80-100°C
 Dissolvent: 2 M HCl-4.7 M HNO₃

Expt. No.	Sample	Volume ml	Total H ⁺		Cl ⁻		SS	
			M	moles	M	moles	M	moles
3	Start	165	6.76	1.110	2.06	0.34		0.207
	End	160	1.72	0.275	1.82	0.29	1.30	0.207
4	Start	170	6.81	1.160	1.95	0.33		0.215
	End	150	1.57	0.236	1.75	0.30	1.39	0.209

4.0 BATCH BOLDDOWN EXPERIMENTS

4.1 Distillation without Oxidants. The solutions from the stainless steel dissolvings in Table 3.1 were adjusted with uranyl nitrate to simulate the total dissolution of Yankee Atomic fuel elements. Experiments were performed using these and other simulated dissolver products to determine whether the nitrate ion associated with the uranyl nitrate and the remaining hydrogen ion in the solutions would remove the chloride by oxidation during a simple evaporation or distillation.

Less than 50% of the chloride could be removed from the dissolver product by evaporation of the solutions to temperatures between 125 and 140°C. Evaporation of high content stainless steel-uranium solutions, containing 1.3 M H⁺, 1.1 M Cl⁻, 178 g U/l, and 44 g SS/l, to 126°C and holding at temperature for 10 minutes removed less than 15% of the chloride. Denitration and frothing occurs at 126°C. Evaporation of a typical low content stainless steel-uranium dissolver product solution containing 2.55 M H⁺, 0.87 M Cl⁻, 33 g/l SS and 120 g/l U to a black froth at 140°C, and dilution back to the original volume with 3 M HNO₃, showed that 50% of the chloride had been removed. In experiments of this type all heat must be applied below the surface of the liquid in order to prevent formation of insoluble oxides on the walls of the vessel.

Figure 4.1 shows the concentrations of H⁺ and Cl⁻ in the distillate during a simple evaporation to 126°C of a high content stainless steel-uranium dissolver product. It can be seen that a volume reduction of approximately 25% may be made to remove excess water with essentially no loss of nitric or hydrochloric acids.

4.2 Chemical Oxidants. Two experiments showed that the addition of an arbitrary 40 g/l of MnO₂ and K₂Cr₂O₇ to high content stainless steel-uranium solutions followed by evaporation to 125°C, dilution to

Figure 4.1 Distillate Composition During Batch Chloride Removal Step by Evaporation.

the original volume, and filtration gave chloride removals of 38 and 73%, respectively. The KMnO_4 reacted at room temperature evolving chlorine and oxidized the chromium to give a brown-colored solution.

4.3 Air and Steam Oxidants. The removal of chloride from high content stainless-uranium dissolver solutions was not enhanced by air sparging during evaporation to 126°C . Results of two such tests showed that an average of 87% of the chloride remained with the still residue; of the 13% removed, 9% was in the condensate and 4% was lost by oxidation.

Sparging steam into the still during the evaporation removed 22% of the chloride, 14% in the condensate and 8% by oxidation. In this test, a 40% reduction was made in the still volume by evaporation before the steam was introduced. This condensate contained 0.28 M H^+ and 0.06 M Cl^- . Steam, amounting to approximately $1/4$ of the original volume of feed, was then passed through the still and collected as condensate containing 1.4 M H^+ and 0.21 M Cl^- . The residue was then heated to 126°C and diluted back to the original volume with 2 M HNO_3 yielding a solution which analyzed 1.64 M H^+ , 0.98 M Cl^- , 44 g SS/l , and 182 g U/l .

4.4 Nitric Acid Oxidants. Figure 4.2 shows the effect on chloride removal of adding 15.8 M HNO_3 to the low stainless-uranium dissolver product and evaporating the solution to 126°C . The still bottoms were then diluted to the original volume with water to simulate solvent extraction feed. It may be seen that chloride removal is nearly proportional to the initial acidity of the still over the range of 2 M to 7 M . Chloride removal from 7 M still feed was about 95%, but the solvent extraction feed contained 0.03 M Cl (or 1000 ppm). The specification for chloride in solvent extraction is 30 ppm .

A sample of the still bottoms at 126°C in an experiment in which the initial acidity was about 4 M , analyzed 3.39 M H^+ , 1.25 M Cl^- , 125 g SS/l and 313 g U/l ; specific gravity was 2.21 g/cc .

Other experiments were performed in which 15.8 M HNO_3 was added semi-continuously to low stainless-uranium dissolver products in order to maintain high acidities in the still. In these experiments the dissolver solutions were evaporated to $1/2$ volume to remove dilute nitric and hydrochloric acids and 0.1 initial volume aliquots of 15.8 M HNO_3 were then added and the mixtures distilled successively. The residue was finally re-evaporated to 126°C . The experiments showed that:

1. An average of 21.7 and 11.7% of the H^+ and Cl^- were found in the distillate collected during the initial 50% volume reduction.
2. The amount of chloride in the still bottoms was inversely proportional to the volume of 70% HNO_3 added and distilled (Fig. 4.3).
3. The amount of chloride volatilized appeared to be constant (average 36.2%) regardless of the volume of 70% HNO_3 added, up to 0.8 volume.

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Figure 4.2 Effect of Initial Acidity of Still Feed on Chloride Removal in Batch Distillations - Simulated Low Stainless-Uranium Dissolver Products Butted with HNO_3

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Figure 4.3 Batch Chloride Removal by Distillation

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4. An equal volume of 70% HNO_3 reduced the chloride concentration of extraction feed prepared from still bottoms to 0.001 M, (36 ppm) which is nearly within specifications of 30 ppm. This indicates that solution must be at least 9.0 M in HNO_3 before the oxidation rate of chloride and the relative volatility of hydrochloric acid are high enough to produce the desired chloride removal. This is in agreement with the results of Reid et al.²³

On the basis of these results, an equal volume of 15.8 M HNO_3 must be added to the dissolver product before evaporation-distillation³ to remove chloride from Yankee Atomic dissolver solutions by this particular flowsheet. It appears that adjustment of dissolver solutions to about 9 M acid is the requirement for the general case; this value will change, of course, with changes in salt content. Since a large volume of acid is required a rectification and chloride stripping column should be added to the evaporator-still to conserve reagents.

5.0 BATCH STRIPPING AND RECTIFICATION FOR CHLORIDE REMOVAL

Figure 5.1 shows one version of a possible batch-operated dissolution and chloride removal system for use with Yankee Atomic fuels, as suggested originally by W. H. Lewis and others.⁴⁴ This system uses a rectifier-stripper column to recover concentrated nitric and hydrochloric acids as a top product. Several experiments have shown that it is not possible to separate pure hydrochloric acid from nitric acid or pure water from a mixture of nitric and hydrochloric acids.¹⁷

Initially the dissolver is charged with the stainless-clad UO_2 fuel and an integral dissolution performed with dilute aqua regia. The dilute acid overhead from the rectifier may be saved or discarded as desired; it may prove advantageous at this point to evaporate about 25% of the dissolver product to remove water and dilute acid in order to simplify the next stripping-rectification step. Following the dissolution, an equal volume of recovered 15.8 M HNO_3 is added from the nitric acid oxidizer hold-tank to the dissolver product, and the two volumes of combined solutions distilled to 1/4 of the original volume of dissolver product. The vapors are fed to the rectification-chloride stripping column, and the 1 volume of 15.8 M HNO_3 that had been added to the still is recovered in the reboiler free of chloride. The 3/4 volume of mixed acids from the dissolver product is recovered overhead as condensate suitable after adjustment of acidity for recycle to the next batch dissolution. Water is then added to the 1/4 volume residue in the still to bring it to the original 1 volume of solvent extraction feed.

The distillation-oxidation procedure not only removes chloride, but serves to remove the nitric acid in excess of that required for extraction feed from the dissolver product. The high temperature (126°C) and acidities reached in the still also serve to dehydrate any emulsion-forming silica that might be in the fuel.

Figure 5.1 Batch Darex Process for Yankee Atomic Fuel Dissolver, Chloride Removal and Acid Recovery System.

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In order to test this scheme, laboratory scale feed adjustment-chloride removal experiments were made using a Pyrex feed adjustment (FAT) tank (left), nitric acid reboiler (bottom), and 14" chloride stripping (lower) and 14" nitric rectification (upper) column, and packed condenser and condensate take-off (right) (Fig. 5.2). The equipment did not permit initial boil-off of excess water from the dissolution as proposed above. Consequently, these experiments were performed by adding one volume of concentrated acid directly to the dissolver product.

The system was operated by charging 1 volume of simulated Yankee Atomic dissolver product, containing 33 g SS/l, 125 g U/l, 3 M H^+ , and ca. 1 M Cl^- and 1 volume of 15.8 M HNO_3 to the FAT tank, and evaporating to 1/4 volume, which is reached at about 126°C. The vapors were fed to the mid-point of a glass helices-packed column having a rectification section above and stripping section below the feed point. Boiling 15.8 M HNO_3 vapor was used to strip chloride from the vapors refluxing above the reboiler.

The one volume of nitric acid that had been added to the FAT tank was recovered in the reboiler, slightly diluted by water from the feed, and the 3/4 volume of mixed HNO_3 -HCl distilled from the dissolver solution was recovered as condensate. The 1/4 volume left in the FAT tank was diluted to its original volume with water to make a solvent extraction feed. Using V/L ratios of 0.61, 0.70, and 1.0 in the chloride stripping section, as measured by the boil-up rates of the FAT tank and reboiler and condensate take-off rate, the solvent extraction feeds prepared from the FAT tank products averaged 51 ppm of chloride, the nitric acid in the reboiler contained about 10 ppm, and the composite condensate contained about 0.75 M HCl and 6.0 M HNO_3 . About 64% of the total chloride, based on 1.5 M HCl-5 M HNO_3 dissolvent, was lost as non-condensable gases due to the inefficiency of the packed condenser used in these experiments. Work by Kitts, Perona, and Clark^{16,17} has shown that recoveries of greater than 95% are possible in properly designed equipment. Further tests in modified equipment are underway to study conservation of chloride.

Table 5.1 presents the complete data from the tests. It may be seen that some of the water in the original dissolver product ended up in the reboiler, diluting the acid, and that after a number of cycles the chloride-stripping efficiency of the reboiler acid would be lowered. This was due, however, to equipment design, number of available plates in the columns, and dilution of the reboiler acid through column drainage on completion of the runs. Runs 5-8 show the effect of recycling 1 volume of acid from the reboiler through the FAT tank, to act as oxidant, and back to the reboiler for 4 cycles. The results show that satisfactory solvent extraction feed may be made by distillation, and that with better equipment the reagents may be recycled for more efficient conservation of materials. Future experiments will include water-removal steps, material balances on the dissolution, feed preparation, and reagent recycles.

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Fig. 5.2 - Laboratory Scale Distillation Apparatus

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Table 5.1 Batch Chlorine Removal by HNO_3 -Distillation of
Simulated Yankee Atomic Dissolver Product

Experiment No.	2	3	4	5	6	7	8
Boilup Rates (ml/min)							
FAT Tank	5.8	5.8	5.8	5.8	5.8	1.3	1.3
Reboiler	7.0	7.0	7.0	11.4	11.4	10.9	10.9
Condensate							
Removal (ml/min)	1.3	1.3	1.3	1.5	1.5	1.3	1.3
Cl stripper V/L	0.61	0.61	0.61	0.70	0.70	1.00	1.00
Still Feed: *							
Vol, ml	500	500	500	500	500	500	500
M H^+	9.38	9.38	9.54	9.88	9.99	9.40	9.90
M Cl^-	0.39	0.39	0.50	0.48	0.44	0.45	0.43
Reboiler Feed:							
Vol, ml	930	1000	1000	1000	1000	1000	1000
M H^+	15.8	15.8	15.8	15.5	15.5	15.1	15.0
ppm Cl^-	0	0	0	6	16	10	10
Reboiler Prod:							
Vol, ml	1180	1250	1260	1260	1260	1180	1240
M H^+	15.2	15.0	15.3	15.3	15.0	15.0	14.9
ppm Cl^-	18	6	8	11	22	10	34
Extraction Feed: **							
Vol, ml	250	250	250	250	250	250	250
M H^+	1.74	1.7	1.37	1.14	3.92	1.72	1.91
ppm Cl^-	18	75	85	39	60	80	45
Condensate:							
Vol, ml	180	180	180	180	180	180	180
M H^+	6.59	6.36	7.67	8.17	4.25	5.16	4.56
M Cl^-	0.54	0.67	0.50	0.51	0.78	0.73	0.79

* Still Feed = 250 ml 125 g U/l, 33 g SS/l, 3 M H^+ , 0.9 M Cl^- + 250 ml 15.8 M HNO_3 .

** After dilution with water to original volume of 250 ml.

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APPENDIX I

YANKEE ATOMIC²²

NUCLEAR	Core 1	Core 2
Average Thermal Flux	1×10^{13}	1.9×10^{13}
Thermal Power, Mw	392	480
Specific Power	18.3 kw/kg U	19.8 kw/kg U
Loading, kg U	21,400	24,200
Irradiation Time (full power days)	417	417
Load Factor, %	80	80
Burnup, Mw/mt U	7,600	8,200
Enrichment Initially	2.6%	2.6%
Pu Produced	5000 g/T U	5000 g/T U

<u>ELEMENT</u>	<u>Core 1</u>	<u>Core 2</u>
Type	Rod	Rod
Fuel	UO ₂ Pellets	UO ₂ Pellets
Pellet Diameter	0.30"	0.30"
Pellet Length	0.30"	0.30"
Pellet Density	10.2 g/cc	10.2 g/cc
Rod Active Length	90"	102"
Rod Total Length	92"	104"
U Content, kg	0.92	1.045
Tube Material	304 SS	304 SS
Tube ID	0.305"	0.305"
Tube Thickness	0.015"	0.015"
Tube OD	0.336"	0.335"
Pellet Radial Clearance	0.0017"	0.0025"

SUB-ASSEMBLY

	<u>Cores 1 and 2</u>
Type	Bundle
Number of Elements	64, 72, 80, or 81
Construction	The 8 or 9 rods of each row welded to notched strips which mesh with spacer strips to form roughly square bundle.
Rod Spacing	0.425" center-to-center on square pitch
Bundle Size	3.8" x 3.8" x 3.4" x 3.4"

ASSEMBLY

	<u>Core 1</u>	<u>Core 2</u>
Type	Square Array	Square Array
Number of Sub-assemblies	4	4
Number of Rods	305 or 306	305 or 306
Construction	Bolted	Bolted
Overall Length	107"	119"
Assemblies per Core	76	76
Outside Width	7-1/2"	7.495"
Active Section Before Irrad., kg		
UO ₂	319	362
304 SS Clad	54	60
304 SS Spacers	0.3	0.4
Active Section After Irrad., kg		
U ²³⁵	5.3	6.18
U ²³⁶	0.2	0.3
U ²³⁸	273	208
U	278	315
Pu ²³⁹	1.24	1.33
Pu ²⁴⁰	0.13	0.136
Pu ²⁴¹	---	0.136
Pu	1.37	1.6
SS	54	60
FP	2	2.6

O_2		<u>38</u>	<u>43</u>
Total Active Section		373	423
SS Rods, Ends		<u>45</u>	<u>45</u>
TOTAL ASSEMBLY		418	468

SHIPPING

Cooling Time Before Shipment,*	90 days	90 days
Uranium Shipped per Year, kg	15,000	22,000
First Shipment	Oct. 1961	-----

APPENDIX II

Proposed Feedend Solvent Extraction Cycle** for Yankee Atomic Fuel

HAF (100 vols): 124 g U/l, 33 g SS/l, 2 M HNO_3 , 0.61 g Pu/l, 0.01 M $NaNO_2$
 HAS (20 vols): 3 M HNO_3
 HAX (150 vols): 30% TBP
 HAP (150 vols): 83 g U/l, 0.405 g Pu/l, 0.2 M HNO_3 , 30% TBP
 HAW (120 vols): 1.9 M HNO_3 , 27.6 g SS/l, <0.01% U, <0.01% Pu
 HCU (300 vols): 41.4 g U/l, 0.20 g Pu/l, 0.1 M HNO_3
 HCK (300 vols): 0.01 M HNO_3

*Varies (Henry C. Mikszewicz, Yankee Atomic Electric Co.,
 441 Stuart Street, Boston, Massachusetts, CO-6-5800)

**Batch equilibration test of feed stage extraction using Pu tracer showed
 UE^0 of 9.8 and PuE^0 of 4.0.