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Chemistry-Separation Processes for Plutonium and Uranium

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CONTINUOUS SOLVENT RECOVERY PROCESS USING PULSE CONTACTING COLUMN

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CHEMICAL TECHNOLOGY DIVISION

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CONTINUOUS SOLVENT RECOVERY PROCESS USING PULSE CONTACTING COLUMN

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Experimental Work By:

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1.0 ABSTRACT

This report presents the progress in the laboratory development of a continuous solvent recovery process for the tributyl phosphate-diluent mixture used in the Purex Process. Investigations were made with ½" diameter pulse columns, using a nine foot sodium carbonate contacting column and a four foot water wash column.

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2.0 INTRODUCTION

The solvent used in the Purex Process consists of a mixture of 30% tributyl phosphate and 70% Amsco, a hydrocarbon diluent. This organic mixture acquires the following contaminants in the Purex Process:[8] plutonium, uranium, fission product activity, nitric acid, butyl alcohol, and mono and dibutyl phosphates. The uranium and plutonium represent a small loss to the solvent during the extraction cycle, while the nitric acid and fission product activity are present because of a slight solubility of these materials in the organic phase. The mono and dibutyl phosphates and butyl alcohols are present as impurities in new solvent or are introduced by the hydrolysis of the tributyl phosphate.

The plutonium and beta activity present a hazard to operating personnel when large quantities of the organic are stored in unshielded areas, and the residual uranium is detrimental if the organic is to be used in certain scrubbing operations in the Purex Process. The presence of butyl alcohols, and mono and dibutyl phosphates in the organic mixture, increase greatly the solvent extraction of plutonium and fission products. All of these impurities are removed rapidly from the solvent by sodium carbonate except fission product activity which requires considerable contact time.

The specifications for recovered organic, suitable for reuse in the process, are: beta activity, less than 300 c/m/ml; uranium, 0.00005 mg/ml; total acid, including mono and dibutyl phosphates, nil; alpha activity less than 10 c/m/ml, and a reducing normality less than 0.001, (i.e., free of butyl alcohols). Specifications for the diluent is that the Kauri Butanal number[7] is low, (i.e., in the order of 30).

[redacted]

Until the inception of this program, the solvent was reconditioned, or recovered, by a batch process consisting of solvent washing with sodium carbonate and water.[3-4] This process required a large solvent inventory, large tanks for organic storage, a long contact time, appreciable operating floor space, and personnel, and high cost for equipment. In addition several batch contactings were necessary to obtain adequate solvent decontamination.

In order to improve the overall effectiveness of solvent clean-up, a continuous solvent recovery process seemed desirable, so a program was initiated employing the optimum chemical conditions of the batch process and using the pulse column as the contacting unit for the sodium carbonate and used organic.[6]

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3.0 SUMMARY

A continuous solvent Recovery Process has been developed, removing the deleterious impurities from the Purex Process used organic. The operability of the process was satisfactorily demonstrated with organic feeds solutions from ORNL and Hanford level runs. The fission activity remaining in the treated organic was 125 and 300 c/m/ml, respectively, corresponding to a decontamination factor of about 100.

The investigation was carried out in a $\frac{1}{2}$" diameter pulse column[1-2] having a nine feet carbonate contacting and a four feet water wash section, using 0.10M sodium carbonate, at about 50°C, as the contacting agent and water, at room temperature, as wash to remove entrained sodium carbonate solution. The contacting time required for good activity removal was obtained by operating the carbonate contacting column with the organic as the continuous phase, giving 18-36 minutes contacting time in the column.

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4.0 APPARATUS

The column assembly, consisting of lines, tanks, column end plates and column plates, used in the continuous solvent recovery investigation were fabricated of stainless steel.

1. Tanks

The tanks were connected in series, an air line pressurizing each tank with filtered, dried air to a pressure of six pounds/square inch (Figure 1) thus eliminating the pumps necessary for feeding solutions into the column.

2. Columns

In the investigations of the continuous solvent recovery process, two types of column arrangements were used; a compound column and a two-column system. In each case the column consisted of a Pyrex glass column 0.506 ± 0.002" in diameter. When employed as a compound column, the lower six feet were used for the carbonate contacting section, with the top three feet being the water wash section (Figure 4). When employed as a simple column, in the two column system, the entire nine feet were used as the carbonate contacting section, and the organic was water washed in a second, four feet long, one half inch in diameter column (Figure 2).

3. Column Heating Unit

Each three foot segment of the carbonate contacting section of the cclumn was wrapped with nichrome ribbon 1/8" wide and having a resistance of 2.549 ohms/foot. The two lower sections of the column were connected in series with a common variac regulating the current to the heating units, thus controlling the column temperature. The carbonate contacting

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FIGURE 1

Tank assembly showing funnel, valves, compressed air line and sight glass. The sight glass is used for checking flow rates by feeding directly from the content of the sight glass.

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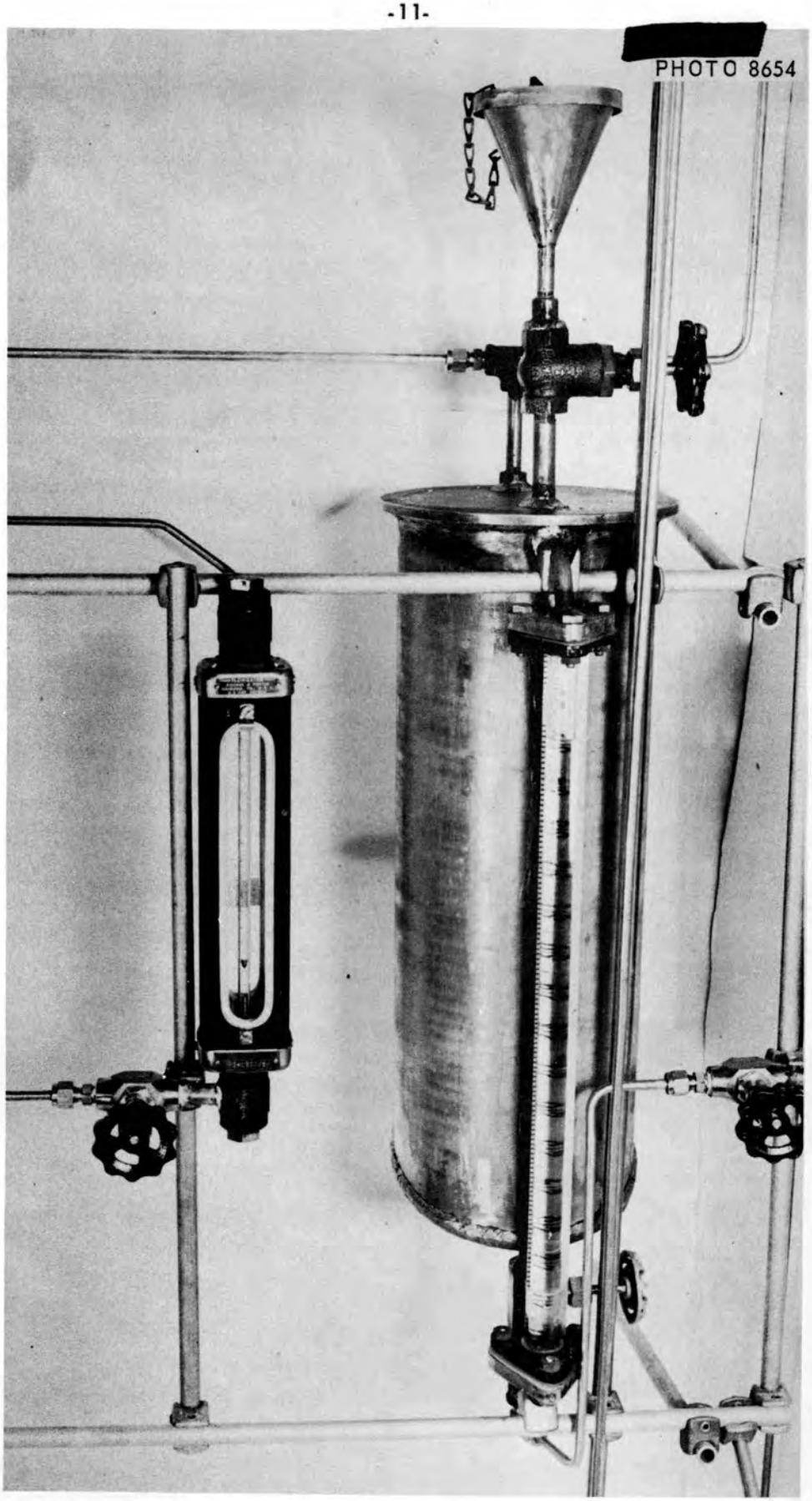
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FIGURE 4

The entire nine feet of carbonate contacting column showing penthouse atop the "walk-in" hood. The carbonate feed tank was located in the attic to obtain the necessary head for operation. The contacted organic was cascaded directly to the four foot water wash column.

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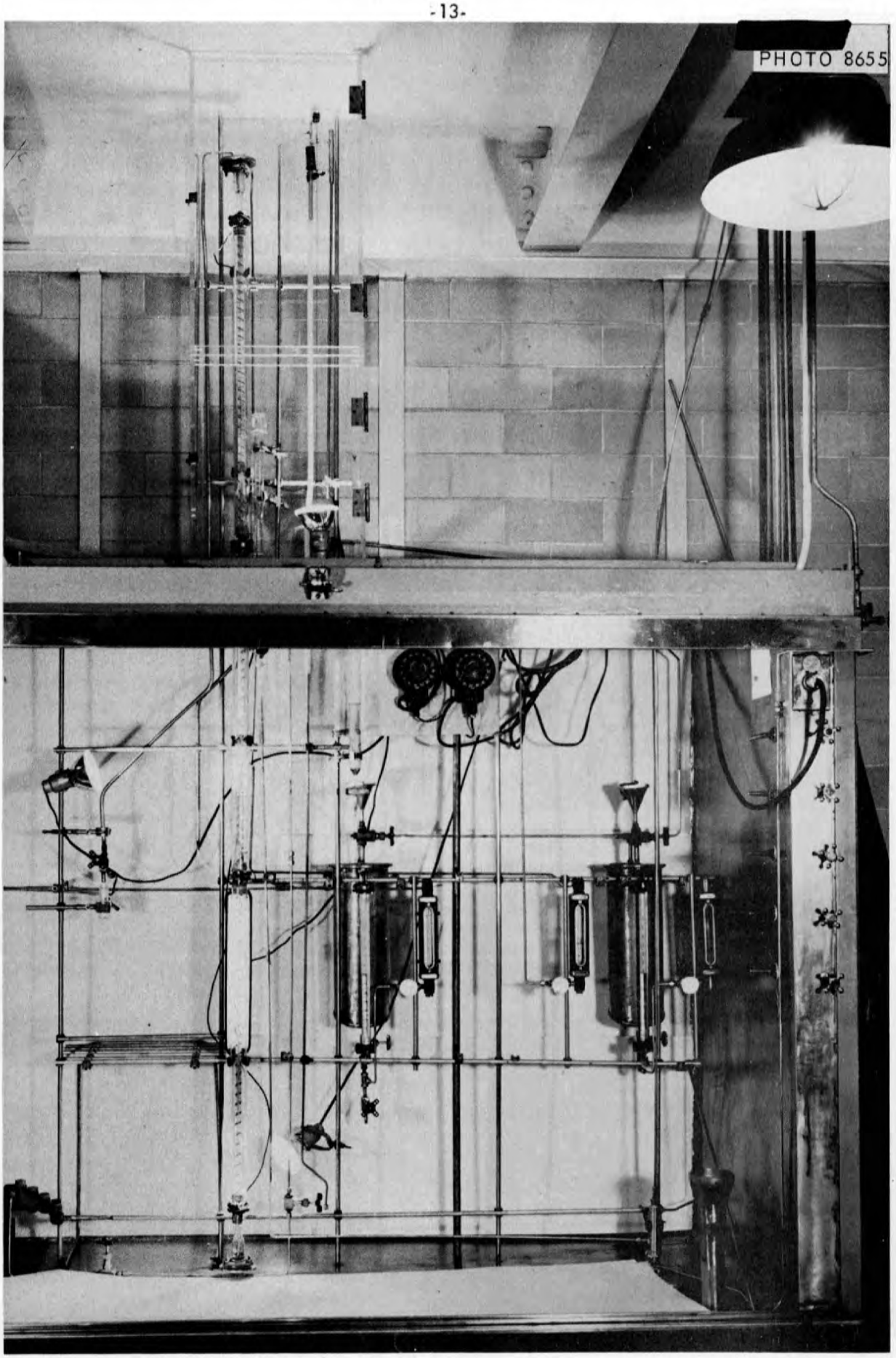
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FIGURE 2

Water wash column showing tanks, rotometers jackleg assembly and pulse generator for the wash column. The wash column was employed as a pulse column having the organic as the continuous phase.

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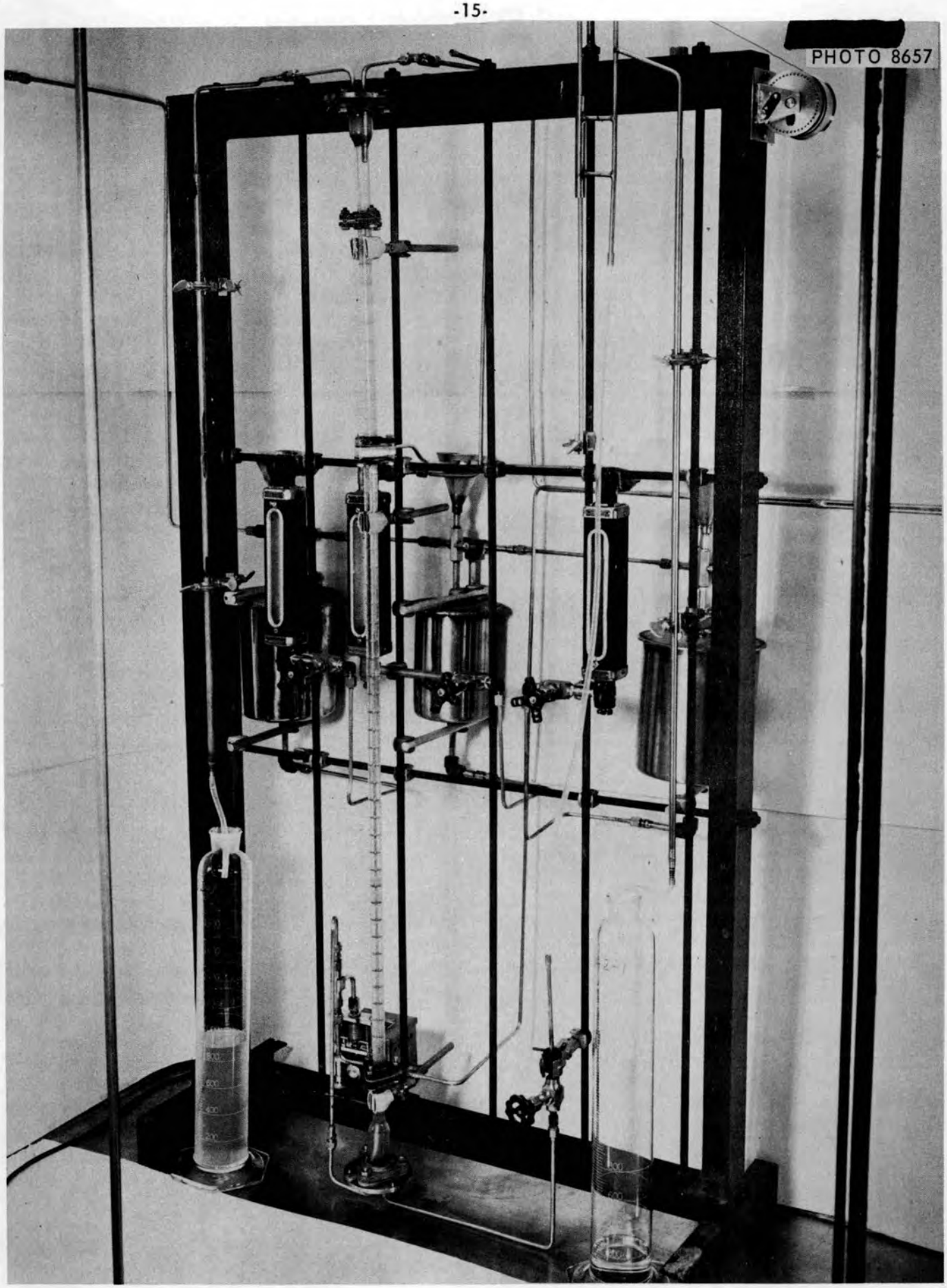
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column and heating units were located inside a one and one half inch diameter glass column (Figure 3) to protect the heating unit and maintain a constant temperature of 45°C around the contacting column.

4. Column Assembly

The column assembly shown in Figure 4 is typical for a one half inch pulse column with the exception of the organic overflow which leaves the top of the column through a one-eighth inch diameter line and spills into a one-fourth inch diameter line to prevent syphoning. Also, the organic from the carbonate contacting column cascaded directly into the pulse generator of the water wash column, thus eliminating an organic catch tank, rotometer and feed pump.

5. Plates

The plates were made from 22 gauge stainless steel disks 0.504" in diameter and had 32 holes, 0.039" in diameter drilled on an equilateral triangular patter (Figure 5). A 3/32" diameter hole was drilled through the center of the plate, and the plate assembly rod, connecting the 36 plates in the assembly, passed through this opening. Each plate had a 20-21%"free space"or open area.

6. Plate Assembly

The plate assembly consisted of a stainless steel rod 36" long, 1/16" in diameter upon which the plates were spaced one inch apart by means of stainless steel spacers. The entire assembly was then placed inside the one-half inch diameter glass column.

7. Pulse Generators

During the investigations for the continuous solvent recovery

[redacted]

FIGURE 3

The column heating unit for the 1/2" column located inside a 2" column with thermometer. The nichrome ribbon 1/8" wide had a resistance of 2.5 ohms/foot. The picture shows tiny bubbles existing within the column.

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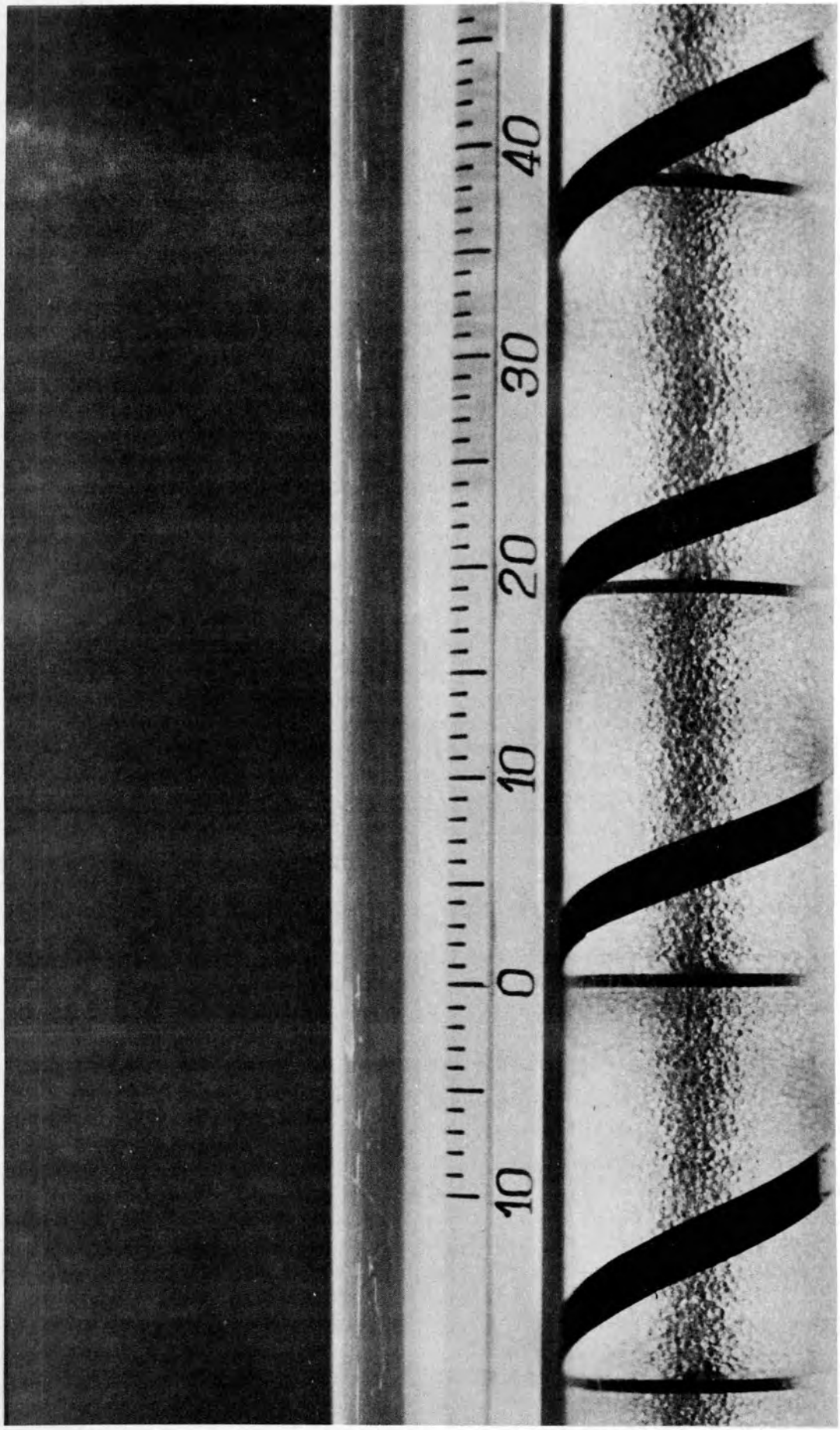

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FIGURE 5

Column plates showing open space or "free area" - also thickness of the plates. The plates are 22 gauge stainless steel 0.504" in diameter having 32 holes 0.039" in diameter. The plates have 21% free area.

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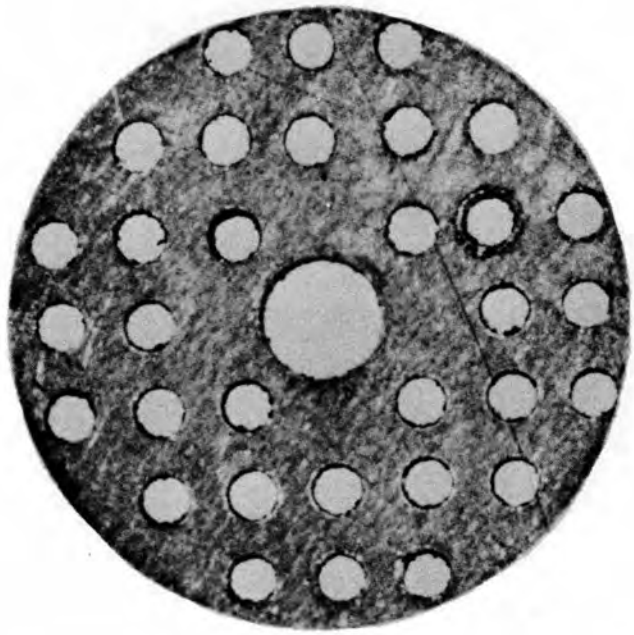
PHOTO P-108 (A)



process, two types of pulse generators were used. For the 9' carbonate contacting column a 1/4 H.P., 38 RPM Milton Roy pump was used with the two check balls from the outlet side removed to obtain the desired "push-pull" effect upon the solutions within the column. The piston stroke could be regulated from 0-3" giving an amplitude in the column from 0-1.20". The 4' water wash column was pulsed by a 3/4" bellows pump activated by a 1/8 H.P., 60 RPM motor and producing an amplitude in the column from 0-1.10" (Figure 6).

8. Air-Jet

An air jet was used as an air lift to transfer the used carbonate solution to the head tank, located in the attic in order to permit gravity feed to the carbonate contacting column (Figure 7).

9. Jackleg Assembly

The jackleg for the carbonate contacting column, operated with the organic as the continuous phase, was about 5' in length forming a "T" at the top with the right arm of the T being 2-3 inches lower than the left arm. The right arm of the "T" had a regulating valve in the line which permitted the passing of a 1/5 volume of used carbonate solution continuously to "waste", and allowed the remaining 4/5 to spill directly into the carbonate catch tank. Above, and spilling directly into the carbonate catch tank, was the "fresh carbonate" make up tank, from which a 1/5 volume of fresh carbonate solution spilled directly into the used carbonate in the carbonate catch tank (Figure 8). Located near the bottom of the jackleg was an interface control valve used to control the interface position by controlling the amount of solution leaving the column through the jackleg.

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FIGURE 6

The pulse generator for the water wash column. A 3/4" Bellows pump, 1/8 H.P. motor geared to 60 RPM. The pulse amplitude was regulated by raising or lowering the bar directly above and activated by the cam.

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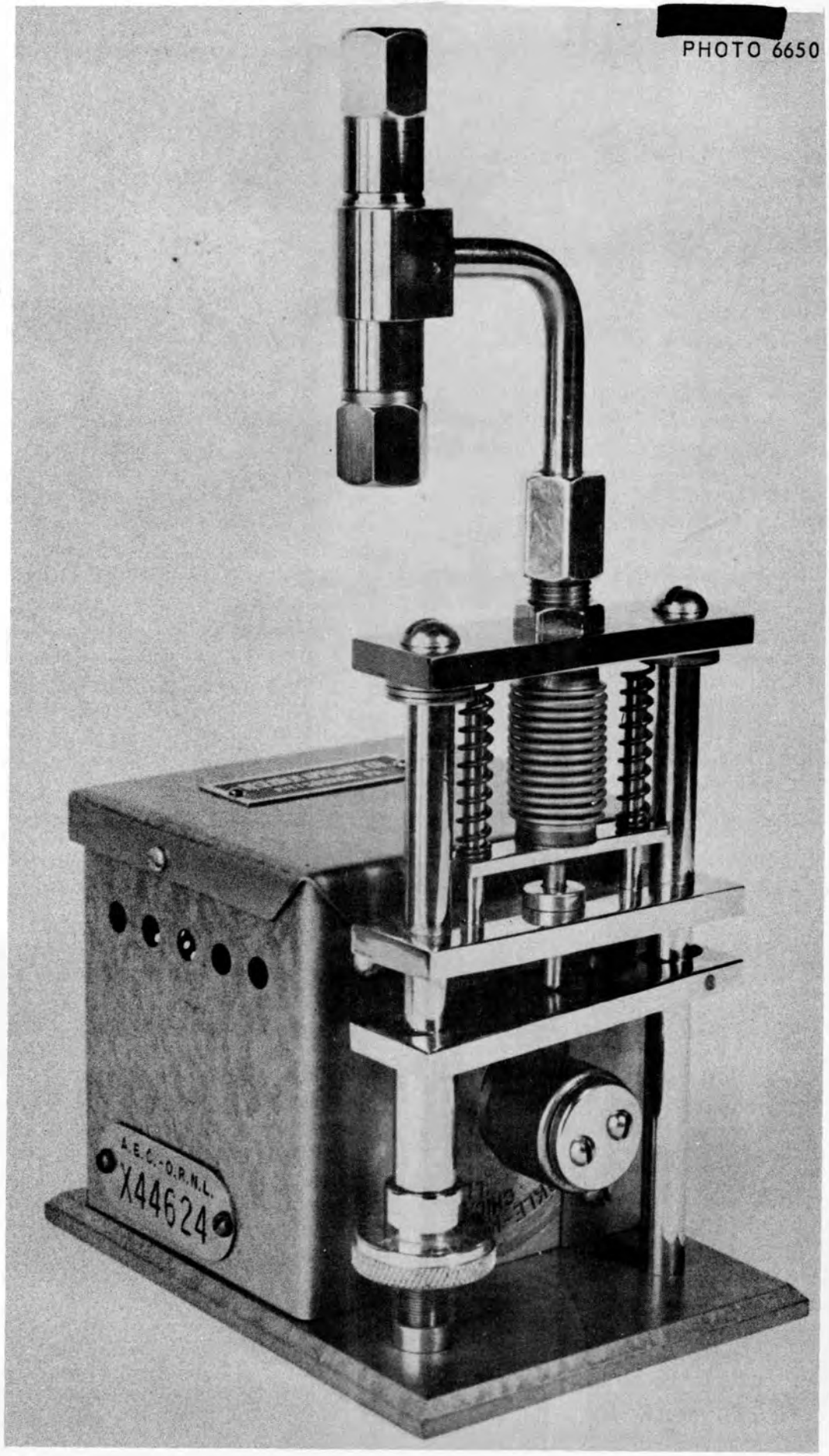

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FIGURE 7

Air Jet - used as an "Air Lift" to lift the used carbonate solution to the Carbonate head tank located in the attic.

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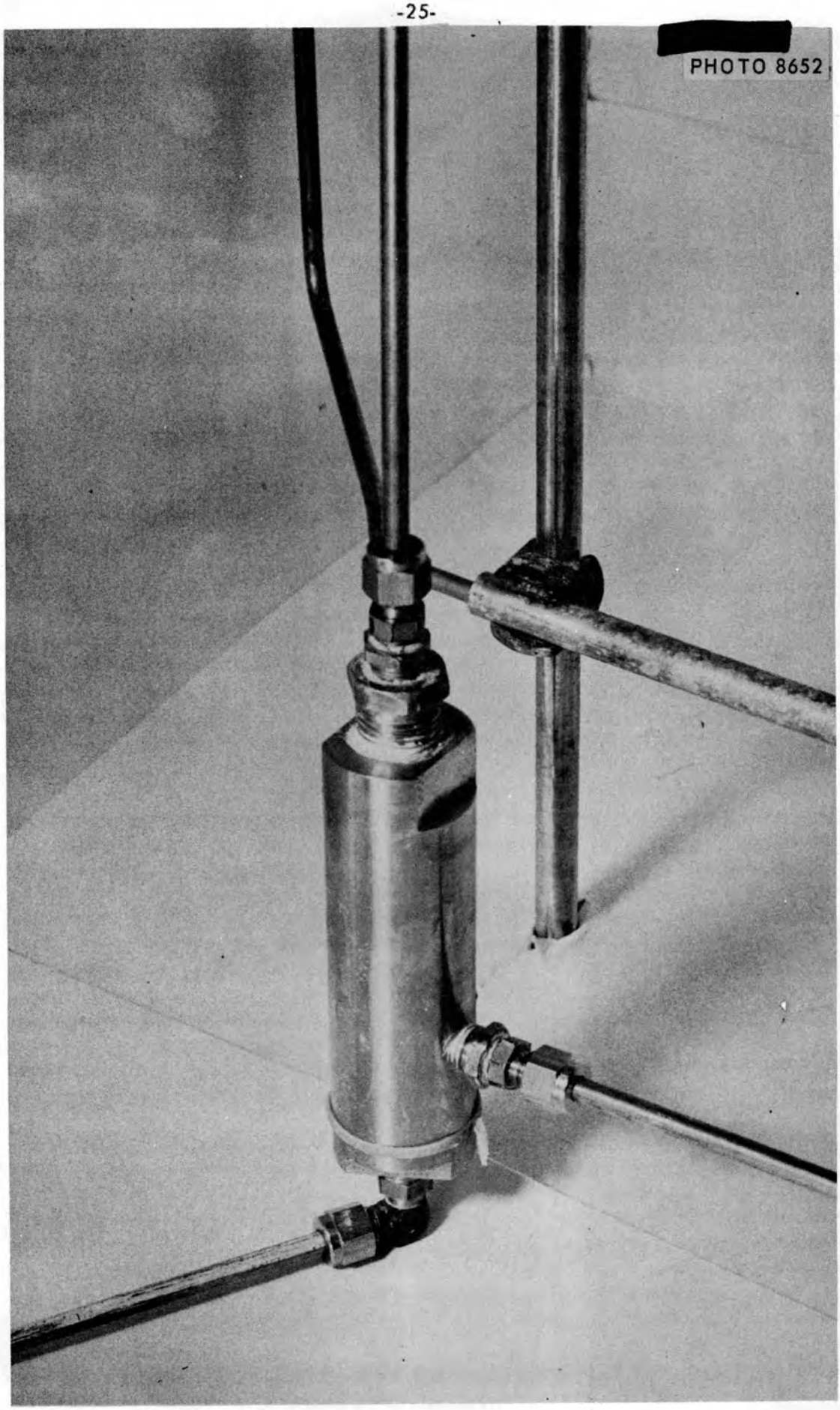

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FIGURE 8

Top of the jackleg assembly - the right side of the "T" controls the used carbonate to waste while the remaining 4/5 empties into the glass receptacle on the left. Above and emptying into this used carbonate receptacle is the "fresh" carbonate solution, adding a 1/5 volume to the used carbonate solution keeping the reagent volume and concentration constant.

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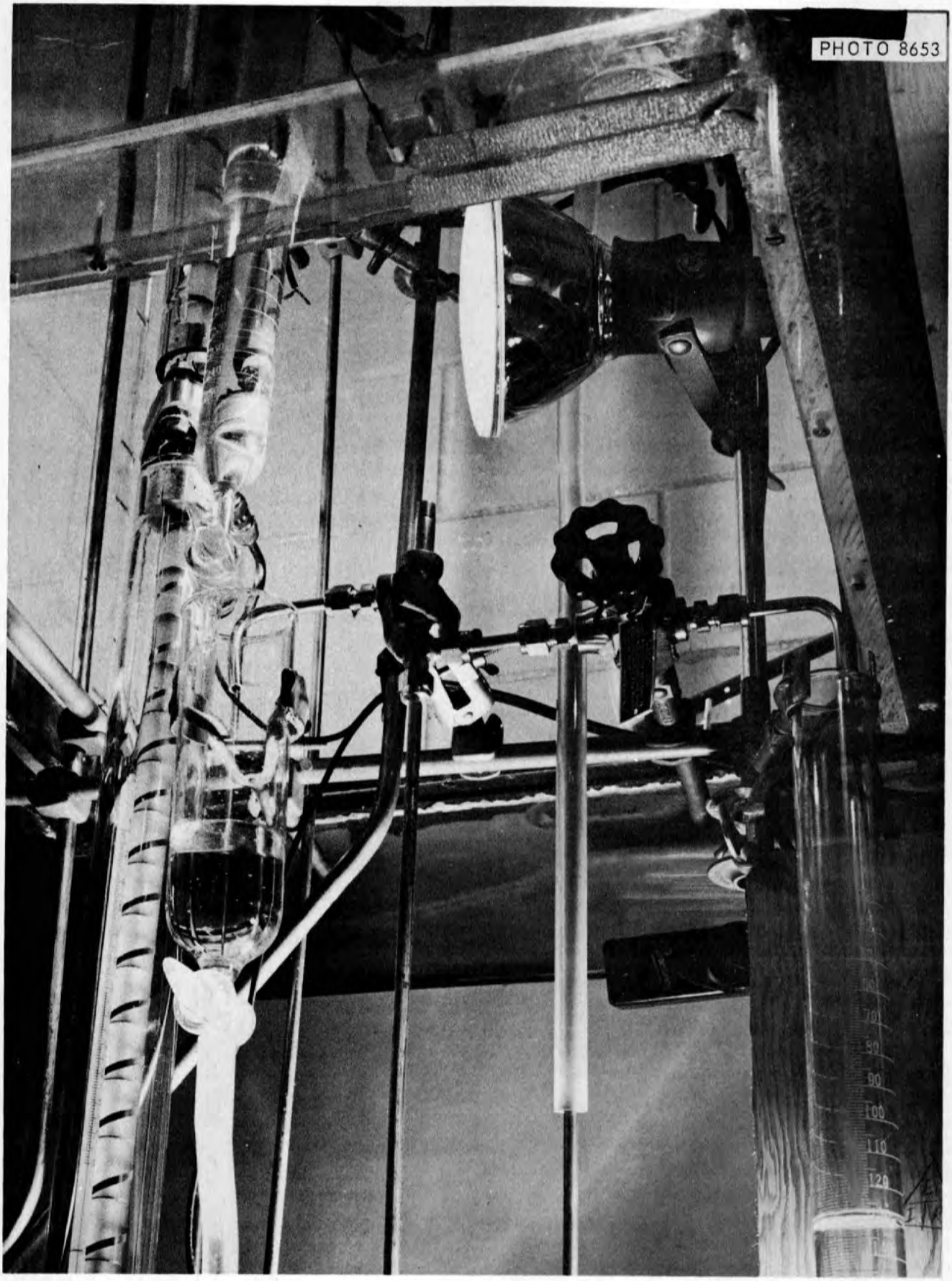
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5.0 PROPOSED PROCESS

5.1 Recommended Flowsheet

The recommended process for the recovery of used solvent consisted of the following: The used organic from the Purex Process passed to an organic head tank. Five volumes of this organic passed through a rotometer, and were pulsed directly into the bottom of the carbonate contacting column. One volume of 0.10 molar sodium carbonate solution was fed into the top of the column, descending countercurrently through the continuous organic phase. Pulsing in the column was accomplished with a Milton Roy Pump having a three inch stroke and giving a pulse amplitude in the column of 1.2". The five volumes of contacted organic upon leaving the contacting column cascaded directly to, and was pulsed into, the bottom of the packed, water wash column where it was contacted with one volume of demineralized water. Ascending the water wash column, the organic overflowed into an organic catch tank where the tributyl phosphate content was determined and adjustments made if needed. The organic was then ready for reuse.

The used carbonate solution leaving the contacting column through the jackleg was divided, with a 1/5 volume being concentrated and sent to waste storage, while the remaining 4/5 volume was blended with a 1/5 volume of fresh carbonate solution and air lifted to the head tank for recycle.

The water wash may be recycled in the same manner as the carbonate solution with the 1/5 volume of water wash being used to prepare the carbonate solution or, alternatively, concentrated and stored as waste.

The optimum solvent contacting solution for the continuous Solvent Recovery Process was established as a 0.10M sodium carbonate solution.

[redacted]

The molarity of the carbonate solution used was not found to be critical between the range of 0.10-0.50 molar, but molarities of 1.5-2.0 molar produced some "salting back" effect of the fission activity. (Figure 10)

Water was found to be the optimum wash for removing the entrained sodium carbonate solution from the treated solvent. In addition to removing entrained sodium carbonate, which was undesirable used with solutions containing plutonium, water washing, also removed 300-800 c/m/ml of fission product activity leaving only 200-300 c/m/ml of which 90-95% was ruthenium. Nitric acid was investigated as a wash in this column and it was not found to be more effective than water.

5.2 Recommended Equipment

Optimum column operation was obtained with the two-column system operation. The two-column system consisted of a nine feet column for the carbonate contacting and a four feet column for water washing. The carbonate contacting column was operated with the organic as the continuous phase, while the water wash column was operated with the aqueous as the continuous phase.

Two types of pulse generators were investigated for the carbonate contacting column; a 60-cycle, 60 RPM, 3/4" Bellows pump, which produced a stable emulsion in the column until the amplitude was reduced to 0.30". The 38 RPM Milton Roy Pump having a piston stroke of 0-3" gave a pulse amplitude of 0-1.2". The best results were obtained with the Milton Roy Piston Pump operating with a pulse amplitude of 1.2".

In both column systems the plates for the columns were 0.504" in diameter, spaced one inch apart, containing 32 holes 0.039" in diameter.

The Continuous Solvent Recovery Process required a jackleg arrangement providing a continuous "draw off" of the used carbonate solution and a continuous addition of fresh carbonate solution. The continuous addition and withdrawal maintains a constant volume and carbonate concentration, making the process a continuous one. This arrangement was accomplished by dividing the carbonate stream by means of a jackleg having two outlets; one two inches below the other and containing a valve regulating the 1/5 volume of continuous draw off.

The column heating unit consisted of a nichrome ribbon 1/8" wide and having 2.549 ohms/ft. resistance. This high resistance ribbon reduced the ribbon required to heat the column, and thereby provided the maximum in column visibility. The ribbon was wrapped spirally around the 1/2" glass column, spirals 1" apart, and the three feet section of column had total resistance of 15 ohms. The nichrome ribbon was attached to and controlled by a variac, maintaining a temperature of about 45°C in the column. This heated column was placed inside a 1-1/2" diameter glass column to maintain an even temperature and for protection of both operator and heating unit.

5.3 Recommended Operating Conditions

In the Solvent Recovery Process, either batch or continuous, the contact time for the sodium carbonate solution with the used organic is an important factor in the removal of the fission activity from the organic.[4] The uranium and nitric acid removal occurs rapidly and this reaction consumes the bulk of the reagent used in the process. Runs were made investigating the effect of column retention time upon the removal of fission activity. These runs showed the reaction to be time dependent and succes-

sive contactings, such as in column with the aqueous phase continuous, did not successfully remove the fission activity. Inverting the phases in the carbonate contacting column and thereby operating with the organic phase continuous, increased the contact time from four minutes to thirty-six minutes. The increase in contacting time increased the beta activity decontamination factor two-fold. By reducing the flow rates from 10.5 to 7.5 ml/min. and operating an organic continuous column, the decontamination factor was increased slightly but not enough to warrant the use of the smaller flow rate. (Figure 11)

The two-column system of operation was selected because it has several advantages over the compound column. (1) There is no dilution of the carbonate solution by the water wash and the carbonate and water wash solutions may be recycled a miximum of five times successfully. (2) The two-column system gives longer contact time for each operation.

Two important factors in determining flooding in the two-column system were found to be temperature and pulse amplitude. Operating with the column heated to 45°C and a pulse amplitude of 1.2 inches the column showed no signs of flooding at a throughput of 700 gal./hr./sq. ft.

It is recommended that the carbonate contacting column be heated to 45°C to accelerate the reaction between fission activity and the sodium carbonate solution. This was the maximum feasible temperature, and above 50°C there was considerable bubbling or gassing of the carbonate solution, causing disturbance of equilibrium within the column. Runs were made investigating the effect of heat upon fission activity removal, and the heated column was found to be superior to the unheated column by a factor

of two. Heat also permitted operation of the column with higher pulse amplitudes because of a lesser tendency to form emulsions. (Figure 12)

5.4 Conclusions

Data from the previously mentioned work not only proved the continuous Solvent Recovery Process to be feasible, but to have the following advantages over the Batch Process:

1. A beta decontamination factor of only 30 is obtained with the Batch Process compared to 100 obtained with the Continuous Process.

2. The initial cost of the Batch Process is greater than that of the Continuous Process.

3. The Continuous Process requires less operating personnel than batch operation.

4. Continuous operation requires only 8% of the organic inventory required by batch operation.

5. The Continuous Process requires less operating floor space than the Batch Process.

6. Since the solvent inventory is small in the Continuous Process, less radiation shielding is required.

[redacted]

6.0 EXPERIMENTAL INVESTIGATIONS AND RESULTS

6.1 Selection of the Continuous Phase

From batch data it is known that the solvent decontamination factor increases with increased carbonate contacting time. Increased contacting time was obtained by changing from aqueous to organic phase continuous, (Runs 1-7, Table 1 and Figure 9). Runs 1-3, inclusive were made under the flowsheet conditions given in Figure 9, utilizing the organic from an ORNL level Pilot Plant run containing 8.7×10^3 c/m/ml of fission activity.

Data from this group of runs, made with a compound column having six feet of carbonate contacting section and three feet of water wash section, may be summarized as follows:

1. Runs 1-3, inclusive, using an aqueous phase continuous gave a beta decontamination factor of 35 with about 1000 beta c/m/ml remaining in the treated organic.

2. Runs 4-7, inclusive, using an organic phase continuous gave a beta decontamination factor of 75 with 130-175 beta c/m/ml remaining in the treated organic.

These data indicate that a higher beta decontamination factor was obtained by using a pulsed carbonate contacting column with a continuous organic phase. Changing from an aqueous to organic continuous phase increased the contacting time for the carbonate from four to 18 minutes with a flow rate of 17.5 m/minute. When the flow rate reduced to 10.5 ml/minute the contacting time in the column was increased to 36 minutes, thus increasing the decontamination factor.

Using the flow ratio; organic: carbonate: water wash of 5:1:1,

[redacted]

and flows of 12.5:2.5:2.5 ml/min., the column was operated near flooding conditions for the $\frac{1}{2}$" pulse column.

6.2 Selection of the Pulse Generator

Two types of pulse generators were investigated for the carbonate contacting column during the continuous solvent recovery investigation. One was a 3/4 inch Bellows pump having a 1/8 H.P., 60-cycle motor attached to an eccentric cam which activated a 3/4" Bellows 60 cycles/minute. The Bellows was attached to the organic feed line and column, pulsing directly into the bottom of the carbonate contacting column.

The other type of pulse generator was a Milton Roy Piston Pump having a 1/4 H.P., 60-cycle motor geared to a 38 RPM pump. This also was attached directly into the organic feed line, pulsing directly into the bottom of the carbonating contacting column.

The investigating runs showed the 38 RPM Milton Roy pump to be the better pulse generator for the carbonate contacting column. Using the 38 RPM Milton Roy pump as the pulse generator for the contacting column and pulse amplitude of 1.20", a fission activity decontamination factor of 100 was obtained (Table 2). When the 60 RPM 3/4" Bellows pump was used, the pulse amplitude had to be maintained around 0.35-0.40 because of the basic conditions existing in the column to reduce emulsion formation and column flooding. With the low pulse amplitude, the beta decontamination factor was reduced from 100, obtained with the Milton Roy, to 80 with the 3/4" Bellows pump.

In both cases the outlet checks ball of the pump were removed, giving a "push-pull" effect upon the column, thus moving both solutions countercurrently through the column. Without removing the outlet check

balls, each pump applies only a direct push upon the organic with no pull upon the falling aqueous. Consequently, flooding occurred at a lower flow rate.

6.3 The Effect of Heating the Carbonate Contacting Column

The removal of beta activity from used solvent is a time controlled reaction. Therefore, the contacting time between the reagent and used organic is very important. In order to employ the short contact time available in the column most effectively, operation at elevated temperatures was resorted to.

Data obtained from the investigating runs 7-15 showed the heated column (45°C) to be far superior to the unheated column for the removal of fission activity. The treated organic from the heated column contained 250 beta c/m/ml compared to 750 beta c/m/ml remaining in the treated organic from the unheated column (Figure 12). Contacted organic, from an ORNL and a Hanford activity level run, contained 330 c/m/ml and 1600 c/m/ml of fission product activity, respectively, in the treated solvent.

6.4 The Effects of Periodic Acid upon Ruthenium Decontamination

The fission activity remaining in the treated organic was found to be 90-95% ruthenium. The effectiveness of periodic acid upon the removal of rutheium depends upon the specie of state in which the ruthenium exists in the organic. Periodic will remove one state or species of ruthenium and have very little or no effect upon another state. 0.0001 M periodic acid was found to have an overall effect that was questionable in the removal of ruthenium. (Figure 13)

Periodic acid was also added to the water wash column to investi-

gate its effect upon ruthenium removal here. The effect proved to be too unpredictable to warrant the recommendation of periodic acid in either the contacting or the water wash column.

6.5 Recycling the Sodium Carbonate Solution

From the batch investigation it was learned the carbonate solution could be reused several times satisfactorily removing the fission activity from the used organic. Using these data a series of five runs was made to investigate the number of times the sodium carbonate solution could be successfully recycled. The treated organic from Run #5 contained 250-295 beta c/m/ml, the maximum activity tolerable in treated organic. Accordingly, a maximum of five recycles for the sodium carbonate solutions is recommended.

From this series of runs the fission activity associated with the organic on repeated reuse of the sodium carbonate was established. Run #1 has 150-170 beta c/m/ml, Run #2 had 180-210 beta c/m/ml, Run #3 had 290 c/m/ml, Run #4 had 180-210 beta c/m/ml, and Run #5 had 290-260 beta c/m/ml. (Table 6.) No reason or explanation could be found for the highest activity being in the treated organic from Run #3. The uniform buildup of uranium and fission activity in the recycled sodium carbonate solution by successive recycles is shown in Figures 14 and 15.

6.6 Selection of Water Wash Column

The removal of the entrained sodium carbonate and 500-800 c/m/ml of fission activity, was an important consideration in the investigation of the type of water wash column to be used in the two-column system of the continuous Solvent Recovery Process (Table 3). From the standpoint of effectiveness, cost of operation, personnel required, maintainance, and

initial cost, the packed tower, with the solutions moving countercurrently through the column, and the aqueous continuous phase, was felt to be best.

6.61 Static Wash Column

The three types of columns investigated were: (1) A static wash column, allowing the organic to bubble through the static column with periodic removal and renewal of the water wash. This was not a continuous process and the water wash became quite contaminated with impurities removed from the solvent phase.

6.62 Packed Tower

A packed tower was employed and five volumes of organic passed countercurrently through one volume of distilled water. This type column required no pulse generator thus reducing the initial installation cost of the column.

6.63 Pulse Column Aqueous Continuous Phase

The pulse column with aqueous as the continuous phase was investigated. This proved to be very efficient but required a pulse generator for plant size operation, and did not show the additional advantages necessary to justify its use over the packed tower.

6.64 Pulse Column Organic Continuous Phase

The pulse column with the organic as the continuous phase gave the best decontamination factor of the four types of columns investigated. However, being a pulse column, the cost of a large pulse generator more than offset the slight additional decontamination factor obtained from this type of column.

[redacted]

6.7 Continuous Recycling of the Sodium Carbonate Solution

Having recycled the sodium carbonate solution five times, successfully removing the nitric acid, uranium and fission products activity from the used organic, a process was developed in which a 1/5 volume of the used carbonate solution was replaced with a 1/5 volume of fresh carbonate solution, thus rendering the process a continuous one. (Figure 16.)

For the process to operate continuously, the previously described jackleg arrangement (Figure 8) was used allowing a 1/5 volume of used carbonate solution is drawn off and concentrated for waste. A 1/5 volume of fresh carbonate solution is added to the remaining 4/5 volumes of used carbonate solution which is returned to the head tank for reuse. The fresh carbonate solution spilled directly into the used carbonate solution catch tank compensating the 1/5 volume being drawn off. The same jackleg assembly can be used with the water wash column thus making a continuous process of the water wash also. Instead of concentrating the water wash draw off, this solution may be used for the carbonate make up solution.

[redacted]

[redacted]

7.0 CONCLUSIONS AND FUTURE PROGRAM

The mechanical and chemical feasibility of the continuous solvent recovery is assured, although additional design data, particularly on the interface control mechanism for organic continuous operation and column capacity must be accumulated.

The future program should be devoted to Pilot Plant scale studies of interface control, flooding studies and scale up from the laboratory columns. The continuous Solvent Recovery Process is now being installed in the ORNL Pilot Plant and should be ready for further investigations in the near future.

[redacted]

- APPENDICES -

8.1

Dwg. #13914

SCHEMATIC FLOWSHEET FOR
CONTINUOUS SOLVENT RECOVERY PROCESS

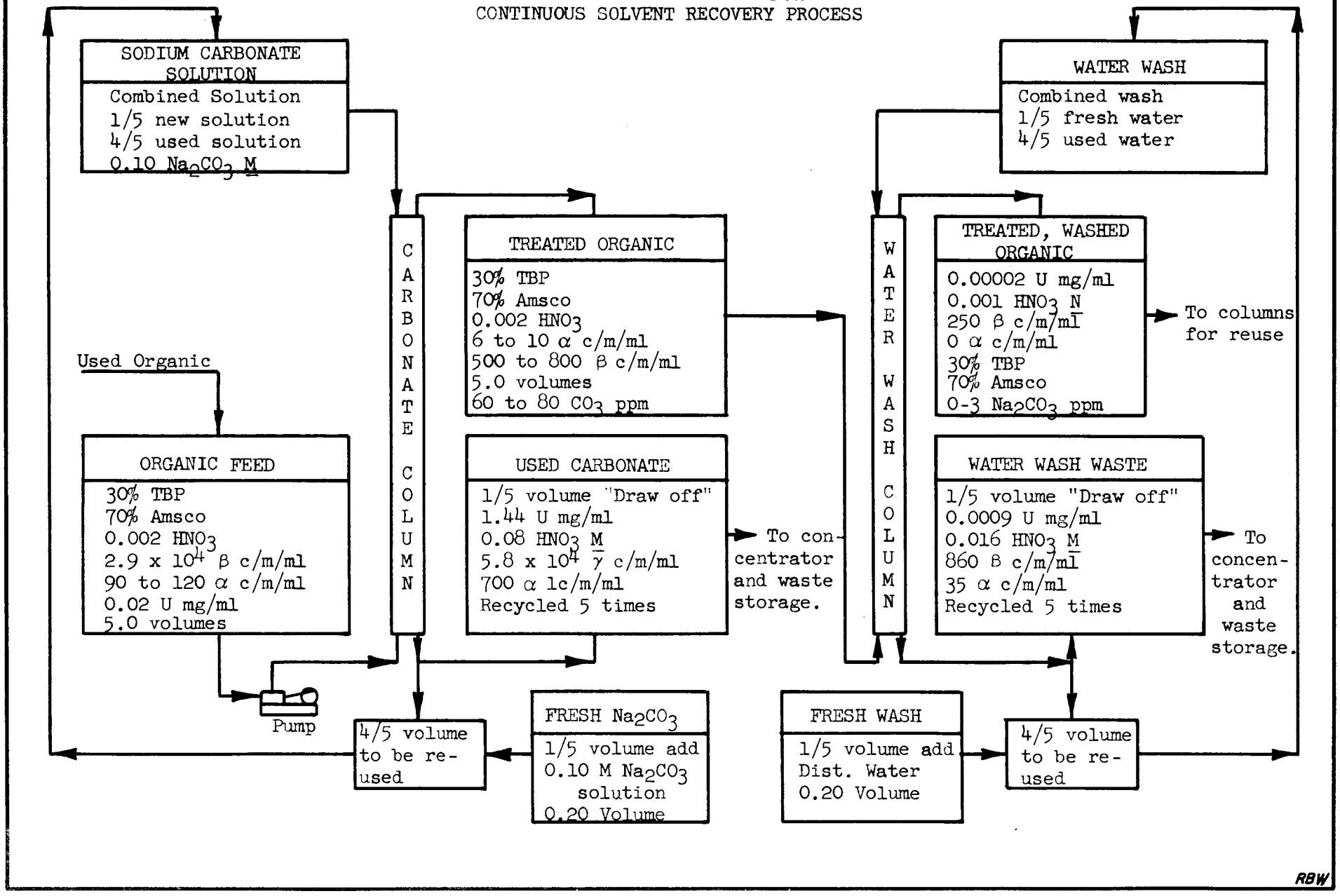


Figure 9

Dwg. #13915

Comparison of Fission Product Activity Removal under Operation of Aqueous Phase Continuous and Organic Phase Continuous

Feed - CW
U - 0.036 mg/ml
H - 0.010 N
Pu-368 c/m/ml
β - 9.9×10^3 c/m/ml
Ru- 5.8×10^3 c/m/ml

Carbonate:
Na_2CO_3 - 0.10M

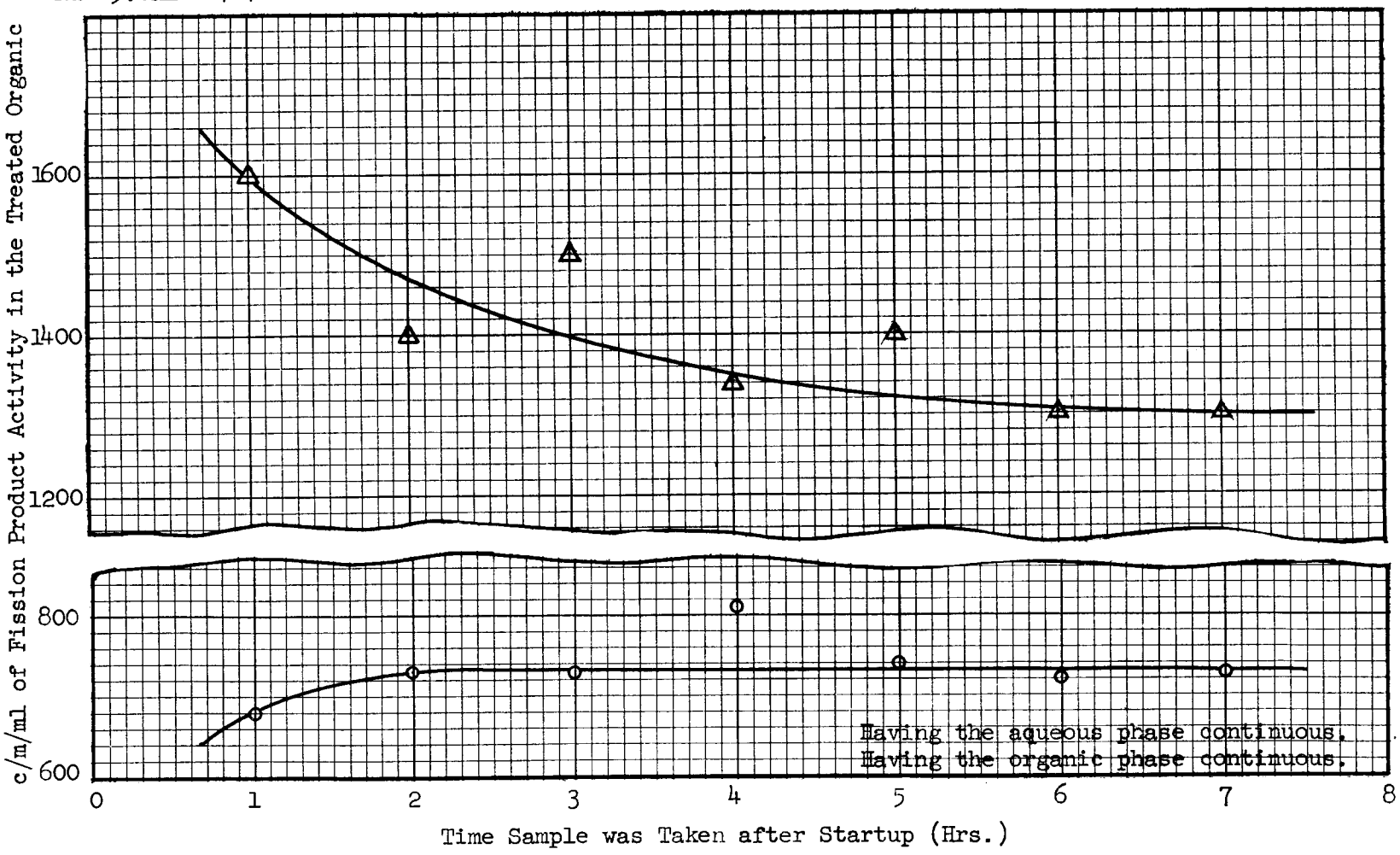
Wash:
0.10M HNO_3

Rates: Flows:
Feed-5.0-12.5 ml/min.
CO_3 -1.0- 2.5 ml/min.
Wash-1.0- 2.5 ml/min.

Column - Compound
Carbonate - 6' $\frac{1}{2}$" I.D.
Wash - 3' $\frac{1}{2}$" I.D.

Amplitude:
0.60" in Column

Plates:
1" Apart
32-Holes
0.039" in
Diameter



Dwg. #13916

Figure 10

The Effect of Sodium Carbonate Concentration upon the Beta Removal from Used Organic in the Continuous-Solvent Recovery Process Using the ½" Pulse Column

Feed: CW
U -0.014 mg/ml
H -0.011 N
Pu-174 c/m/ml
β -2.46x10^4 c/m/ml
Ru-1.1x10^4 c/m/ml

Wash:
Dist. Water

Cont. Phase:
Aqueous

Flow Rates:
Feed - 7.5 ml/min.
CO_3 - 1.5 ml/min.
Wash - 1.5 ml/min.

Temperature:
Top - 55°C
Bottom - 45°C

Column - Compound:
CO_3-Section-6' ½" ID
Wash- " -3' ½" ID

Amplitude:
1.20" in Column

Plates:
1" Apart
32 Holes
0.039" in Diameter

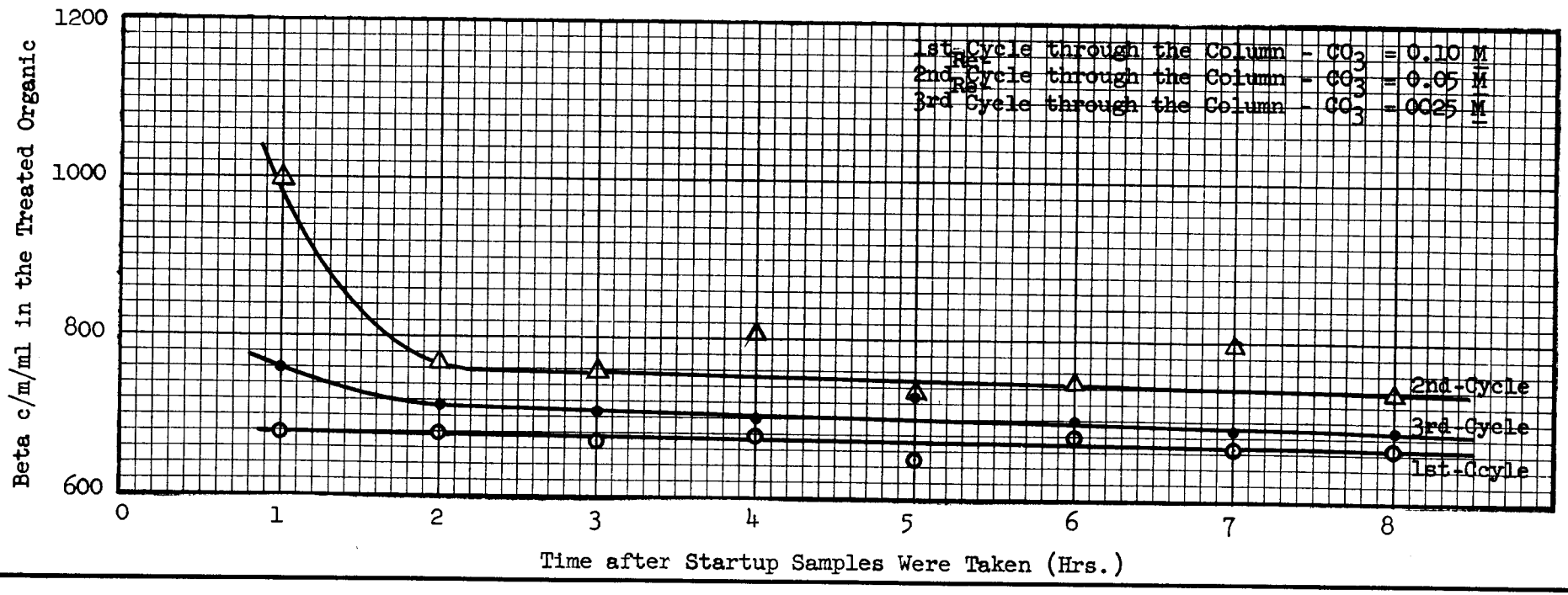


Figure 11

The Effect of Flow Rates (time in the column) upon Beta Removal

in the Sodium Carbonate Solution Using the Two Column System

Feed:
U - 0.012 mg/ml
HNO_3 - 0.004 N
Pu - 44 c/m/ml
β - 2.9×10^4 c/m/ml
Ru - 1.14×10^4 c/m/ml

CO_3:
0.10M + 0.001M $HIO_4 \cdot 2H_2O$

Wash:
Dist. H_2O

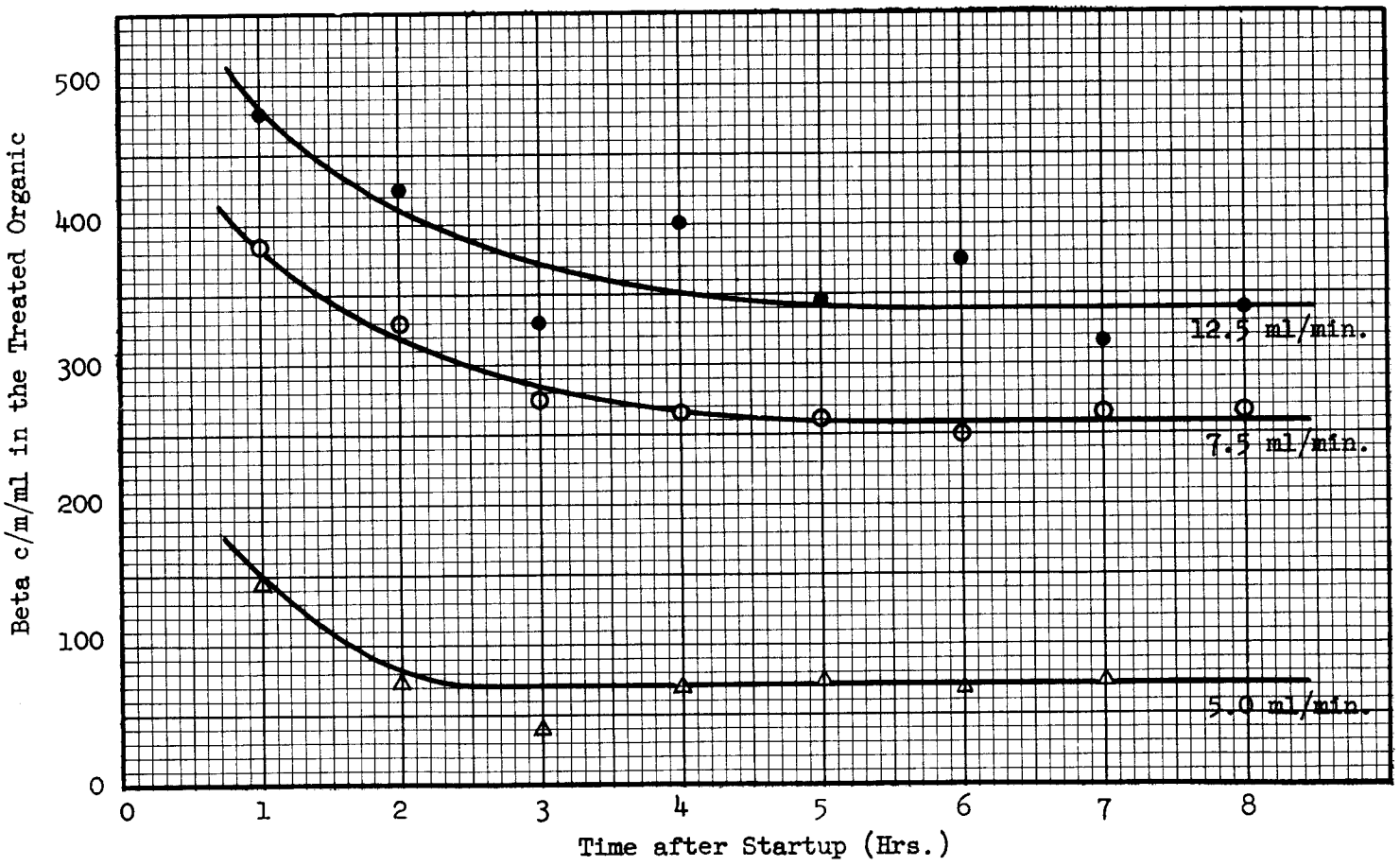
Column:
CO_3 -Section-9'
Wash-Section-4'
½" I.D. Pyrex

Amplitude:
1.20" in Column

Temperature:
Top: 56°C
Bottom: 45°C

Flow Rates:

Feed-5.0	12.5
CO_3 -1.0	2.5
Wash-1.0	2.5



Dwg. #13918

Figure 12

The Effect of Column Heating upon Beta Removal in the Continuous Solvent Recovery using the ½" Pulse Column

Feed: CW
U - 0.036 mg/ml
HNO_3 - 0.010 N
Pu - 368 c/m/ml
β - 9.9 x 10^3 c/m/ml
Ru - 5.8 x 10^3 c/m/ml
ORNL Level Feed Runs

Co_3 - 0.10M Na_2CO_3
Wash- 0.10M HNO_3

Flows:
12.5 ml/min. Feed
2.5 ml/min. CO_3
2.5 ml/min. Wash

Column - Compound:
Co_3 - Section - 6' ½" I.D.
Wash- Section - 3' ½" I.D.

Amplitude:
0.60" in Column

Plates:
1" Apart
32-Holes
0.039" in Diameter

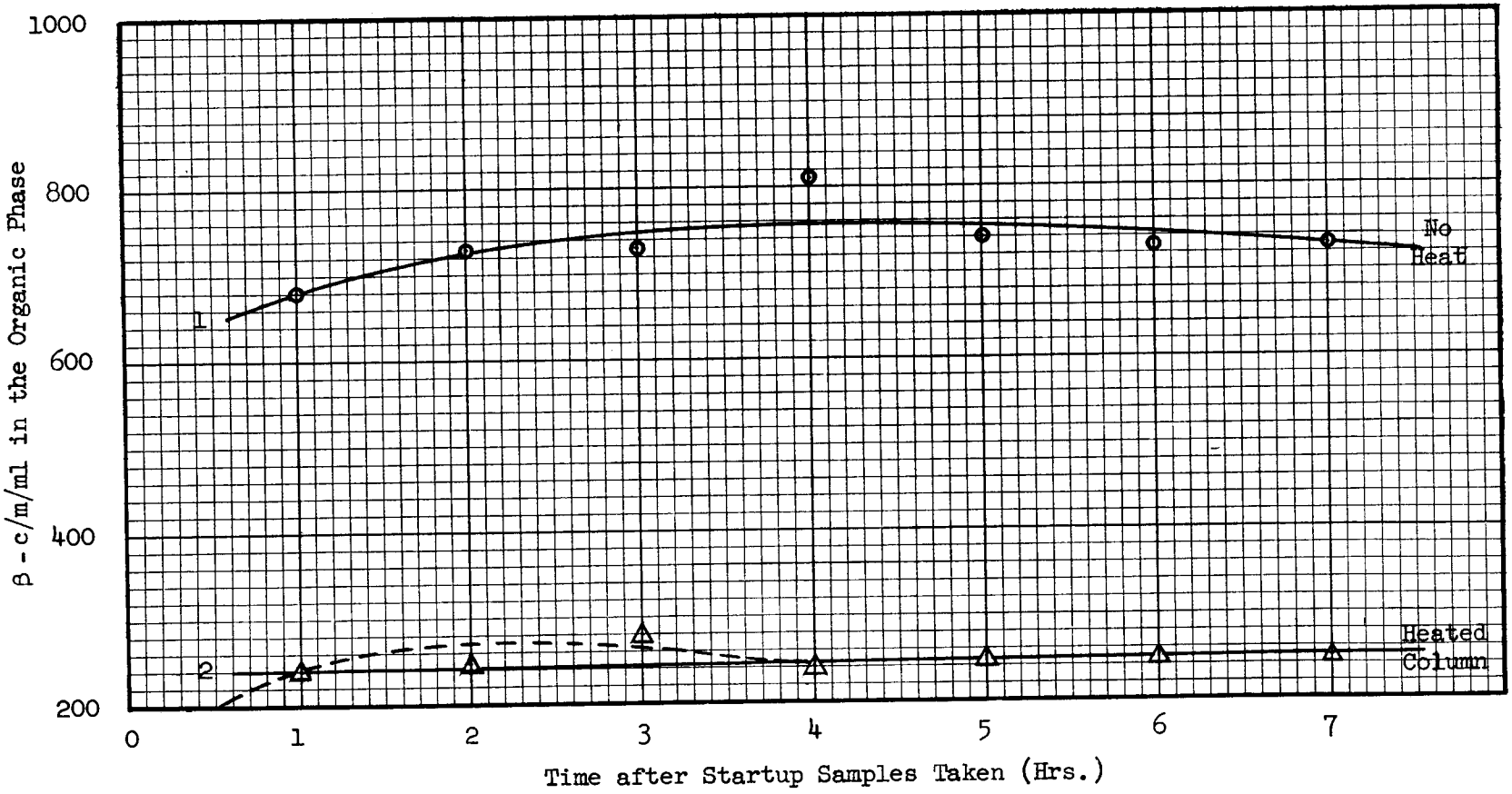


Figure 13

To Investigate the Effect of Heat and Periodic Acid

V.S. Beta Activity Removal from Used Organic in $\frac{1}{2}$" Pulse Column

Feed:
U - 0.012 mg/ml
HNO_3 - 0.004 N
Pu - 44 c/m/ml
β - 2.9×10^4 c/m/ml
Ru - 1.1×10^4 c/m/ml

Column - Compound:
CO_3 -Section-6'-$\frac{1}{2}$" I.D.
Wash -Section-3'-$\frac{1}{2}$" I.D.
Amplitude: 0.80" in Column
CO_3: 0.10 M Na_2CO_3 + HIO_4 when shown in graph.

Flow Rates:
Feed - 7.5 ml/min.
CO_3 - 1.5 ml/min.
Wash - 1.5 ml/min.
Wash:
Dist. H_2O

Plates:
1" Apart
32 Holes
0.039 " in Diameter

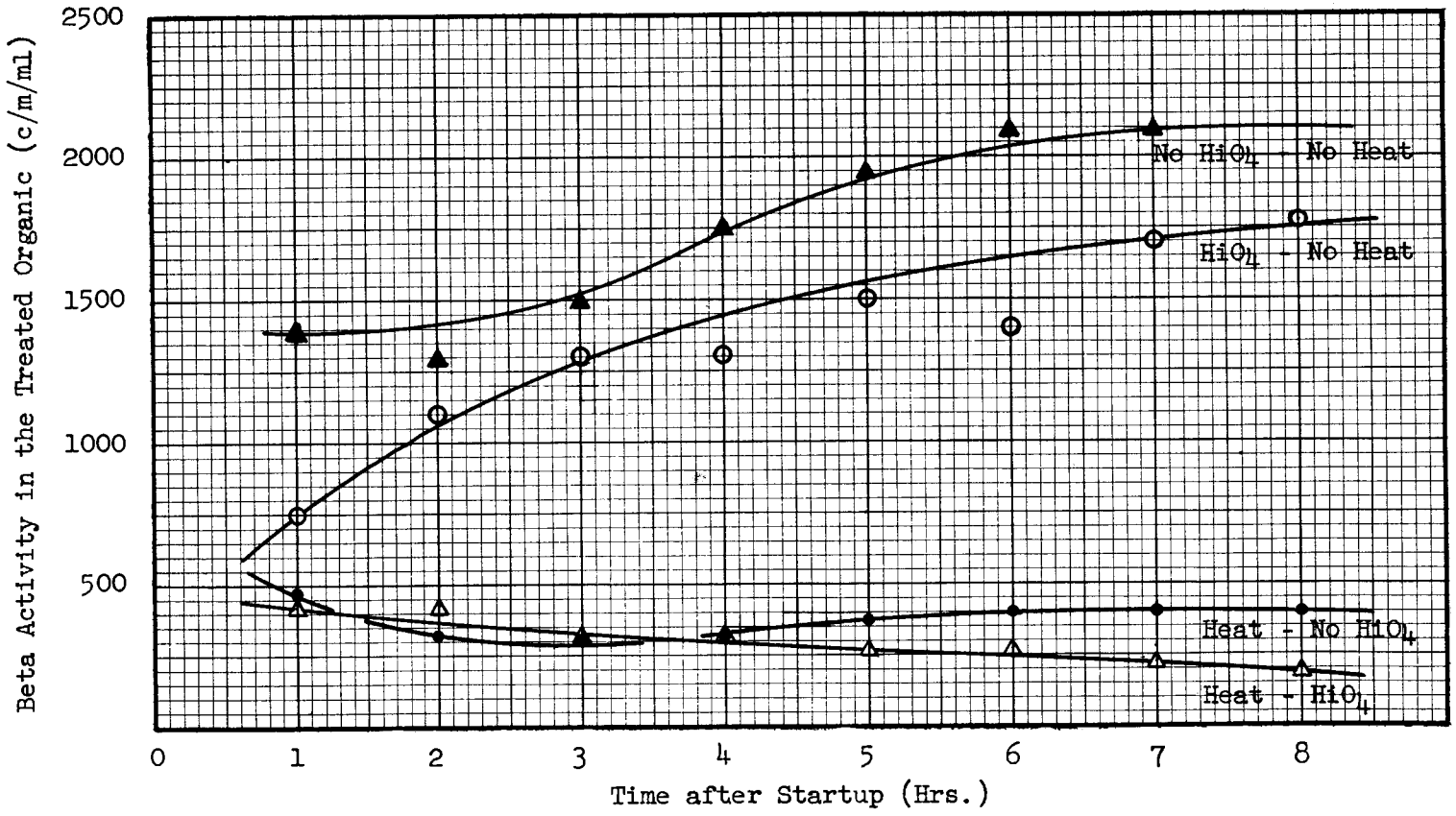


Figure 14

The Effect of Recycling Sodium Carbonate Five Times without Addition or withdrawal, upon the Beta and Uranium "Buildup" in the Carbonate Solution

Feed:
U - 0.320 mg/ml
H - 0.010 N
Pu - 183 c/m/ml
β - 2.34×10^4 c/m/ml

Carbonate Sol.:
0.10 M Na_2CO_3

Wash:
Distilled Water

Columns:

	HT.	- Cont. Phase	- Amp. in Column
Carbonate Column:	9' Long	- Organic	- 1.20 Inches
Wash Column:	4' Long	- Aqueous	- 0.43 Inches

Plates:
22 gauge S.S. 32 Holes 0.039" Diameter
1" Apart

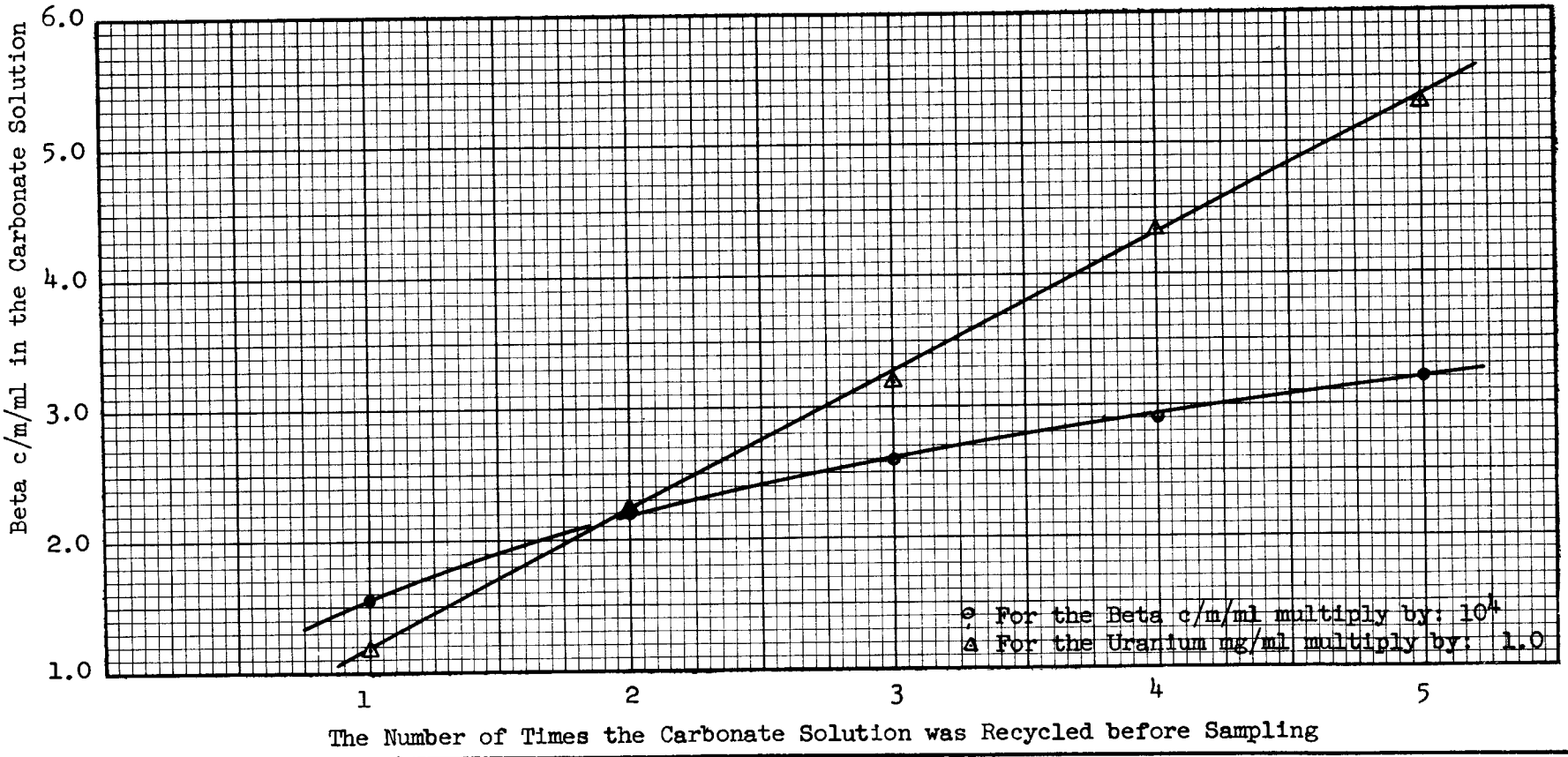


Figure 15

The Effect of Recycling the Carbonate Solution

upon the Beta Activity Removal - Recycled Five Times

Feed - CW
U - 0.320 mg/ml
H - 0.010 N
β - 2.34 x 10^4 c/m/ml
Pu- 183 c/m/ml
Ru- 1.0 x 10^4 c/m/ml
Origin -(CW)
ORNL

Column - CO_3
9' Long ½" I.D.
Column - Wash
4' Long ½" I.D.
Column - Temp.
Top - 55°C
Bottom - 45°C

Flow Rates
Feed - 7.5 ml/min.
CO_3 - 1.5 ml/min.
Wash - 1.5 ml/min.
Pulse Amp. **RPM**
CO_3 - Col - 1.20" - 38 RPM
Wash - Col - 0.40" - 60 RPM

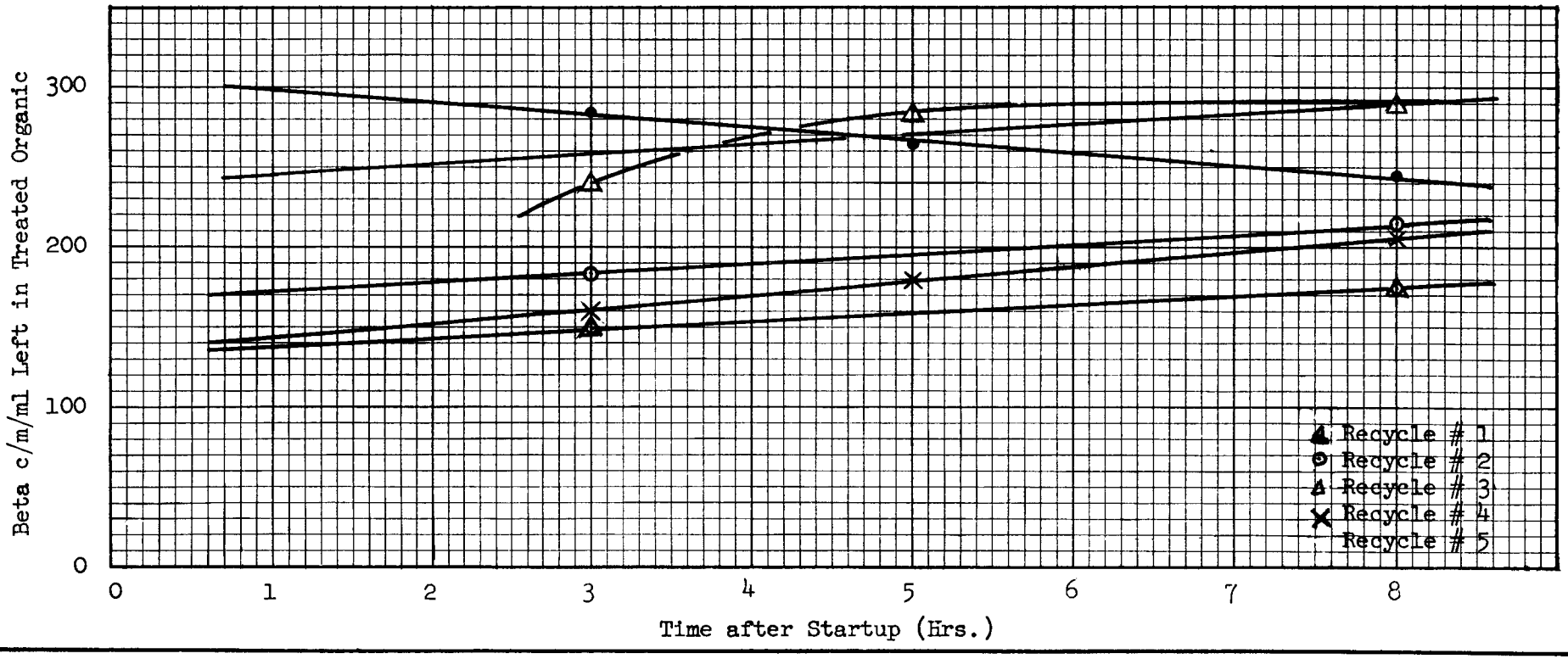


Figure 16

Continuous Recycling of the Aqueous Sodium Carbonate with a 1/5 Volume Addition of New Solution and a 1/5 Volume Draw Off V.S. Fission Activity Removal

Feed - CW

U - 0.092 mg/ml
HNO_3 - 0.003 N
Pu - 90 c/m/ml
β - 2.05×10^4 c/m/ml
Ru - 8.3×10^3 c/m/ml

Column - 2 Columns

9' Carbonate-Section
4' Wash -Section

Pulse Amplitude

1.20" in CO_3 Column
0.43" in Wash Column

Flow Rates

Feed - 7.5 ml/min.
CO_3 - 1.5 ml/min.
Wash - 1.5 ml/min.

Temperature

CO_3 Column - 45°C Bottom-45°C
Wash Column - Room Temperature

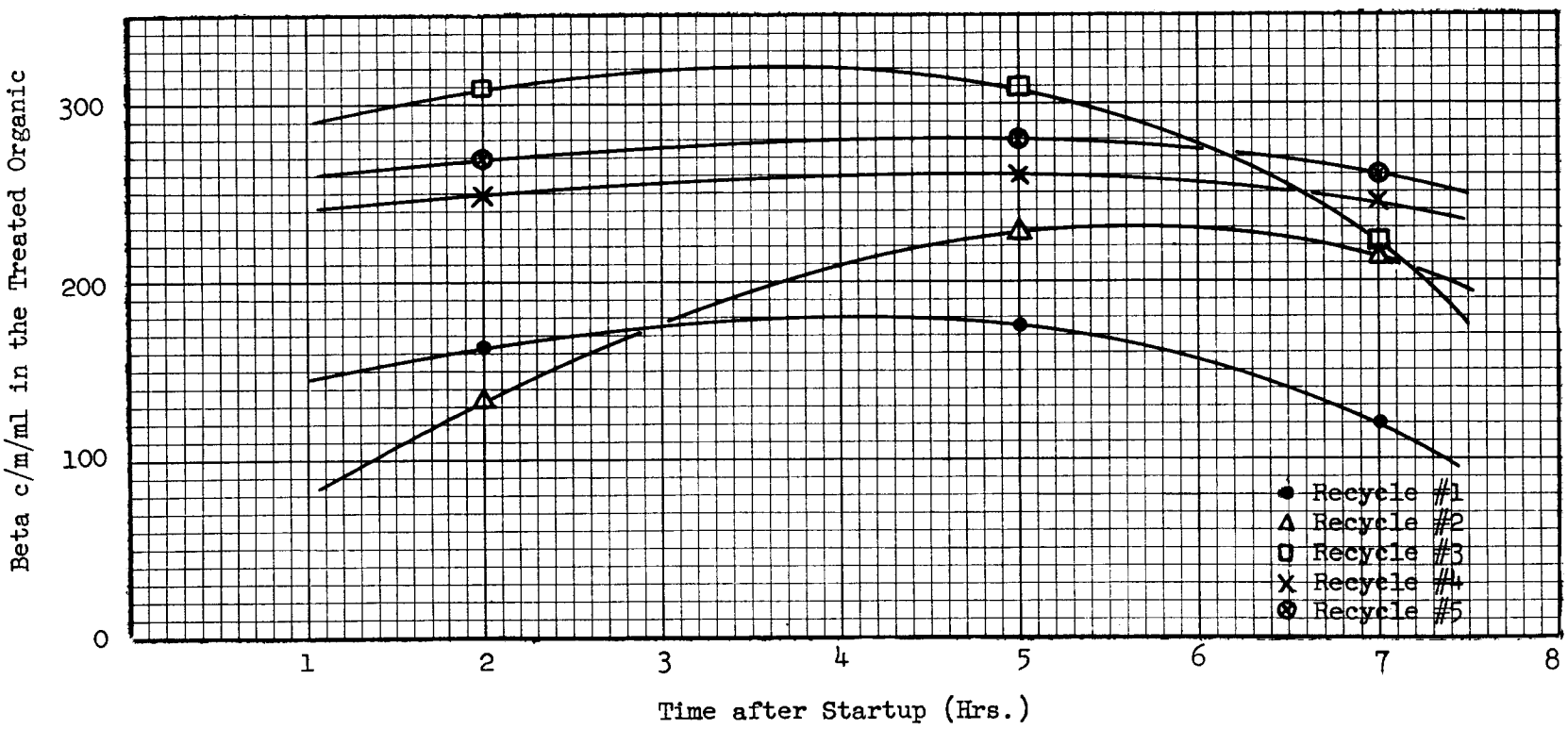
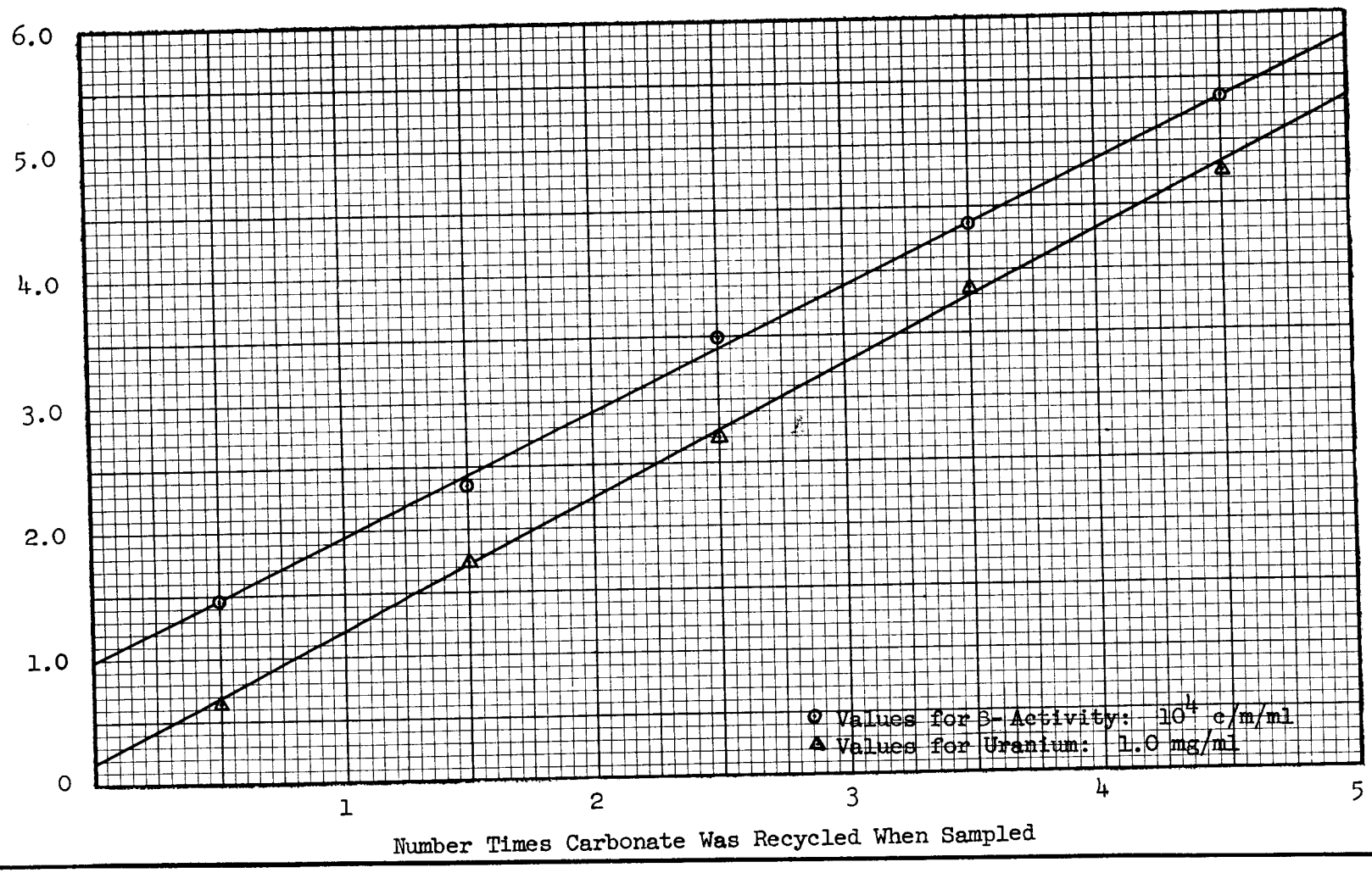


Figure 17

The Uranium and Fission Activity Build-up in the Recycled Sodium Carbonate Solution



[redacted]

8.3 Tables 1-6 Experimental Investigations

TABLE 1

ORGANIC VS AQUEOUS AS THE CONTINUOUS PHASE IN THE REMOVAL OF FISSION ACTIVITY FROM THE USED ORGANIC IN THE CONTINUOUS SOLVENT RECOVERY PROCESS

Feed - Used Organic from ORNL Level Runs

U	- 0.036 mg/ml	Ru	- 5.8×10^3 c/m/ml
HNO_3	- 0.010 N	Zr	- 640 c/m/ml
Pu	- 368 c/m/ml	TRE	- 18 c/m/ml
Gross	- 8.7×10^3 c/m/ml	UX-1	270 c/m/ml

Plates

1" apart
32 holes
0.039" Diam.

Column-Compound

Carbonate-Section - 72"
Wash - Section - 36"

Flow Rates

Feed - 12.5 m/min.
CO_3 - 2.5 m/min.
Wash - 2.5 m/min.

Run	Pulse Generator for Col.			Molarity of Sol.		The Cont. Phase	Treated Organic Analysis (All Fission Activity in c/m/ml)								Raffinate Analysis Activity in c/m/ml				Column Temp. °C.		Time in the Column
No.	Type	Amp.	RPM	CO_3	Wash	In Col.	U-mg/ml	H - N	Gross	Ru	Zr	TRE	UX-1	Pu	U mg/ml	Basic (N)	Gross	Pu	Top	Bot.	Min.
1	Milton Roy	0.60"	38	0.10	0.00	Aqueous	0.0100	0.010	1.1×10^3	348	48	LZ	38	12	0.086	0.08	1.5×10^4	280	22°	22°	4
2	"	0.60"	38	0.10	0.10	"	0.0100	0.011	1.0×10^3	778	29	L2	21	14	0.080	0.08	1.2×10^4	255	22°	22°	4
3	"	0.60"	38	0.10	0.10	"	0.0060	0.002	995	494	62	L4	42	26	0.320	0.15	2.4×10^4	610	22°	22°	4
4	"	0.60"	38	0.10	0.10	Organic	0.0020	0.008	405	360	27	L10	10	8	0.125	0.15	1.8×10^4	282	22°	22°	18
5	"	0.60"	38	0.10	0.10	"	0.0040	0.003	176	160	--	--	--	22	0.055	0.04	1.5×10^4	109	22°	45°	18
6	"	0.60"	38	0.10	0.00	"	0.0010	0.005	136	166	--	--	--	28	0.058	0.08	1.0×10^4	125	22°	45°	18
7	"	0.80"	38	0.50	0.00	"	0.0010	0.002	128	158	--	--	--	0	0.046	0.27	1.4×10^4	128	22°	45°	18

Fission Activity Reported in c/m/ml at 10% Geometry
Pu Results Reported in c/m/ml at 52% Geometry.

[redacted]

[redacted]

TABLE 2

THE EFFECT OF FLOW RATES, AMPLITUDE AND CARBONATE CONCENTRATION UPON THE REMOVAL OF FISSION ACTIVITY FROM THE USED ORGANIC FROM A HANFORD LEVEL RUN

Feed - Used Organic Hanford Level Runs

U - 0.0120 mg/ml
H - 0.004 N
Pu - 44 c/m/ml
Gross β - 2.91×10^4 c/m/ml
Ru - 2.12×10^4 c/m/ml
Zr - 1.26×10^4 c/m/ml
Wash - Dist. H_2O

Plates - SS

1" apart
32 holes
0.039" in Diam.

Column - Compound

CO_3 - Section - 72"
Wash- Section - 36"
Top - 22°C
Bottom - 45°C

Flow Rate Ratio

Feed - 5 ml/min.
Carbonate - 1 ml/min.
Water Wash - 1 ml/min.

Run No.	Molar of CO_3	Pulser			Cont. Phase	Treated Organic Analysis						Raffinate Analysis				Col. Flows ml/min			Time in the Column
		Type	Amp.	RPM		U mg/ml	H^+(N)	Gross β	Ru	Zr	Pu	U mg/ml	Basic (N)	Gross β	Pu	Feed	CO_3	Wash	
8	0.10	Milton Roy	0.80	38	Organic	0.0005	0.002	316	278	21	0	0.018	0.079	2.70×10^4	71	5.0	1.0	1.0	45 min.
9	0.20	"	0.80	38	"	0.002	0.00	740	803	77	5	0.028	0.200	3.7×10^4	92	5.0	1.0	1.0	45
10	0.20	"	0.80	38	"	0.002	0.00	1.8×10^3	1.6×10^3	164	0	0.037	0.200	4.4×10^4	112	12.5	2.5	2.5	18
11	0.20	"	0.40	38	"	0.002	0.010	2.9×10^3	3.1×10^3	34	6	0.026	0.170	4.5×10^4	123	12.5	2.5	2.5	18
12	0.10	Bellows	0.14	60	"	0.0002	0.004	861	928	25	0	0.028	0.106	3.15×10^4	167	5.0	1.0	1.0	45
13	0.10	"	0.32	60	Aqueous	0.0004	0.003	879	1.0×10^3	67	0	0.031	0.080	3.2×10^4	96	7.5	1.5	1.5	27
15	0.10	Milton Roy	1.20	38	Organic	0.0001	0.005	192	238	18	0	0.194	0.061	2.9×10^4	360	7.5	1.5	1.5	27

[redacted]

[redacted]

TABLE 3

INVESTIGATING THE TYPE OF WATER-WASH COLUMN BEST FOR
REMOVING THE ENTRAINED CARBONATE SOLUTION AND ACTIVITY FROM THE TREATED ORGANIC

Feed - Pilot Plant Organic - ORNL Level

U - 0.039 m_e/ml
H - 0.003 N
Pu - 50 c/m/ml
Gross β - 6.3×10^3 c/m/ml
Ru - 2.1×10^4 c/m/ml
Zr - 1.2×10^3 c/m/ml
CO_3 - molar
0.10 M

All F.P. results reported in c/m/ml at 10% Geometry.
All Pu results reported in c/m/ml at 52% Geometry.

Columns

CO_3 Contacting - 108"
Water Wash - 48"
Plates - S.S.
1" apart

Flow Rates

Feed (cw) - 7.5 ml/min.
Carbonate - 1.5 ml/min.
Water Wash - 1.5 ml/min.

Run No.	Type of Column	Pulser			Continuous Phase	Treated Organic Content					Raffinate Content				Col. Temp. °C		Time in Column
		Type	Amp.	RPM		U mg/ml	H^+ (N)	Gross β	Ru	Pu	U mg/ml	Gross β	Basic N	Pu	Top	Bot.	
1	CO_3 - Col.	Milton Roy	1.20"	38	Organic	0.0004	0.002	650	328	31	0.118	1.67×10^4	0.130	151	56°	45°	36 min.
	Wash- Col.	Static Water Column.			Aqueous	0.0004	0.002	205	159	0	0.003	235	0.001	12	22°	22°	26 Sec.
2	CO_3 - Col.	Milton Roy	1.2	38	Organic	0.0012	0.008	155	142	0	0.148	1.66×10^4	0.176	135	56°	45°	36 Min.
	Wash- Col.	Bel.	0.36	60	Organic	0.0006	0.003	0	---	--	0.0029	164	0.002	90	22°	22°	26 Sec.
3	CO_3 - Col.	Milton Roy	1.32	38	Organic	0.0009	0.002	236	184	1	0.138	2.15×10^4	0.189	195	55°	45°	36 Min.
	Wash- Col.	Bel.	0.36	60	Aqueous	0.0002	0.001	---	---	0	0.0016	215	0.0016	92	22°	22°	26 Sec.

[redacted]

[redacted]

TABLE 4

RECYCLING THE SODIUM CARBONATE SOLUTION IN THE COMPOUND COLUMN WITH THE WATER WASH DILUTING THE CARBONATE SOLUTION WITH EACH RECYCLE (1:1)

Feed - Used Organic Hanford Level Run

U - 0.045 mg/ml
HNO_3 - 0.011 N
Pu - 174 c/m/ml
Gross β - 2.5×10^4 c/m/ml
Ru - 2.1×10^4 c/m/ml
Column - Compound
Carbonate - 72"
Water Wash - 36"

Pulser

Milton Roy
38 - RPM
1.20" Amp.

Flow Rates

Feed - 7.5 ml/min.
CO_3 - 1.5 ml/min.
Water - 1.5 ml/min.

Plates

1" apart
36 holes
0.039" in Diameter.

F.P. Activity given in c/m/ml at 10% Geometry.
Pu Results computed in c/m/ml at 52% Geometry.

Run No.	Condition of the Carbonate Sol.	CO_3 Conc. (M)	Cont. Phase in Col.	Organic Content (Treated)					Raffinate Content				Cond. of Treated Organic	Column Temp. °C.		Time in Column Min.
				H^+ (N)	U mg/ml	Gross β	Ru	Pu	U mg/ml	Gross β	Basic (N)	Pu		Top	Bot.	
1	First Cycle	0.10	Aqueous	0.003	0.00016	556	587	0	0.044	2.5×10^4	0.100	440	Turbid	55°	45°	27 min.
2	Second Recycle	0.05	Aqueous	0.007	0.00014	742	828	0	0.048	3.7×10^4	0.059	411	Turbid	55°	45°	27 min.
3	Third Recycle	0.025	Aqueous	0.010	0.00028	609	575	0	0.065	3.4×10^4	0.045	337	Turbid	55°	45°	27 min.

[redacted]

[redacted]

TABLE 5

THE EFFECT OF RECYCLING THE SODIUM CARBONATE CONTINUOUSLY WITH A 1/5 VOLUME ADDITION OF NEW AND A 1/5 VOLUME "DRAW OFF" OF USED ORGANIC UPON THE REMOVAL OF FISSION PRODUCTS FROM USED ORGANIC

Feed - CW Pilot used Organic From Hanford Level Runs

U - 0.092 mg/ml
H - 0.003 N
Pu - 90 c/m/ml

Gross β - 2.05×10^4 c/m/ml
Ru - 2.1×10^4 c/m/ml
Zr - 1.2×10^3 c/m/ml

Fission Activity reported in c/m/ml at 10% Geometry.
Pu in c/m/ml at 52% Geometry.

2 - Column System

Carbonate - Section = 9'
Wash - Section = 4'

Rates — Flows

Feed - 5.0 - 7.5 m/min.
Carbonate - 1.0 - 1.5 m/min.
Wash - 1.0 - 1.5 m/min.

Col. Temp. °C.

Top. Col. - 55°C
Bottom Col. - 45°C

Run No.	Type of Col.	Pulser			Cont. Phase	Organic Content				Raffinate Content				Cond. of Organic	Column Temp. °C		Time in the Column
		Type	Amp.	RPM		U mg/ml	H^+ (N)	Gross β	Pu	U mg/ml	Basic	Gross β	Pu		Top	Bot.	
1	CO_3	Milton Roy	1.20	38	Organic	.00012	0.001	1.6×10^3	0	.16	0.010	2.0×10^4	348	Turbid	55°C	45°C	36 min.
	Wash	Bel.	0.43	60	Water	.0005	0.001	152	0	.00006	0.001	230	16	Clear	--	--	4 min.
2	CO_3	Milton Roy	1.20	38	Organic	.00006	0.004	1.4×10^3	6	0.53	0.120	3.9×10^4	482	Clear	55°C	45°C	36 min.
	Wash	Bel.	0.43	60	Water	.00003	0.005	192	0	0.0010	0.002	1.3×10^3	25	Clear	--	--	4 min.
3	CO_3	Milton Roy	1.20	38	Organic	.00006	0.001	1.3×10^3	0	0.730	0.109	4.0×10^4	1.1×10^3	Clear	55°C	45°C	36 min.
	Wash	Bel.	0.43	60	Water	.00002	0.002	279	0	0.009	0.001	520	60	Clear	--	--	4 min.
4	CO_3	Milton Roy	1.20	38	Organic	.0004	0.01	1.3×10^3	0	1.32	0.109	4.5×10^4	1.6×10^3	Clear	55°C	45°C	36 min.
	Wash	Bel.	0.43	60	Water	.00002	0.02	251	0	0.04	0.04	840	42	Clear	--	--	4 min.
5	CO_3	Milton Roy	1.20	38	Organic	.00020	0.006	1.3×10^3	0	1.44	0.08	5.8×10^4	1.7×10^3	Turbid	55°C	45°C	36 min.
	Wash	Bel.	0.43	60	Water	.00004	0.008	266	0	0.04	0.01	860	70	Clear	--	--	4 min.

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TABLE 6

THE EFFECT OF RECYCLING THE SODIUM CARBONATE SOLUTION FIVE TIMES, WITHOUT DILUTION, UPON THE REMOVAL OF FISSION ACTIVITY FROM USED ORGANIC

Feed: (CW) Used Organic ORNL Level

U - 0.320 mg/ml
H - 0.010 N
Pu - 183 c/m/ml

Gross β - 1.4×10^4 c/m/ml
Ru - 5.8×10^3 c/m/ml
Zr - 1.2×10^3 c/m/ml

2 - Column System

CO_3 - Column - 108"
Wash - Column - 48"

Flow Rates

Feed (CW) - 7.5 ml/min.
Carbonate - 1.5 ml/min.
Wash - 1.5 ml/min.
CO_3 Conc. 0.10 M

Plates

1" apart
36 holes
0.039" in Diameter

Column Temp. °C

Top - 55°C
Bottom - 45°C

Run No.	Type of Column	Pulser Type	Pulser (in) Amp.	Pulser RPM	Cont. Phase in Col.	Treated Organic U mg/ml	Treated Organic H^+ (N)	Treated Organic c/m/ml Gross β	Treated Organic c/m/ml Pu	Raffinate Content U mg/ml	Raffinate Content (N) Basic	Raffinate Content c/m/ml Gross β	Raffinate Content c/m/ml Pu	Cond. of Treated Organic	Time in Col. (Min).
1	CO_3	Milton Roy	1.20"	38	Organic	0.0005	0.000	500	1	1.250	0.108	1.5×10^4	600	Turbid	36
	Wash	Bellows	0.43"	60	Aqueous	0.0001	0.000	225	0	0.0003	0.002	235	12	Clear	4
2	CO_3	Milton Roy	1.20"	38	Organic	0.0005	0.005	355	4	2.250	0.107	1.6×10^4	900	Clear	36
	Wash	Bellows	0.43"	60	Aqueous	0.0001	0.005	215	0	0.0005	0.003	510	35	Clear	4
3	CO_3	Milton Roy	1.20"	38	Organic	0.0002	0.007	635	1	3.000	0.130	1.89×10^4	970	Turbid	36
	Wash	Bellows	0.43"	60	Aqueous	0.00001	0.008	285	0	0.013	0.023	2.3×10^3	30	Clear	4
4	CO_3	Milton Roy	1.20"	38	Organic	0.0005	0.005	350	0	4.360	0.148	2.6×10^4	2500	Clear	36
	Wash	Bellows	0.43"	60	Aqueous	0.0002	0.007	180	0	0.095	0.003	2.0×10^3	30	Clear	4
5	CO_3	Milton Roy	1.20"	38	Organic	0.0001	0.001	675	0	5.280	0.112	3.22×10^4	2800	Clear	36
	Wash	Bellows	0.43"	60	Aqueous	0.00004	0.002	204	0	0.00030	0.010	5.7×10^3	37	Clear	4

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