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SOLVENT EXTRACTION IN HTGR REPROCESSING
INTERIM DEVELOPMENT REPORT

by
G. W. Reddick

Prepared under
Contract E(04-3)-167
Project Agreement No. 53
for the San Francisco Operations Office
U.S. Energy Research and Development Administration

February 1976

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ABSTRACT

Acid-Thorex flowsheets using tributyl phosphate (TBP) to process feed which simulated that from HTGR fuels were tested in pulsed solvent extraction columns. Tests were performed to study the effects of solvent degradation and solids in the feed and decontamination performance using tracer Zr-95. Equipment recommendations for each process step are made. The Acid-Thorex process is expected to provide relatively trouble-free operation in commercial reprocessing of HTGR fuel.

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INTRODUCTION

This interim report contains results from the solvent extraction development work at General Atomic Company funded by the ERDA Thorium Utilization Program. The work reported here was obtained from the pilot plant runs since its initial operation (March 1974) through October 1975 (Runs 1 through 48).

The purpose of the work includes:

1. The testing of the application of the Acid-Thorex process developed by ORNL (Ref. 1) to the processing of HTGR fuel.
2. The identification and testing of necessary and potentially beneficial process modifications.
3. The optimization of pulse column designs.

The development work to date has dealt primarily with fertile fuel particle processing although selected information is also applicable to solvent extraction processing of the fissile fuel.

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1. SUMMARY

The solvent extraction pilot plant at General Atomic Company was used to test the Acid-Thorex flowsheet on feeds representative of those from HTGR fuels. Pulsed columns with diameters of 2 and 3 inches were used in the pilot plant operations. Using 30% TBP solvent, greater than 99.9% of the thorium and uranium in the feed was recovered in the extraction step. Typically, the thorium and uranium separation in the first cycle (partition flowsheet) resulted in a uranium product with less than 100 ppm thorium. The thorium product contained less than 100 ppm uranium.

The thorium content in the uranium product increases with the addition of DBP (to simulate solvent degradation) but can be decreased by the addition of fluoride to the thorium strip solution (1BX).

Two basic first cycle flowsheets were tested. Due to the effects of the solvent degradation products on thorium stripping, the partition flowsheet is preferred over the costrip flowsheet.

The studies used tracer quantities of zirconium-95 to simulate fission products. Greater than 90% of the zirconium which was added to the feed was removed in the extraction-scrub step. Most of the remaining zirconium which was extracted followed the path of the thorium in the uranium and thorium separation step.

Operations with solids in the feed were tested. Some loss of column capacity was observed. The effective zirconium decontamination was also reduced.

Effects of a third phase (second organic phase) in the extraction-scrub operation were also determined. The third phase caused no significant operating difficulties when it occurred within the mixing area of the column.

The work performed to date indicates that the Acid-Thorex flowsheet is adaptable to HTGR fuels and should provide relatively trouble-free operation in commercial reprocessing using pulse columns. Equipment requirements are similar to those in other remotely-operated facilities.

2. REQUIREMENTS

The solvent extraction flowsheet and equipment for HTGR fuel reprocessing must be designed to recover the uranium and thorium in the aqueous feed stream produced by head-end operations. The solvent extraction system must separate the U-233 and thorium and also remove to a high degree the fission products, other actinides and chemical impurities. To achieve the required product purity, multiple solvent extraction cycles are needed. This interim report deals primarily with the first cycle of the solvent extraction process for the fertile particles, though some of the contained data is also applicable to fissile particle processing (Purex process).

The guidelines used in the GAC development work include the following:

1. As a practical limit, the maximum column cartridge height was to be 40 feet.
2. Column capacities with modern cartridges were to be determined for the Acid-Thorex process.
3. Preferred interface position establishing organic or aqueous continuous operation was to be determined for each column.
4. Column heights were to be chosen to minimize thorium and/or uranium losses and maximize the decontamination of fission products.
5. The maximum thorium loss via the first cycle extraction column raffinate stream would be less than 0.1%.
6. The maximum uranium loss per solvent extraction cycle would be less than 0.1%.

7. For uranium and thorium separation the criteria are as follows:
 - a. In the partition cycle uranium product stream, thorium concentration would be less than 500 parts per million parts of uranium.
 - b. In the partition cycle thorium product stream, the uranium concentration would be less than 20 parts per million parts of thorium.
8. The zirconium decontamination factor for the first cycle was to exceed 5 to prevent zirconium precipitation in the feed stream to the second thorium cycle.
9. The overall fission product decontamination factor required for the solvent extraction system as a whole is greater than 10^4 for uranium and greater than 10^3 for thorium.

As a general requirement, the process which is developed must also be applicable to remote operation and maintenance.

3 . EQUIPMENT DESCRIPTION

The solvent extraction pilot plant facility at General Atomic Company is comprised of the following major equipment items:

1. Five 2- and 3-inch (5.1 and 7.6 cm) diameter glass columns with overall heights up to 28 feet (9 meters).
2. Thirty-three stainless steel tanks ranging in capacity from 10 liters to 900 liters.
3. Feed and transfer pumps, intercolumn airlifts, flow rotameters, and metering pumps.
4. Interface sensing and control instrumentation
5. Pulse generators.
6. A thermosyphon evaporator fabricated from stainless steel with a boil-off capacity of about one liter per minute.
7. Heat exchangers.

The solvent extraction columns are assembled from sections of commercially available glass pipe. (The columns and associated equipment are shown in Figures 1, 2, 3, 4.) Stainless steel inserts between pipe sections provide means for connecting inlet streams, for sampling, and for supporting the column internals. Glass pipe crosses are used at the column ends as disengaging sections. Stainless steel plates on the cross ends provide means for connecting inlet, outlet and vent lines as well as interface sensor instruments and pulse lines. The column internals are made from commercially available sieve and nozzle plates. These plates are supported by a central rod with tubing sections used as plate spacers. The plate section heights used were varied and are listed in Appendix B.

Fig. 1. The top two levels of the solvent extraction pilot plant at General Atomic Company. The columns are in the plexiglas cabinet in the center of the photograph.

Fig. 2. An overall view of the solvent extraction pilot plant

Fig. 3. The top level of the solvent extraction pilot plant.

Fig. 4. The bottom level of the solvent extraction pilot plant.

All of the tanks are stainless steel construction. Each tank, as well as the evaporator, is equipped with nitrogen purged dip tubes to provide manometer or recorder liquid level readouts. Selected tanks and the evaporator are also equipped to provide specific gravity readouts. A network of stainless steel tubing is used for intertank transfers and stream flows to the columns.

Solution transfers between tanks and from the tanks to the columns are made with centrifugal or positive displacement pumps. Flows to the columns are controlled at the desired rates with visual rotameters and manual control valves. Shell and tube heat exchangers, which were commercially obtained, are used to control the temperatures of the streams to the columns.

Flows between columns are maintained by pumps or airlift systems when overflow hydraulics do not allow cascade flow to succeeding columns.

The pulse generators consist of teflon* bellows activated by variable throw cranks driven by variable speed motors. One-inch (2.5 cm) pipe and flexible hose connect the bellows to the column bottoms.

The product concentrator is a thermosyphon unit constructed of all stainless steel material. The single tube bundle utilizes low pressure steam. A packed section in the vertical vapor space between the feed point and the thermosyphon loop is used for stripping of organic materials from the feed by the rising vapor.

The solvent used in the pilot plant is technical grade tributyl phosphate (TBP) in normal paraffin hydrocarbon (NPH) diluent. The TBP is ordinarily 30% by volume in the organic phase. The NPH is a straight-chained hydrocarbon purchased to Atlantic Richfield Hanford Company and Allied Chemical-ICP specifications. NPH is 98% normal-C₁₀H₂₂ to normal-C₁₃H₂₈ with less than 0.2% aromatics by weight. The density is approximately 0.75 g/ml.

* duPont trademark.

4. FLOWSHEET

The nominal flowsheets in Figure 5, 6 and 7 were developed based on previous work (Refs. 1, 2, 3, 4). Some changes from previous flowsheets have been made due to the unusual characteristics of HTGR fuels. These changes are required due to the significant quantities of fission products which equilibrium discharge HTGR fuels will contain. Significant quantities of solids from head-end processing of the graphite fuel blocks are also present.

Initial development of the flowsheet and work in the pilot plant tests were used to assist in designing the solvent extraction system for the ICPP pilot plant (Ref. 5). Subsequent work has included work on flowsheets which are applicable to HTGR Recycle Demonstration Facility (HRDF) (Ref. 10) as well as ICPP. The HRDF flowsheet incorporates the results of some of the work reported here.

Figure 5 contains the partition flowsheet. In this case the uranium and thorium in 1AF feed (Stream No. 1) are extracted into the organic phase (30% TBP in NPH). Most of the fission products remain in the aqueous phase, which leaves via the bottom of the column as the 1AW waste (Stream No. 4).

The section above the 1AF feed point is called the 1S column (or scrub section). The purpose of this section is to scrub the fission products from the organic phase back into the aqueous phase.

In the 1BX and 1BS columns the thorium and uranium are separated from each other. The nitric acid concentration and phase flow ratios are adjusted to preferentially strip the thorium from the organic into the aqueous phase. The thorium leaves the 1BS column via the 1BT, aqueous product stream(stream #9). The uranium remains in the organic phase, which leaves the top of the 1BX column via the 1BU stream(stream #11).

The uranium is stripped from the organic phase into the aqueous phase in the 1C column. The 1CW organic stream #14, which leaves the 1C column, is treated in the solvent treatment system (Figure 5). The

STREAM	STREAM NO.	RELATIVE FLOW	COMPOSITION		
			U (G/L)	Th (G/L)	HNO ₃ (M)
1AF	1	100	35	348	1.0
1AS	2	104			0.01
1AX	3	1000		(30% TBP)	
1AW	4	262		(FISSION PRODUCTS)	
1AA	5	32			13.0
1SP	6	1000	3.5	35	
1AIS	7	26			5.0
1BX	8	600			0.2
1BT	9	600	Trace	102	
1BS	10	179		(30% TBP)	
1BU	11	1180	2.98		
1CX	12	593			0.01
1CU	13	593	5.93		
1CW	14	1180		(30% TBP)	
1WW	15	59		(NPH)	
1WS	16	59		(NPH)	
1WU	17	593	5.93		
2AF	18	6	233		
2AX	19	118		(5% TBP)	
2AW	20	41		(WASTE)	
2AS	21	34			2.0
2AP	22	118	11.8		
2CX	23	59		(ORGANIC)	0.01
2CW	24	118			
2CU	25	59	23.6		
2WW	26	6		(NPH)	
2WS	27	6		(NPH)	
2WU	28	59	27.5		
U PRODUCT	29	6	233		

Fig. 5. Partition flowsheet

Fig. 6. Solvent washing flowsheet

STREAM	STREAM NO.	RELATIVE FLOW	COMPOSITION		
			U (G/L)	Th (G/L)	HNO ₃ (M)
1AF	1	100	14	348	<1.0
1AS	2	104			0.01
1AX	3	1000		(30% TBP)	
1AW	4	262		(FISSION PRODUCTS)	13.0
1AA	5	32			
1SP	6	1000	1.4	35	
1AIS	7	26			5.0
1CX	8	1000			0.01
1CW	9	1000		(ORGANIC)	
1CP	10	1000	1.4	35	
1WW	11	100		(NPH)	
1WS	12	100		(NPH)	
1WP	13	1000	1.4	35	
2AF	14	100	14	348	
2AX	15	130		(5% TBP)	
2AW	16	139		255	
2AS	17	39			2.0
2AP	18	130	10.8		
2CX	19	65		(ORGANIC)	0.01
2CW	20	130			
2CU	21	65	21.6		
2WW	22	6		(NPH)	
2WS	23	6		(NPH)	
2WU	24	65	21.6		
U PRODUCT	25	6	233		

Fig. 7. Co-strip flowsheet

solvent treatment is intended to remove impurities, solvent degradation products, and fission products prior to reuse (Ref. 7). The solvent treatment operation can be performed in a pulsed column or in a batch wash with a sodium carbonate solution.

If a concentration step is required on the 1CU uranium product stream from the 1C column, the stream will first be contacted with countercurrent NPH flow in the 1W column. This step reduces the amount of organic materials (such as TBP) which enter the concentrator (Ref. 5).

Figure 7 contains the alternate costrip flowsheet. In this flowsheet the first cycle 1A-1S system is the same as the partition flowsheet in Figure 5. However, the flowsheet in Figure 7 consists of stripping of uranium and thorium together from the organic to the aqueous phase in the 1C column. The organic phase from the 1C column is treated in the solvent treatment system (Figure 6).

Following diluent washing (1W column) and concentration, the thorium and uranium solution is fed to the second cycle. This second cycle can consist of: (a) the first cycle of the partition flowsheet (Figure 5) with 30% TBP solvent with only slight modifications or (b) the second cycle in Figure 7. The primary function of the second cycle in Figure 7 is the selective extraction of uranium. This separation from thorium is accomplished using 5% TBP as the solvent. The Figure 7 flowsheet was selected by Allied Chemical for the HTGR reprocessing pilot plant (Ref. 5). The General Atomic Company's HTGR Target Recycle Facility (TRP) and HRDF flowsheets have a 30% TBP second cycle without intercycle concentration of the uranium (1CU) stream (Ref. 10). Selection of the optimum flowsheet approach for second uranium cycle will be the subject of future studies.

5. EXPERIMENTAL

The data collected during pilot plant solvent extraction runs included flow rates, pulse amplitudes and frequencies, temperatures, samples of discharge streams from each column, samples of the contents of intermediate column locations and visual column effects. The samples were collected under steady state conditions. Steady state in the pilot plant equipment is established after about 2 or 3 hours of sustained operation at nominal rates.

Flooding curves were obtained for each column operation by determining flooding points for several volume velocities. Flooding occurs at preselected flow rates when the pulse frequency is raised so high that the two phases cannot pass countercurrently through the column. Under a total flooded condition, one of the phases leaves the column at the same end at which it enters.

The method for obtaining a flooding point consisted of: (1) establishing the countercurrent flow in the column and (2) incrementally increasing the pulse frequency until flooding occurred. At each pulse frequency the column operation was allowed to reach an equilibrium operation prior to increasing the frequency the next increment. The time needed to allow equilibrium to be established after a frequency change was about 5 to 10 minutes. Each incremental change in the frequency was about 2 to 5 cycles per minute when the flooding frequency was being closely approached.

The pulsers are set up to produce a preselected amplitude. The amplitude which is normally used is one inch (2.5 cm). The frequency was obtained by visual counting.

The cartridge (plate type and spacing) for each column was selected based on capacity and efficiency data. The cartridges which were tested were derived from previous studies (Ref. 6).

Non-radioactive zirconium was added to the feed to duplicate the total zirconium concentration expected in equilibrium HTGR fuels. A tracer quantity of radioactive Zr-95 was added to the 1AF feed in selected runs to provide a means of determining the zirconium distribution throughout the five-column system. Radioactive zirconium was used because the analytical methods for zirconium determination at dilute concentrations in the presence of uranium and thorium were not well developed.

Temperatures of selected streams and columns were monitored and controlled. The extraction-scrub and partition-scrub systems were operated at ambient conditions of 25°C to 30°C. The solvent wash and stripping column were operated at about 35°C and 50°C, respectively.

6. DISCUSSION AND RECOMMENDATIONS

6.1 COLUMN CAPACITIES

Flooding curves for each representative column are included in Appendix A. For organic continuous columns, the stable and flooded regions are shown. For aqueous continuous columns, three regions exist: (1) stable zone, (2) cyclic flood zone, and (3) total flood zone. All of these curves were obtained in 2-inch diameter glass columns under nominal flowsheet conditions.

The presence of solids in the feed to the 1A system reduces column capacity by reducing the frequency at which flooding occurs for a given flow rate. This characteristic is indicated in Figure 1 of Appendix A.

The presence of DBP influenced the flooding characteristics. The presence of DBP in the 1C column in particular changed the dispersion characteristics enough that the flooding curve was significantly shifted. As the level of DBP in the 1C column increased, the pulse frequency at which flooding occurred also increased.

Plate characteristics and spacing influence flooding. Typically, increasing the percent free area or the hole size in a plate will increase the capacity. Also, increasing the spacing between plates will typically increase capacity. However, the changes mentioned which increase capacity may reduce the efficiency of the column. The cartridges (type of plates and spacing) selected for each column are the best known for the required operations at the present time.

6.2 COLUMN EFFICIENCY

The efficiency with which each column performs its function is dependent not only on the flowsheet, but also the physical configuration

of the columns and internals and operation of the mechanical equipment. After the desired equipment has been determined, the optimization of the flowsheet can begin.

The equipment is optimized by pilot plant operation at selected volume throughput rates and pulse frequencies for each column represented in the flowsheet. Data collected from these operations are used to specify the relative column diameter, the plate type, hole sizes in the plates and plate spacing. Appendix B contains the data obtained from pilot plant operation (Refs. 12, 13, 14, 15, 16).

The efficiency of each column operation in the flowsheet has been satisfactory in pilot plant testing. Discussion of each column follows.

6.2.1 1A Column (1A-1S, Extraction-Scrub Efficiency)

The 1A column operation consists of coextraction of thorium and uranium into 30% TBP solvent from an aqueous feed stream. Most of the fission product decontamination occurs in this column. In the pilot plant operation, greater than 99.9 percent of thorium and uranium can be recovered. Under these conditions the arithmetic decontamination factor* for zirconium is about 10 to 100. Changing conditions to improve thorium and uranium recovery will generally increase fission product extraction and thus reduce effective fission product decontamination.

Extending the 1A column length above the 1AF feed point (scrub section) from 9 feet to 27 feet increases the zirconium decontamination without reducing thorium and uranium recovery.

The 1A-1S flowsheet continues to be tested and optimized for fission product decontamination using non-radioactive zirconium and tracer quantities of radioactive zirconium.

*The arithmetic decontamination factor or DF is the ratio of zirconium to thorium in the feed divided by the ratio of zirconium to thorium in the product.

6.2.2 1BX-1BS (Partition Efficiency)

The 1BX and 1BS columns are used to partition the thorium and uranium. Separation of uranium from thorium has reduced uranium in the 1BT (aqueous product) stream to less than 100 parts of uranium per million parts of thorium. The 1BU (organic product) stream typically contained 100 to 1000 parts of thorium per million parts of uranium. (Separation factors are about 10^3 and 10^4 for thorium from uranium and uranium from thorium, respectively.

The addition of DBP (dibutyl phosphate) to simulate solvent degradation increased the carryover of thorium to the 1BU, organic stream. Methods developed to reduce this carryover are discussed in Section 6.6.

6.2.3 1C Column (Uranium Stripping Efficiency)

The uranium stripping operation has resulted in greater than 99 percent recovery in the 1CU aqueous product of the uranium from the 1BU stream which feeds the 1C column. An exception to this recovery occurred with the introduction of DBP into the system. This information is discussed in Section 6.6.

By visual observation of the color of the solutions, most of the mass transfer of uranium from the organic phase to the aqueous phase occurred in the bottom 5 feet of the 1C column (15 feet of total column length). Additional transfer in the top 10 feet of the column reduced the uranium concentration in the organic phase to near unstrippable values of about 10^{-3} g/l (Ref. 7).

6.2.4 Column Recommendations

The recommended column heights were derived from pilot plant experience. The recommended cartridge height for each column is presented in Table 1. These heights are required to meet the guidelines outlined in

TABLE 1
RECOMMENDED COLUMN INTERNALS DESCRIPTION^(a)

Column	Height of the Cartridge ft (m)		Nozzle Plates (Stainless Steel)			Plate Spacing, in. (cm)
			Nozzle Direction	Hole Size in. (mm)	Free Area (%)	
1A	15	(4.6)	Down	1/8 (3)	23	2 (5)
1S	25	(7.6)	Down	1/8 (3)	23	2 (5)
1BX	20	(6.1)	Up	1/8 or 3/16 (3 or 5)	23	Graded ^(b)
1BS	25	(7.6)	Up	3/16 (5)	23	2 (5)
1C	20	(6.1)	Up	3/16 (5)	23	Graded ^(b)
1W	20	(6.1)	Up	3/16 (5)	23	Graded ^(b)
1O	20	(6.1)	Down	1/8 (3)	23	2 (5)
2A	15 ^(c)	(4.6)	Down	1/8 (3)	23	2 (5)
2S	25 ^(c)	(7.6)	Down	1/8 (3)	23	2 (5)
2C	20	(6.1)	Up	3/16 (5)	23	Graded ^(b)
2W	20	(6.1)	Up	3/16 (5)	23	Graded ^(b)
2O	20	(6.1)	Down	1/8 (3)	23	2 (5)

(a) For pulse amplitudes of one inch for each column.

(b) Plate spacing varies from 4 inches at the bottom of the column to 2 inches at the top.

(c) Heights of 15 and 25 ft are recommended for the 2A and 2S, respectively, with 30% TBP. Heights of 20 ft for the 2A and 2S columns will be required for 5% TBP operations.

the Requirements Section. These column heights are specifically for the ICPP pilot plant. Analytical data were used to estimate HETS (height equivalent to a theoretical stage) values for selected operations using McCabe-Thiele calculations (see Appendix B). Phase equilibrium values for each calculation were estimated from the available data.

The mechanical requirements for these columns are no different than for those in plants having remote operation and maintenance. Therefore, remote equipment and instrumentation demonstration was not included in this study since these operations have been demonstrated in industrial or government reprocessing facilities.

6.3 CONTINUOUS PHASE RECOMMENDATIONS

The continuous phase recommendations are based primarily on two considerations. The first is the effectiveness of the column to perform the required separation (efficiency). The second is the operability (control) of the column under its given flowsheet conditions. The recommended continuous phase for each column is given in Table 2.

The columns in which the continuous phase is of particular concern are the 1A column and the 1BX column. The reasons for an organic continuous 1A column and an aqueous continuous 1BX column are discussed below.

The operation of the 1A column is a special case since the feed to the column is supplied directly from head-end processing. This feed contains not only the soluble heavy metals of interest, i.e., U and Th, but the soluble fission products, metallic impurities and small insoluble particles. Thorium and uranium are extracted into the lighter organic stream from the aqueous feed stream. The aqueous waste stream which exits the bottom of the 1A column contains most of the fission products and metallic impurities.

TABLE 2
RECOMMENDED CONTINUOUS PHASE FOR THE SOLVENT EXTRACTION COLUMNS

<u>Column</u>	<u>Continuous Phase</u>
1A	Organic
1S	Organic
1BX	Aqueous
1BS	Aqueous
1C	Aqueous
1W	Aqueous
10	Organic
2A	Organic
2S	Organic
2C	Aqueous
2W	Aqueous
20	Organic

However, at the column interface a layer of insoluble material tends to accumulate. One characteristic of this insoluble material (interfacial crud) is that fission products are present since many fission products are associated with the solids, e.g., zirconium. By operating the 1A column in an organic continuous mode, the interfacial crud and associated fission product activity are located at the waste end of the column (Figure 8). This operating method serves to improve fission product separation by locating a large potential contributor as far as possible from the product end of the column. Solvent degradation is likewise reduced since significant dose from the fission products occurs below the feed point (Ref. 8). The bottom interface also allows interfacial crud to slough off to the aqueous waste stream, preventing a progressive increase in the size of the crud layer.

An aqueous continuous 1BX column (partition column) is recommended based primarily on column control. Moreover, the efficiency of an aqueous continuous 1BX column is also typically as good as or better than an organic continuous operation based on HETS (height equivalent to a theoretical stage) calculations.

Recovery from a flooded condition (loss of countercurrent flow of the phases) in the aqueous continuous 1BX column is much easier than with an organic continuous operation. A very wide zone of cyclic flooding exists for the aqueous continuous system. Not only does the cyclic flooding zone help give a warning to the column operator, but it produces a negligible change in column thorium separation efficiency when the cyclic flood first develops. Only a very small loss in efficiency in the aqueous continuous 1BX column occurs because a holdup of the organic phase develops near the bottom of cartridge mixing area. If the uranium concentration in the 1BXT thorium stream (aqueous effluent stream from 1BX column) increases due to a flood in the aqueous continuous column, the uranium would be separated from the thorium in the 1BS column.

Fig. 8. 1A Column interface.

Flooding an organic continuous 1BX column holds up the aqueous 1BX stream and hence would result in rapid partition failure (i.e., thorium exits the column with the uranium product). The flooded organic continuous 1BX column recovers from a flood very slowly on lowering the flow rate and/or pulse frequency.

The 1C and 1BS columns were operated with the aqueous phase continuous. In these columns, as in the 1BX column (aqueous continuous), a cyclic flood occurred prior to a total flood.

The second cycle columns exhibit flooding characteristics similar to their respective organic or aqueous continuous column in the first cycle. However, the operation of an organic continuous column with 5% TBP in NPH was not satisfactory since recovery from a flooded condition was very difficult. In most cases the column must be shut down and re-started following flooding.

The solvent wash columns (10, 20) are operated with the organic phase continuous (bottom interface) for two reasons: (1) organic continuous operation increases the organic phase holdup, increasing contact time, and (2) the solids in the system tend to exit with the spent carbonate wash solution.

In the pilot plant the diluent wash column (1W column in Figure 5) was operated with the aqueous phase continuous (top interface) to cause any interfacial crud to exit the column with the diluent. The diluent was NPH (normal paraffin hydrocarbon material), which is used routinely as the diluent in the other pilot plant solvent streams. The operation in the pilot plant was carried out with no significant operating difficulties. Flooding tests were performed. Flooding occurred with the organic phase held up in the bottom of the column. Recovery from flooding was accomplished without any complications.

For diluent washing the aqueous to organic flow ratio was about 10 to 1. The phase dispersion for this pilot plant operation was very similar to that observed in other aqueous continuous operations. The aqueous product in the bottom disengaging section was always cloudy, indicating possible entrainment of organic into the aqueous phase. This observation suggests that steam stripping towers should still be required on product concentrators even though diluent washing is done to remove TBP.

6.4 CARTRIDGE RECOMMENDATIONS FOR EACH OPERATION

The cartridge recommendations for each column are made based on column efficiency and capacity. The cartridge descriptions are contained in Table 1. All of the recommended plates are stainless steel nozzle plates with 23% free area. The holes in the plates for the aqueous continuous operations are 3/16-inch diameter. The hole size recommended for the organic continuous operation is 1/8-inch diameter. An optimum hole size for the 1BX column has not yet been clearly established. Plates with 1/8-inch diameter holes are satisfactory, but pilot plant runs are being made to determine if 3/16-inch diameter is preferred on the basis of improved capacity (throughput) characteristics.

It is recommended that the 1A-1S system be built as a single compound column. Operation as a single column removes the control problems inherent with a stacked two-column system in which three phases (two organic phases) are present at the disengaging sections at the interacting ends of the columns. (See Section 5.8 for discussion)

6.5 ZIRCONIUM DECONTAMINATION

The flowsheet is designed so that most of the decontamination of zirconium and other fission products occurs in the 1A-1S system. To test the effectiveness of the flowsheet for zirconium decontamination, non-radioactive zirconium plus tracer quantities of radioactive Zr-95 have been used. The zirconium was added to the 1AF feed for these tests

because it is one of the more difficult fission products to remove in the Acid Thorex process. Its behavior in the solvent extraction operation is of primary interest.

The radioactive Zr-95 was added to improve measurement of zirconium throughout the five-column system. Two analytical methods were used for measurement of radioactive zirconium. One method consists of counting with a detector using a NaI crystal. This method counts both Zr-95 and its daughter Nb-95. To measure only Zr-95, a GeLi detector was used.

The flowsheet is designed to recovery > 99.9% of the thorium and uranium in the feed. However, the heavy metal concentration in the organic phase at the feed point has a direct relationship on fission product decontamination. As this organic phase concentration increases, the fission product decontamination also increases. The maximum thorium concentration in the organic phase is limited by several factors. Losses from the 1A column via the 1AW stream will develop when the organic phase approaches saturation (thorium solubility limit). Increasing the phase ratio (aqueous to organic flow ratios) will increase losses, requiring lower thorium concentration at the feed point.

Optimization of the 1A column was aimed toward recovering all of the thorium and uranium and yet extracting as little of the fission products as possible. For zirconium the arithmetic decontamination factor (DF) was as high as 34 in the extraction section (below the feed point) in the pilot plant 1A column.

Considerable decontamination can be obtained in the scrub section (above the feed point) if the phase ratios and acidity are used to advantage. In the pilot plant the arithmetic decontamination factor (DF) across the scrub section was as large as 30.

In the 1BX column, the zirconium typically follows the aqueous phase. About 90 to 99 percent of the zirconium which enters the 1BX-1BS system

in the 1SP stream leaves via the 1BT stream. This significant separation occurs because the conditions (phase ratio, acidity and fluoride) which partition the uranium and thorium are also favorable for the zirconium from uranium separation.

Some additional decontamination of zirconium may be obtained in the 1C column. This separation occurs because some zirconium is unstripable, possibly due to formation of zirconium complexes with organic degradation products which remain in the organic phase.

The second uranium cycle flowsheet (5% TBP) was also tested for zirconium decontamination. Very good decontamination was obtained. An overall DF of greater than 7000 was obtained for zirconium-niobium. This decontamination was obtained while recovering greater than 99.8% of the uranium in the feed. Most of the decontamination was effected in the 2A column; however, some additional decontamination was obtained in Both the 2S and 2C columns.

6.5.1 1A Column Zirconium DF

To improve the zirconium decontamination in the 1A-1S system, additions to the 1AS stream were tested. The addition of 0.001M fluoride to the 1AS stream does not improve zirconium-thorium separation significantly. This is probably due to the thorium complexing the fluoride and lowering the free fluoride to a level which is ineffective in altering zirconium distribution.

Some initial tests using 0.01M EDTA (ethylenediamine-tetraacetic acid) in the 1AS stream produced some positive results. After the EDTA was added to the 1AS stream in the second half of one pilot plant run, the zirconium DFs improved. The DFs for zirconium-niobium and zirconium, respectively, increased to 225 and 135 from 92 and 41 before the EDTA addition. The improvement came mostly in the 1A column suggesting that EDTA improves the extraction section zirconium DF.

Although the EDTA in the 1AS stream apparently improved the DF for zirconium, the addition of DBP to the 1A column to simulate solvent degradation subsequently returned the zirconium levels in the 1SP stream to higher than experienced prior to the EDTA and DBP additions. Further testing is required to ascertain the benefits, if any, of EDTA on zirconium separation in the presence of DBP.

6.5.2 Solids in Feed

The effect of solids in the feed is one aspect of processing HTGR fuel which has particular interest. Some solids are expected to carry through from the head-end operations. Therefore, the effect of small quantities of graphite or fuel particulates on the system was investigated.

In the pilot plant graphite and/or carbide material was added to the leaching or feed adjustment operation prior to use in the solvent extraction runs. All solvent extraction feed produced from pilot plant leaching operations had visually detectable suspended solids. In the cases when graphite or carbide was added to the leaching or feed adjustment steps, the amount of the suspended solids was noticeably increased.

The solids introduced to the 1A column were visually observed in the column below the feed point. The solids typically follow the aqueous stream in the column.

A buildup of solids occurs at the bottom interface. However, an equilibrium point for solids buildup at the interface is reached at which the solids are continuously sloughed to 1AW aqueous waste stream and no further interface buildup occurs.

Under the worst conditions tested for feed solids, some loss of column capacity was experienced. Up to 0.5 weight percent carbon was added to the 1AF feed. The capacity change was affected by the change

in the dispersion characteristics. This change occurs because the solids are typically on the interface between the aqueous droplets and the organic continuous phase. Thus, changes in mixing characteristics result. The 1A column developed flooding at lower relative column throughputs when solids were present. However, the efficiency of thorium and uranium extraction in 1A column remained within controllable limits. Under the worst operating conditions caused by the presence of solids, the maximum thorium and uranium losses were about 0.35 and 0.25 percent, respectively.

Solids in the feed also affect the zirconium decontamination. Zirconium decontamination was reduced by as much as a factor of five when feed was produced by normal head-end operations. The proposed cartridge (Table 1) for the 1A column and the current 1A flowsheet appears to be adequate for solids content up to 0.5 wt.% carbon without seriously affecting the column operation or efficiency.

6.6 SOLVENT DEGRADATION

Degradation of the solvent occurs not only due to radiolysis but also acid hydrolysis. The TBP (tributyl phosphate) degradation is the source of degradation products which have the greatest effect on thorium and uranium purification with solvent extraction. The primary degradation product of TBP is DBP (dibutyl phosphate), (References 7, 11). DBP remains in the organic phase under most operating conditions. The NPH is a proven diluent which undergoes little degradation (Ref. 7).

Thorium forms a complex with DBP which most affects the operation of the solvent extraction system. This complex causes reduced partition efficiency and also precipitates in the uranium stripping column (1C column) due to the lower acid concentration. After prolonged buildup of DBP in the 1C (stripping) column, some of the thorium-DBP is purged to the solvent wash system. The thorium which reaches the 10 column (solvent wash system) precipitates as thorium hydroxide.

The flowsheet composition of the 1BX and 1CX streams was altered to contain $0.005M F^-$ and $0.001M F^-$, respectively, in addition to the normal nitric acid concentration. The purpose of the fluoride addition to the 1BX stream was to reduce thorium carryover to the 1C column by DBP. As shown by Figure 9, the desired result of lowered thorium concentration in the 1BU stream was achieved. The presence of fluoride in the 1BX stream improved thorium separation even when no DBP was added. This effect is for the flowsheet uranium and thorium concentrations. Changes of these concentrations or the ratio of thorium and uranium in the 1BX column may produce other results.

The purpose of the fluoride addition to the 1CX stream was to reduce the thorium carryover by DBP from the 1C column to the 1O column. This desired result was achieved since the concentration of thorium in the 1CW stream was reduced by factors of 10 to 100 with fluoride in the 1CX versus no fluoride. This data was obtained from pilot plant runs in which DBP had been added in selected amounts to simulate solvent degradation. Visually the 1O column also showed the positive effects at the interface when fluoride was added to the 1CX. No white precipitate (thorium hydroxide) formed at the interface as had occurred in runs which had DBP present but no fluoride in the 1CX stream.

6.7 COEXTRACTION-COSTRIP

In addition to the partition flowsheet, the costrip flowsheet in Figure 7 was also tested. Under unirradiated conditions the costrip cycle demonstration was accomplished with little difficulty. However, simulated solvent degradation by the addition of DBP (dibutyl phosphate) caused significant operating difficulties in the costrip (1C column) and solvent wash operations. (Solvent washing is shown on the flowsheet in Figure 6. The solvent wash operation consists of contacting the stripped solvent, stream No. 9, with a dilute sodium carbonate solution.)

DBP caused precipitation of a thorium-DBP complex in the 1C column. Visually, a very heavy organic precipitate was sloughed from the column via the 1CP stream (stream No. 10 in Figure 7). A very heavy frothy

Fig. 9. Effects of fluoride in IBX stream

interface formed at the top of the 1C column. Some flaky solids also formed at the interface. A dispersion change occurred in the center of the column as a film was formed on the plates. (Figures 10, 11, 12 show the progressive effect of DBP addition on the 1C column; Figure 10 is before the DBP addition.) The film caused the normally aqueous wetted plates to be wetted by the organic phase. A higher pulse frequency is required to maintain the column efficiency. However, since the film deposition and its effect occur primarily in the center section of the column, increasing the pulse frequency too much can result in a cyclic flood developing near the bottom of the column.

The perturbed operation of the 1C column and the DBP caused significant thorium losses to the solvent wash column. In the solvent wash column, white solids (thorium hydroxide) formed at the bottom interface. With time, a heavy buildup of solids formed on the bottom of the solvent wash column. The walls of the column became coated, and solids accumulated on the plates in the lower four to five feet of the column.

No practical method is available to separate DBP and thorium in the first cycle costrip flowsheet without precipitation occurring. Based on pilot plant results, the partition flowsheet is recommended over the costrip flowsheet for the first cycle operation.

The HTGR Target Recycle Plant (Ref. 17) included a coextraction-costrip step as the first cycle of the solvent extraction flowsheet. Due to the work done in the pilot plant with solvent degradation simulation, the solvent extraction flowsheet for the HRDF (Ref. 10) contains the partition flowsheet for the first cycle.

6.8 SPLIT SCRUB COLUMN OPERATIONS

Because of the operating difficulties experienced with split column operations (both 1A-1S and 2A-2S), single compound columns are recommended for each of these operations. This recommendation is based, first of all, on the instability of split column systems under the best of circumstances. Because these systems are operated with the organic phase continuous, there is no cyclic flooding zone between the unflooded

Fig. 10. Normal 1C column dispersion.

Fig. 11. The dispersion characteristics in the 1C column have changed due to the introduction of DBP.

Fig. 12. Progressive effects of DBP in 1C column. Note the size increase in some droplets from Fig. 10.

condition and total flood as occurs with aqueous continuous operations. Therefore, flooding in the organic continuous column can occur with essentially no warning. Recovery from flooding in an organic continuous column is difficult, but recovery from flooding in a split column operation is even more difficult. The split column operation is also a contributor to the column instability, which leads directly to flooding. In the pilot plant operation, much unintentional column flooding in the 1A-1S system occurred due to the cyclical nature of the intercolumn flows.

Operation of the 1A-1S system and 2A-2S system, each as a single compound column, eliminates the need for one interface controller plus the intercolumn transfer systems. The response of the compound column to changes made in flowsheet parameters will be faster since hold-up volume of two disengaging sections in each system (one from each of the 1A, 1S, 2A, 2S columns) will be eliminated. Since the hold-up volume of solvent is reduced, the solvent radiation damage is also reduced.

6.8.1 Third Phase

Thorium and 30% TBP solvent form two phases at thorium concentrations which are present in the 1A-1S system. The presence of the two organic phases (three total phases) can create operating difficulties in a split 1A-1S column system if the density of the second organic phase is about the same as the aqueous phase or if the two organic phases are both present in significant quantities at the disengaging sections.

When two columns were used for the 1A-1S system, the two organic phases which were present in the disengaging sections at the bottom of the 1S column and the top of the 1A column affected operating conditions and the acquisition of representative samples. In the bottom disengaging section of the 1S column, the two organic phases tended to separate above the organic-aqueous interface (Figure 13). Poor interface control developed because the second organic phase is much denser than the average density of the two organic phases when mixed. Also, the thorium reflux increased since the heaviest organic phase consists principally of thorium and TBP.

Fig. 13. The bottom of the 1S column. The heavy second organic phase is shown. The two interfaces are indicated.

In the top disengaging section of the 1A column, the two organic phases also tend to separate. In this case, however, the lighter organic phase overflowed until enough of the heavier organic phase builds up to also overflow. The interrelationship between the top of the 1A column and the bottom of the 1S column resulted in cyclical flow between the columns plus a cyclic reflux of thorium.

Operation of the 1A-1S system as one column also produced a third phase (second organic phase) in the scrub section. However, the presence of the three phases in the mixing area of the column did not create any noticeable problems. The presence of the third phase was easily detected in the glass columns because the column contents became cloudy (see Figure 14--Three phases are present in the upper part of the column). Operating the 1A-1S system as one column, no significant problems occurred at the disengaging section at the top of the column because the overflow thorium concentration was controlled below the concentration at which the third phase will form. This concentration is about 30 to 33 grams per liter.

No third phase formation occurred in the other columns since the thorium concentration in the organic phase was below that required for third phase formation.

6.8.2 Split Scrub Streams

On the flowsheets, Figures 5 and 7, two streams are added to the 1S scrub section. These streams, the 1AS stream (stream No. 2) and the 1AIS stream (stream No. 7), can be combined in actual operation. This combined 1M HNO_3 stream (also called 1AS) is added at the top of the column. The split scrub system is intended to improve fission product scrubbing in the top of the scrub section. However, the acid profile in the column is not changed significantly by the low acid (0.01M HNO_3) addition.

Fig. 14. Cloudy three-phase region in 1A column.

The aqueous to organic ratio is more favorable for fission product scrubbing throughout the column with a single stream addition. Operation with the two split scrub streams (0.01M HNO_3 in 1AS and 5.0M HNO_3 in the 1AIS stream) can cause greater column instability when normal flow variations occur than with the single 1.0M HNO_3 scrub stream. With the partition step following the scrub operation, acid control in the 1BX column is better with a 1.0M HNO_3 1AS scrub stream than with the split scrub.

The 1AIS stream provides an inlet location convenient for the addition of aluminum nitrate (ANN). The purpose of the aluminum nitrate is to complex the fluoride in the 1AF stream (stream No. 1) to increase the separation of the fluoride from thorium and uranium. However, fluoride is added to both the 1BX and 1C columns (via 1BX and 1CX streams, respectively) to moderate the effects of solvent degradation products. Therefore, increased fluoride decontamination in the 1A-1S system by ANN additions is not required. The column design should include a location for the 1AIS stream to give some flexibility though not required in the current flowsheet.

6.9 SOLVENT TREATMENT

Some work has been done to test methods of solvent cleanup. Washing with a 0.27M sodium carbonate solution in a pulsed vertical column is one method which has been used. A batch wash with sodium carbonate has also been used. A method of solvent cleanup with a bed of macroreticular resin was also tested.

The flowsheet for the sodium carbonate wash of the solvent is given in Figure 6. The column is operated with the organic phase continuous. All of the columns, except the solvent wash column, are operated under acid conditions. However, the sodium carbonate neutralizes any nitric acid carried from the stripping column with the solvent. Since the wash column contents are basic, most heavy metals and fission products are not soluble in the solvent phase and are stripped into the aqueous phase.

Under the basic conditions of the wash column, some precipitation of heavy metals and fission products occurred. The solids collected at the interface at the bottom of the column. After an equilibrium amount of solids had built up at the interface, additional solids were sloughed into the spent carbonate wash solution; which exits the bottom of the column.

When significant amounts of thorium reached the solvent wash column, as occurred in the coextraction-costrip flowsheet with the DBP additions, enough solids formed in the column to coat the walls and plates in the bottom third of the column. With the other flowsheets no significant buildup of solids occurred in the wash column. This is true even for the cases with DBP addition if F^- is added to the LBX and LC columns via the aqueous addition streams to those columns.

Batch washing of the solvent with sodium carbonate is equivalent to column washing. Batch washing was used instead of or in conjunction with column washing. In the case of the coextraction-costrip flowsheet, batch washing of the solvent was required since the large amounts of thorium, DBP, and solids in the column prevented an efficient wash operation.

A method developed at Hanford (Reference 9), for solvent cleanup was also tested. This method consists of passing the solvent through a bed of macroreticular resin after solvent washing has been completed. This treatment is used to remove impurities which remain after solvent treatment. These impurities include fission products and solvent degradation products. The analytical technique for DBP measurement in the low concentrations present after solvent washing is not thoroughly developed at GA. Therefore, accurate measurement of the DBP levels before and after ion exchange treatment is not possible. However, the level of radioactive zirconium in the solvent was reduced to levels below the detection limits. Future testing of the ion exchange cleanup is planned.

6.10 AIRLIFT OPERATION - FOAMING

For the continuous intercolumn transfers of solution, airlift systems were used when overflow hydraulics were not adequate. These systems permitted airlifts to be tested for applicability to the various transfers and also eliminated the need for pumps. Few problems have been experienced with the actual airlift operation.

The most significant problem has been the production of foam in the head pot with organic solutions containing thorium and uranium. The difficulty arises when this frothy stream is introduced into the next column. The mixing characteristics of that column are changed by the air which is present in the input organic stream. Flooding will occur in the column under operating conditions which are normally well within the stable region. However, modification of a head pot to reduce and essentially eliminate the carryover of air from the head pot to the next column has been demonstrated.

This headpot with the foam eliminator was made by placing a stainless steel sieve plate cartridge (1/2-inch plate spacing) into a section of glass pipe and connecting this section horizontally into the overflow of the head pot (Figure 15). The sieve plates promote gas-liquid separation as the solvent flows slowly through the cartridge, thus, dissipating the foam

6.11 5% TBP OPERATIONS

No change in techniques is required for the column operation when 5% TBP (tributyl phosphate) in NPH (normal paraffin hydrocarbon) is used as the solvent instead of 30% TBP. Systems operated with 5% TBP solvent are slightly less stable than the 30% TBP systems. The 5% TBP system which created significant difficulties was the organic continuous operation. The difficulties occurred when a column flooded. Recovery from the flood is very difficult, if not impossible, without shutting down

Fig. 15. Airlift Schematic. Typical system for organic overflow. Dropout pot contains packing for foam reduction.

the column and proceeding through a normal startup. A similar problem occurs with the organic continuous 30% TBP system, but recovery from a flooded column is possible without a column shutdown as with the 5% TBP. Where possible, 30% TBP systems should be used preferentially over 5% TBP systems.

7. FUTURE WORK

Future solvent extraction development will include the continuation of work to define and optimize fission product decontamination capabilities. Particular emphasis will be placed on fission product decontamination with solvent degradation simulation and with solids in the feed as additional feed sources become available. A centrifugal contactor (Robatel) will be installed and tested to determine its effectiveness in performing the coextraction step. Of specific interest is the ability of the contactor to handle a third phase (second organic phase) and solids in the feed. Feed and flowsheet requirements for the centrifugal contactor will be established. A centrifugal contactor may effectively reduce the solvent degradation when processing irradiated fuels by reducing the residence time required in the coextraction step.

Additional work will be required to further define the second uranium cycle flowsheet. Further definition is also required for: (1) washing of aqueous product solutions with diluent and (2) concentration of the intercycle product. Concentration tests will include: (1) the effectiveness of steam stripping the feed to remove soluble organic compounds and (2) the ability to meet the feed requirements of the next cycle.

Optimization of solvent washing and solvent cleanup will also continue. Usefulness of solvent cleanup by ion exchange will be evaluated. Other development work will include the following:

1. Effectiveness of ruthenium decontamination will be tested using ruthenium tracer. Ruthenium is of concern due to the difficulty of its separation from uranium and thorium.

2. The capacity and cartridge for a plutonium removal column will be defined. A plutonium removal column is required to meet the plutonium limits which will be placed on the uranium and thorium product.
3. Use of recycle nitric acid and water will be tested.
4. Studies to achieve additional uranium removal from thorium in the second thorium cycle will be made.
5. Studies will also be made to test the effect of different thorium to uranium ratios in the feed.

Design verification studies will be made on the final solvent extraction system specified for HRDF.

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APPENDIX A
FLOODING CURVES

The flooding curves in Appendix A were obtained from operation of 2-inch diameter columns. The plate type and spacing is given for each curve.

Fig. A-1. 1A-1S System Flooding Curve - Organic Continuous.

Fig. A-2. 1BX Column Flooding Curve - 30% TBP.

Fig. A-3. 1BS Column Flooding Curve - 30% TBP.

Fig. A-4. 1C Column Flooding Curve - 30% TBP.

Fig. A-5. 1W Column Flooding Curve (first cycle diluent wash).

Fig. A-6. 2A and 2S Columns - Flooding Curve (2A-2S as two columns).

Fig. A-7. 2W Column Flooding Curve (second cycle diluent wash).

APPENDIX B
PILOT PLANT DATA

Appendix B contains data from pilot plant operations.

The total height of each column cartridge is given. For the 1A-1S system (Table B-1 and Table B-2), the scrub height (above the 1AF feed point) and the extraction height (below the 1AF feed point) are also included.

Throughputs are given as both volume velocity and superficial velocity. Volume velocity is the sum of the phase volumes per unit time per cross-sectional area of the column. The superficial velocity is the linear velocity ($\bar{V}_a$ is the aqueous phase, $\bar{V}_o$ is the organic phase) of each phase based on the total cross section of the column.

The flooding frequency is given for each volume velocity. The other data in each table were obtained at the percent of flooding frequency as given for each operation.

TABLE B-1
LOSS DATA 1A-1S ONE-COLUMN SYSTEM

Total Cartridge Height, ft, (m)	Extraction Height, ft, (m)	Scrub Height, ft, (m)	Volume ^(a) Velocity, (gal/hr/ft ²)	Superficial ^(a) Velocity		Flooding Frequency, (cycles/min)	Data at This Percent of Flooding					
				$\bar{V}_a$, (cm/s)	$\bar{V}_o$, (cm/s)		Aqueous/ Organic Ratio	Percent of Flooding Frequency	Percent Loss		1AW HNO ₃ , (M)	1SP Th, (M)
									U	Th		
18 (5.5)	9 (2.75)	9 (2.75)	910	0.19	0.84	126	0.22	80	0.001	1.3	1.1	0.14
18 (5.5)	9 (2.75)	9 (2.75)	1550	0.32	1.43	120	0.22	80	0.04	1.2	1.1	0.15
18 (5.5)	15 (4.6)	3 (0.9)	670	0.15	0.61	140 ⁺	0.25	~80	0.002	1.4	0.9	0.13
18 (5.5)	15 (4.6)	3 (0.9)	952	0.20	0.88	143	0.23	80	0.003	0.03	0.7	0.12
18 (5.5)	9 (2.75)	9 (2.75)	854	0.22	0.75	147	0.30	80	0.001	4.21	0.5	0.16
22 (6.7)	13 (4.0)	9 (2.7)	847	0.19	0.77	145	0.25	78	0.001	0.12	2.6	0.15
22 (6.7)	13 (4.0)	9 (2.7)	975	0.22	0.88	--	0.25	~85	10 ⁻⁴	0.007	2.2	0.14
22 (6.7)	13 (4.0)	9 (2.7)	925	0.22	0.83	145	0.26	80	0.001	0.02	2.2	0.15
22 (6.7)	13 (4.0)	9 (2.7)	1072	0.26	0.95	147	0.28	80	0.02	0.11	1.7	0.16
22 (6.7)	13 (4.0)	9 (2.7)	690	0.17	0.61	158	0.27	76	0.03	0.04	1.8	0.15
22 (6.7)	13 (4.0)	9 (2.7)	915	0.21	0.82	140	0.26	80	0.24	0.05	1.7	0.16
22 (6.7)	13 (4.0)	9 (2.7)	984	0.23	0.88	137	0.26	73	0.002	0.22	2.3	0.16
22 (6.7)	13 (4.0)	9 (2.7)	846	0.20	0.76	143	0.27	75	0.01	0.01	1.7	0.20
22 (6.7)	13 (4.0)	9 (2.7)	872	0.21	0.78	142	0.27	78	0.002	0.002	3.0	0.15
22 (6.7)	13 (4.0)	9 (2.7)	954	0.24	0.84	140	0.29	80	0.02	0.002	2.5	0.13
22 (6.7)	13 (4.0)	9 (2.7)	776	0.18	0.70	147	0.25	77	0.01	0.34	2.0	0.14
22 (6.7)	13 (4.0)	9 (2.7)	785	0.19	0.70	146	0.28	80	0.01	0.16	2.4	0.15
22 (6.7)	13 (4.0)	9 (2.7)	928	0.22	0.83	140	0.26	79	<0.01	0.11	2.3	0.13
22 (6.7)	13 (4.0)	9 (2.7)	793	0.19	0.71	145	0.26	72	<0.01	0.11	2.4	0.13
22 (6.7)	13 (4.0)	9 (2.7)	864	0.20	0.78	139	0.25	64	0.02	0.03	2.5	0.14
22 (6.7)	13 (4.0)	9 (2.7)	900	0.18	0.84	137	0.22	69	0.05	0.008	2.2	0.15
22 (6.7)	13 (4.0)	9 (2.7)	910	0.19	0.84	137	0.22	69	0.09	0.008	2.1	0.15
22 (6.7)	13 (4.0)	9 (2.7)	742	0.17	0.67	145	0.26	64	0.04	0.008	2.7	0.12
22 (6.7)	13 (4.0)	9 (2.7)	752	0.16	0.69	145	0.23	64	0.05	0.08	2.3	0.10
22 (6.7)	13 (4.0)	9 (2.7)	1046	0.19	0.99	129	0.19	65	0.10	0.01	1.7	0.12
22 (6.7)	13 (4.0)	9 (2.7)	1062	0.17	1.03	129	0.17	65	0.09	0.008	1.6	0.11

(a) $\bar{V}_a + \bar{V}_o = \bar{V}_T$; Volume Velocity (in gallons/hr/ft²) = 885 ($\bar{V}_T$).

TABLE B-2
LOSS DATA 1A-1S TWO-COLUMN SYSTEM

1A Extraction Column													
Total Cartridge Height, ft, (m)	Extraction Height, ft, (m)	Scrub Height, ft, (m)	Volume (a) Velocity (gal/hr/ft ²)	Superficial ^(a) Velocity		Flooding Frequency, (cycles/min)	Aqueous/ Organic Ratio	Data at This Percent of Flooding					
				$\bar{V}_a$, (cm/s)	$\bar{V}_o$, (cm/s)			Percent of Flooding Frequency	Percent Loss		1AW HNO ₃ , (M)	1AP Th, (M)	
									U	Th			
22 (6.7)	22 (6.7)	0.0 (0.0)	629	0.14	0.57	125	0.24	71	0.06	0.005	1.87	0.18	
22 (6.7)	13 (4.0)	9 (2.7)	1040	0.21	0.97	84	0.22	92	0.08	--	2.11	0.13	
22 (6.7)	13 (4.0)	9 (2.7)	1029	0.21	0.95	84	0.22	92	0.04	0.008	2.08	0.17	
22 (6.7)	13 (4.0)	9 (2.7)	1035	0.21	0.96	84	0.22	92	0.05	0.005	2.08	0.15	
22 (6.7)	13 (4.0)	9 (2.7)	679	0.18	0.59	116	0.31	69	0.05	0.02	2.88	0.17	
22 (6.7)	13 (4.0)	9 (2.7)	717	0.18	0.63	112	0.29	71	0.005	0.005	2.32	0.22	
22 (6.7)	13 (4.0)	9 (2.7)	932	0.18	0.87	91	0.20	88	0.04	0.02	1.68	0.17	
22 (6.7)	13 (4.0)	9 (2.7)	972	0.18	0.92	88	0.19	75	0.02	0.09	1.28	0.14	
22 (6.7)	13 (4.0)	9 (2.7)	1043	0.24	0.94	84	0.26	79	0.25	0.35	1.70	0.22	
22 (6.7)	13 (4.0)	9 (2.7)	1080	0.24	0.98	81	0.25	99	0.03	0.008	1.75	0.16	
22 (6.7)	13 (4.0)	9 (2.7)	1077	0.24	0.98	81	0.25	99	0.04	0.01	1.66	0.18	

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1S Scrub Column						Data at This Percent of Flooding					
Cartridge Height, ft, (m)	Volume (a) Velocity, (gal/hr/ft ²)	Superficial (a) Velocity		Flooding Frequency, (cycles/min)	Aqueous/ Organic Ratio		Percent of Flooding Frequency	% Recycle (to 1A)		1SR HNO ₃ (M)	1SP Th, (M)
		$\bar{V}_a$, (cm/s)	$\bar{V}_o$, (cm/s)					U	Th		
18 (5.5)	568	0.07	0.57	155	0.12		60	0.002	19	2.07	0.12
18 (5.5)	931	0.09	0.96	106	0.09		76	1.02	9	2.35	0.11
18 (5.5)	922	0.09	0.95	106	0.09		84	1.16	17	2.60	0.12
18 (5.5)	955	0.09	0.99	106	0.09		90	1.22	21	2.65	0.10
18 (5.5)	603	0.09	0.59	130	0.16		57	1.17	25	1.20	0.09
18 (5.5)	645	0.10	0.63	121	0.16		61	1.78	30	1.13	0.13
18 (5.5)	859	0.10	0.87	98	0.11		76	2.24	24	1.44	0.18
18 (5.5)	886	0.08	0.92	95	0.08		78	1.40	12	2.19	0.12
18 (5.5)	948	0.14	0.93	90	0.15		87	4.85	32	1.47	0.14
18 (5.5)	984	0.14	0.97	88	0.14		91	2.96	33	1.75	0.12
18 (5.5)	991	0.14	0.98	87	0.15		92	4.40	37	1.66	0.14

(a) $\bar{V}_a + \bar{V}_o = \bar{V}_T$; Volume Velocity (in gallons/hr/ft²) = 885 ($\bar{V}_T$).

TABLE B-3
HETS DATA 1BX COLUMN

Total Cartridge Height, ft, (m)		Volume ^(a) Velocity, (gal/hr/ft ²)	Superficial ^(a) Velocity		Flooding Frequency, (cycles/min)	Data at This Percent of Flooding				
			$\bar{V}_a$, (cm/s)	$\bar{V}_o$, (cm/s)		Aqueous/ Organic Ratio	Percent of Flooding Frequency	HETS (Thorium basis), ft, (m)	Arithmetic Decontamination Factor for Uranium	
18 ^(b)	(5.5)	1190	0.51	0.83	120	0.62	80	3.5	(1.07)	9.1
18 ^(b)	(5.5)	1190	0.50	0.84	120	0.60	80	3.0	(0.91)	9.7
15	(4.6)	865	0.37	0.61	40	0.61	80	2.5	(0.76)	10.3
15	(4.6)	1220	0.50	0.88	36	0.56	83	2.1	(0.64)	14.2
15	(4.6)	1375	0.60	0.95	30	0.64	82	3.3	(1.01)	8.3
15	(4.6)	872	0.37	0.62	48	0.60	81	3.0	(0.91)	14.4
15 ^(b)	(4.6)	1145	0.49	0.80	115	0.61	80	4.0	(1.22)	8.7
15	(4.6)	618	0.24	0.46	55	0.52	76	3.1	(0.94)	15.2
15 ^(c)	(4.6)	533	0.21	0.39	58	0.52	80	3.1	(0.94)	9.8
15 ^(c)	(4.6)	547	0.21	0.41	58	0.52	76	2.9	(0.88)	11.8
15 ^(c)	(4.6)	574	0.25	0.40	57	0.62	80	3.5	(1.07)	--
15 ^(c)	(4.6)	572	0.25	0.40	57	0.61	80	5.0	(1.52)	7.8
15	(4.6)	601	0.26	0.42	56	0.62	80	3.15	(0.96)	7.8
15 ^(c)	(4.6)	623	0.25	0.45	55	0.56	91	2.95	(0.90)	7.6
15 ^(c)	(4.6)	610	0.25	0.44	56	0.58	89	3.8	(1.16)	7.2
15	(4.6)	547	0.26	0.36	59	0.72	83	3.7	(1.13)	4.5
15 ^(c)	(4.6)	566	0.25	0.39	58	0.63	84	4.0	(1.22)	7.1
15	(4.6)	613	0.25	0.44	56	0.57	82	2.9	(0.88)	13.5
15	(4.6)	618	0.24	0.46	56	0.53	82	2.8	(0.85)	12.8
15	(4.6)	614	0.26	0.43	56	0.62	79	3.3	(1.01)	5.0
15	(4.6)	599	0.25	0.43	56	0.59	79	3.4	(1.04)	7.0
15	(4.6)	599	0.25	0.43	56	0.58	79	3.4	(1.04)	12.5
15	(4.6)	581	0.25	0.41	57	0.60	90	3.0	(0.91)	9.2
15	(4.6)	572	0.23	0.42	58	0.56	83	2.5	(0.76)	5.2
15	(4.6)	598	0.24	0.44	56	0.56	79	3.3	(1.01)	8.0
15	(4.6)	597	0.24	0.43	56	0.56	79	3.2	(0.98)	8.7

(a) $\bar{V}_a + \bar{V}_o = \bar{V}_T$; Volume Velocity (gallons/hr/ft²) = 885 $\bar{V}_T$.

(b) Organic phase continuous.

(c) DBP (dibutyl phosphate) was added to the 1A column during this pilot plant run.

TABLE B-4
HETS DATA 1BS COLUMN

Total Cartridge Height, ft, (m)	Volume (a) Velocity, (gal/hr/ft ²)	Superficial (a) Velocity		Flooding Frequency, (cycles/min)	Data at This Percent of Flooding				
		$\bar{V}_a$, (cm/s)	$\bar{V}_o$, (cm/s)		Aqueous/ Organic Ratio	Percent of Flooding Frequency	HETS (Uranium basis), ft, (m)	Arithmetic Decontamination Factor for Uranium	
18 (5.5)	595	0.50	0.17	80	3.0	80	2.6 (0.79)	98	
18 (5.5)	925	0.78	0.27	64	2.9	80	2.6 (0.79)	37	
15 (4.6)	950	0.70	0.37	58	3.0	83	2.1 (0.64)	163	
15 (4.6)	940	0.80	0.26	60	3.0	80	-- --	--	
15 (4.6)	714	0.61	0.20	82	3.1	~80	2.0 (0.61)	167	
17 (5.2)	610	0.54	0.15	75	3.5	~80	1.9 (0.58)	1073	
17 (5.2)	534	0.46	0.14	78	3.3	70	2.7 (0.82)	223	
17 (5.2)	543	0.46	0.15	78	3.2	78	2.8 (0.85)	632	
17 (5.2)	608	0.55	0.14	86	4.0	70	2.4 (0.55)	284	
17 (5.2)	662	0.58	0.17	64	3.5	90	2.0 (0.61)	476	
17 (5.2)	667	0.56	0.19	63	3.0	84	2.4 (0.73)	359	
17 (5.2)	624	0.57	0.14	65	4.2	83	1.6 (0.49)	299	
17 (5.2)	640	0.58	0.14	64	4.1	80	2.0 (0.61)	107	
17 (5.2)	641	0.55	0.19	64	3.3	80	2.4 (0.73)	195	
17 (5.2)	661	0.57	0.18	84	3.1	60	2.2 (0.67)	311	
17 (5.2)	646	0.55	0.18	84	3.0	60	2.1 (0.64)	295	
17 (5.2)	678	0.60	0.17	84	3.5	67	1.6 (0.49)	974	
17 (5.2)	662	0.57	0.18	84	3.1	67	2.0 (0.61)	597	
17 (5.2)	629	0.56	0.15	84	3.7	67	1.7 (0.52)	407	
17 (5.2)	638	0.56	0.16	85	3.4	64	3.0 (0.91)	45	
17 (5.2)	608	0.52	0.17	86	3.1	63	3.0 (0.91)	156	
17 (5.2)	643	0.55	0.18	85	3.0	67	2.4 (0.55)	1090	
17 (5.2)	642	0.55	0.18	85	3.0	67	3.0 (0.91)	173	

(a) $\bar{V}_a + \bar{V}_o = \bar{V}_T$; Volume Velocity (in gallons/hr/ft²) = 885 ($\bar{V}_T$).

TABLE B-5
HETS DATA 1C COLUMN

Total Cartridge Height, ft, (m)		Volume ^(a) Velocity, (gal/hr/ft ²)	Superficial ^(a) Velocity		Flooding Frequency, (cycles/min)	Data at This Percent of Flooding					
			$\bar{V}_a$, (cm/s)	$\bar{V}_O$, (cm/s)		Aqueous/ Organic Ratio	Percent of Flooding Frequency	HETS (Uranium basis), ft, (m)		Percent Uranium Loss	Temperature (°C)
18	(5.5)	1050	0.39	0.80	35	0.48	~90	4.7	(1.43)	0.1	Ambient
15	(4.6)	1310	0.49	0.99	60	0.50	90	3.2	(0.98)	0.06	48
15	(4.6)	870	0.33	0.65	98	0.50	95	4.5	(1.37)	0.08	43
15	(4.6)	1650	0.64	1.22	50	0.52	80	<4.7	(<1.43)	--	43
15	(4.6)	1595	0.62	1.18	42	0.52	92	<4.0	(<1.22)	0.04	44
15	(4.6)	872	0.32	0.67	67	0.46	84	3.0	(0.91)	0.05	42
15	(4.6)	1369	0.51	1.04	35	0.49	80	3.3	(1.01)	0.006	46
15 ^(b)	(4.6)	1189	0.45	0.89	55	0.51	70	3.8	(1.16)	0.10	47
15 ^(b)	(4.6)	1204	0.43	0.93	50	0.46	80	3.9	(1.19)	0.04	48
15 ^(b)	(4.6)	1196	0.45	0.90	50	0.50	80	4.0	(1.22)	0.02	48
15 ^(b)	(4.6)	1183	0.43	0.91	50	0.48	80	5.0	(1.52)	1.27	48
15	(4.6)	1262	0.48	0.95	~40	0.51	90	3.4	(1.04)	0.40	46
15 ^(b)	(4.6)	1327	0.60	1.29	50	0.47	70	--	--	0.25	46
15 ^(b)	(4.6)	1242	0.42	0.98	56	0.43	63	--	--	2.13	46
15	(4.6)	1157	0.50	0.81	61	0.62	61	4.5	(1.23)	1.5	49
15 ^(b)	(4.6)	1202	0.48	0.88	58	0.52	79	4.4	(1.34)	4.6	49
15	(4.6)	581	0.22	0.44	95	0.49	71	4.7	(1.43)	3.0	52
15	(4.6)	591	0.21	0.46	95	0.47	71	4.1	(1.25)	0.22	52
15	(4.6)	574	0.22	0.43	104	0.51	62	3.0	(0.91)	0.04	45
15	(4.6)	570	0.22	0.42	104	0.52	62	3.4	(1.04)	0.11	45
15	(4.6)	570	0.22	0.42	104	0.52	62	3.2	(0.98)	0.11	45
15	(4.6)	548	0.21	0.41	97	0.51	79	3.3	(1.01)	0.004	49
15	(4.6)	550	0.21	0.41	97	0.50	79	3.1	(0.94)	0.013	49
15	(4.6)	571	0.21	0.44	96	0.49	73	3.8	(1.16)	0.05	49
15	(4.6)	578	0.22	0.43	95	0.51	74	3.0	(0.91)	0.05	49

(a) $\bar{V}_a + \bar{V}_o = \bar{V}_T$; Volume Velocity (in gallons/hr/ft²) = 885 $\bar{V}_T$.

(b) DBP (dibutyl phosphate) was added to the 1A column during this pilot plant run.