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DECONTAMINATION OF ORNL PUREX PILOT PLANT

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CHEMICAL TECHNOLOGY DIVISION

DECONTAMINATION OF ORNL PUREX PILOT PLANT

H. P. Wood

DATE ISSUED

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Abstract

This report describes the decontamination of Oak Ridge National Laboratory Purex Pilot Plant facilities and presents an appraisal of the decontaminating techniques and reagents which were employed.

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1.0 Introduction

Chemical decontamination of the Purex Pilot Plant facilities was undertaken following the completion of a series of runs in which slugs irradiated from 40 to 450 MWD/T were processed at the rate of 75 kg. of uranium per day. Decontamination was necessary to provide maintenance personnel with adequate working time to make extensive equipment changes, and to give operating personnel adequate access to cells and unshielded areas of roof, gallery and pipe tunnel. In addition, information was sought concerning the effectiveness of decontamination reagents and techniques. These were evaluated on the basis of radioactivity removed from each item of equipment, quantities of chemicals used and manpower required.

2.0 Summary

The Purex Pilot Plant (Figure I) was shut down for clean-up and modification of facilities after 150 days of operation. The decontamination, which preceded conversion work in each area, required approximately 6000 man-hours of operator labor and the expenditure of $5400 for reagents.

Wherever possible, equipment was decontaminated in place. This policy was followed in the case of tanks and columns. However, flanged connections, valves, jets, and pumps proved difficult to decontaminate, and frequently were disassembled for treatment.

During the decontamination program, Pilot Plant activity levels were reduced to the values noted in Table I.

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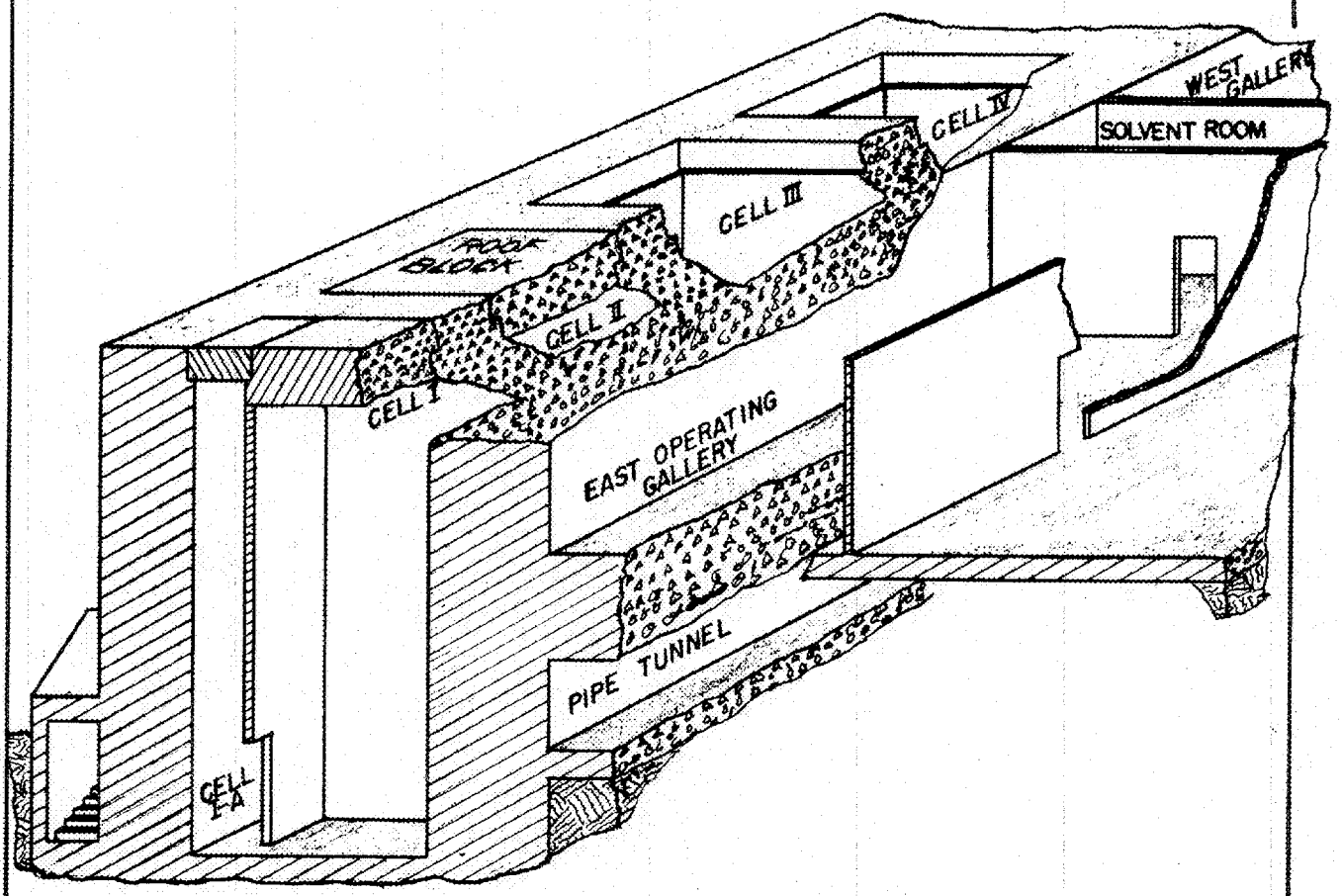

Fig. I

GENERAL PILOT PLANT ARRANGEMENT

HPW 1-15-52
RBW 1-15-52

TABLE I

Summary Decontamination Results

Area	Area Activity Level* (Measured with "Cutie Pie")	
	Before Decontamination	After Decont.
Cell I	1000 R/hr. (estimated)	50 mr./hr.
Cell I-A	100 R/hr. (estimated)	35 mr./hr.
Cell II	1000 mr./hr.	35 mr./hr.
Cell III	350 mr./hr.	30 mr./hr.
Cell IV	5000 mr./hr.	20 mr./hr.
Pipe Tunnel	100 mr./hr.	5 mr./hr.
Roof (east)	200 mr./hr.	5 mr./hr.
East Gallery	35 mr./hr.	5 mr./hr.
Solvent Room	100 mr./hr.	5 mr./hr.

* Cell Readings were taken 4 feet above the floor and 6 feet inside the cell doorway. The other area activities represent the maximum levels encountered 4 feet above floor level by personnel moving through the particular area.

During the course of decontamination work, eight chemicals were evaluated for relative effectiveness. Consumption was: 64,000 lbs. of 68 per cent nitric acid; 2165 lbs. of citric acid; approximately 500 lbs. each of hydrochloric acid, hydrofluoric acid, oxalic acid, caustic and versene; 20 lbs. of periodic acid.

A mixture of 3 per cent by weight hydrofluoric acid and 20 per cent by weight nitric acid (3-20 reagent) was found to be the best decontaminant for stainless steel (types 347 and 309 CB). It was approximately twice as effective as 10 per cent nitric, citric or oxalic acid, and 10 times superior to versene, caustic, or periodic acid. This reagent further proved valuable in attacking the last 5-10 per cent of activity on contaminated equipment where the other chemicals were ineffective.

Although hydrochloric acid exhibited a decontaminating ability equal to hydrofluoric acid, it was used in limited quantity because of intergranular corrosion of stainless steel by the chloride ion. This objectionable characteristic was partially controlled by the use of an organic corrosion inhibitor. Not enough was known about the reliability of the inhibitor, however, to justify more general use of hydrochloric acid.

The corrision rate of 3-20 reagent toward stainless steel is less than 0.06 mils/hr. at 25° C; however, it did attack weld flaws and pump parts (not 18-8 stainless steel or equivalent) which resisted other decontaminants. Column treatment techniques accordingly were modified to avoid reagent pumping and to minimize the exposure of pipe lines to the solution.

Cell I tanks were decontaminated with one-half of the volume of solution required for the tanks in other cells. This was made possible by a spray system built into the Cell I tanks which provided an efficient means for bringing decontaminating solutions into contact with internal surfaces.

Stainless steel liners which had been installed over concrete cell floors and portions of cell walls greatly facilitated decontamination work. The liners prevented contact with and penetration into the concrete by radioactive solutions.

The future use of 3-20 reagent wherever possible and application of the best decontaminating techniques within the limits of the present equipment should permit a reduction in time required for decontamination of this pilot plant equipment from 75 to 38 days with commensurate labor savings and a 20 percent savings of chemical cost.

Electrodecontamination techniques were appraised and found to be impractical for use on the pilot plant tanks. Construction did not permit convenient cathode insertion, and fixed dip pipes interfered with proper current distribution.

Samples of discharged solutions taken for determination of individual fission product activities showed that the A column retained on internal surfaces 0.002 per cent of the total beta activity passed through it during process operation. This activity contained niobium-ruthenium beta in a 60-1 ratio compared to an average niobium-ruthenium ratio in process solutions of 1-10, indicating selective deposition of niobium during column operation.

3.0 Decontamination Procedures

The decontamination work proceeded from Cell IV to Cell I, in that order, with clean-up of other areas accomplished as manpower availability permitted. Work in several cells was carried on simultaneously, the final clean-up in each being coordinated with the conversion schedule. Particular care was observed when each cell was released to conversion crews that all lines and tanks were free of corrosive chemicals and radioactive solutions and that associated pumps, steam-jets, etc. were taken out of service.

Cell activity levels (Figures II-V) and permissible working times* at the time decontamination was started were:

Cell I	1000 R/hr.	no access
Cell I-A	100 R/hr.	no access
Cell II	1000 mr./hr.	4 minutes in areas not adjacent to hot columns

* (Calculated cell working times were based on the permissible daily exposure of 60 mr.)

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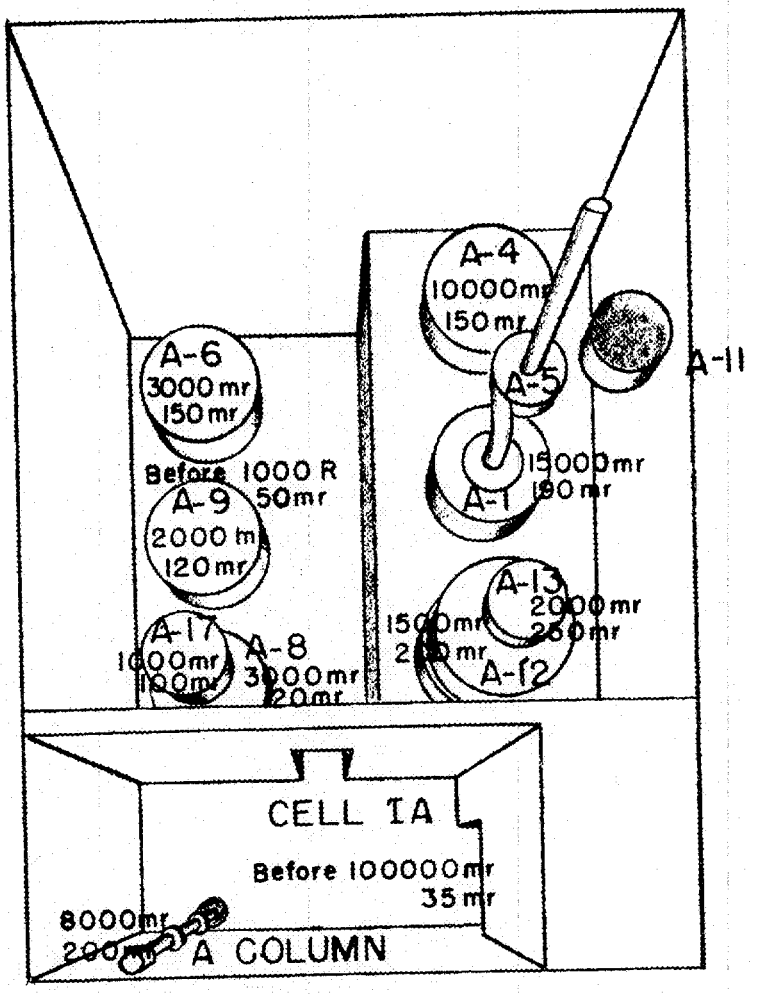
CELL I



NOTE: Tank & column readings taken after cell access gained.
General cell background is an "in use" estimate.

FIG. II

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CELL II

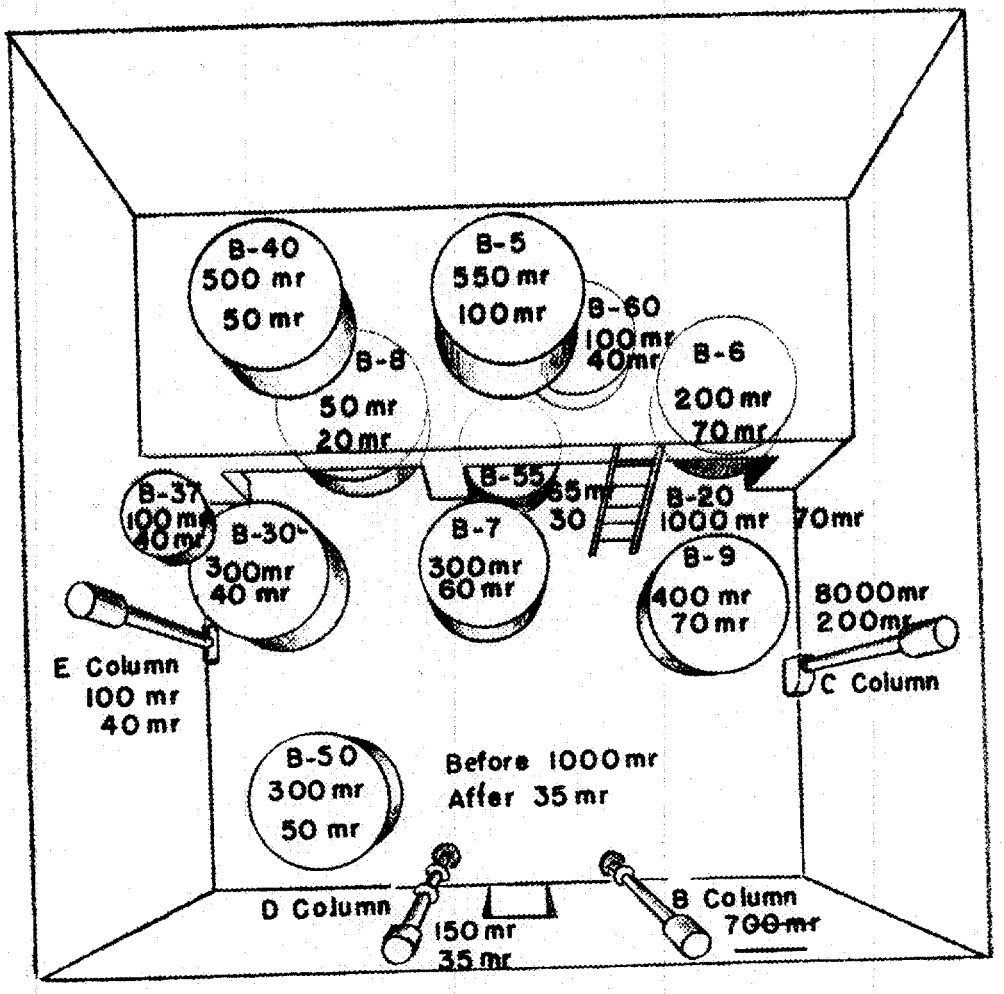

FIG. III

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CELL III

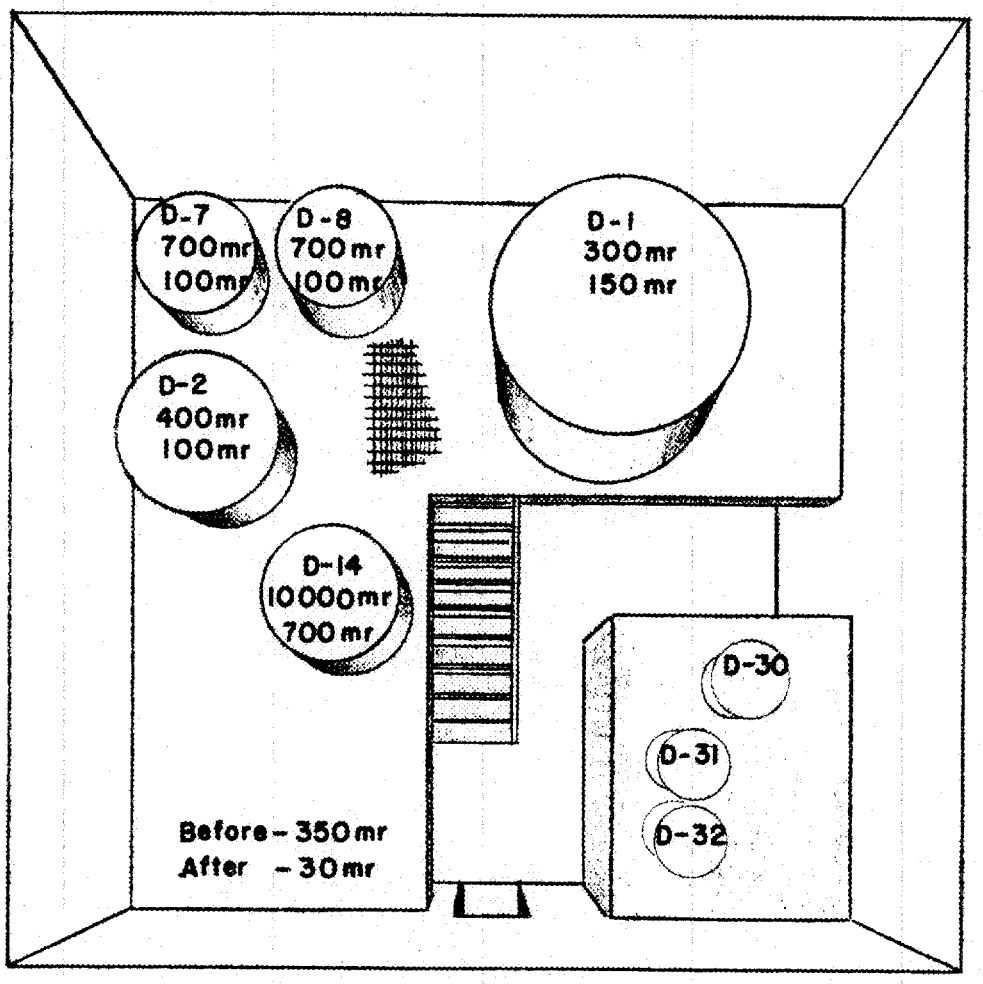

FIG. IV

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CELL IV

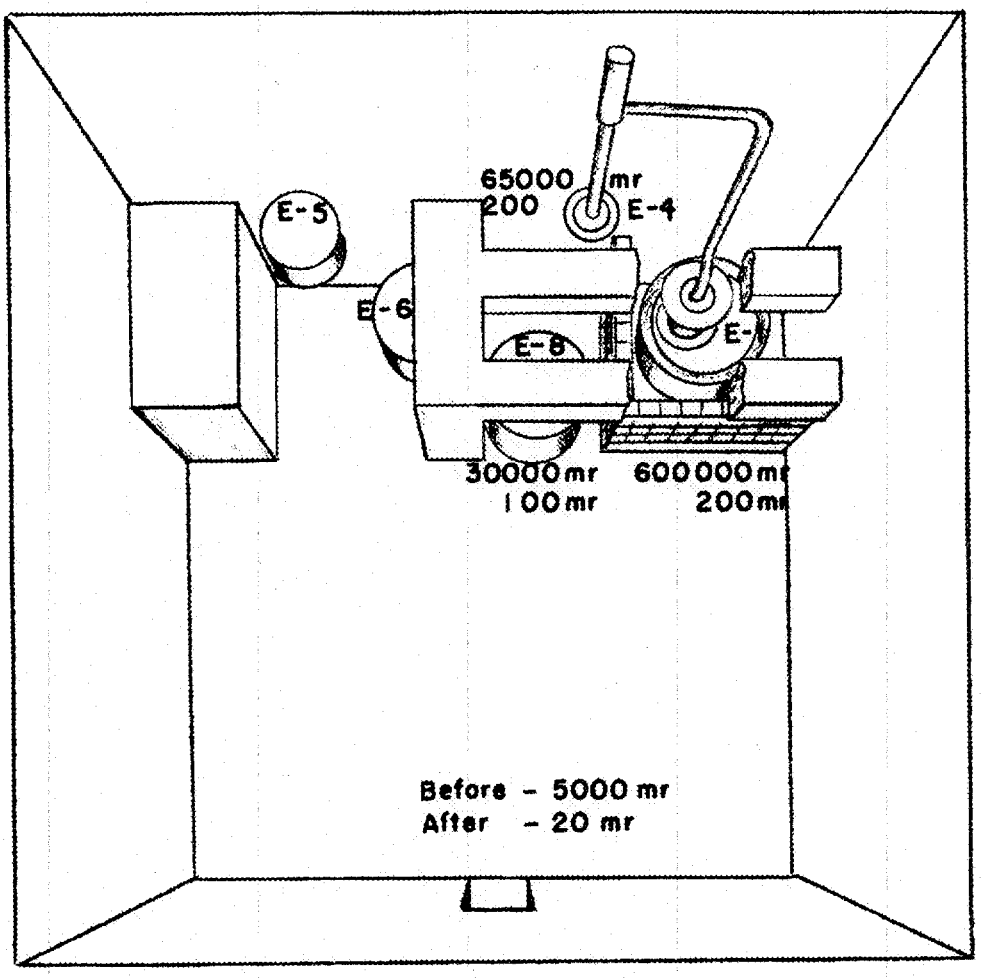

FIG. V

Cell III	350 mr./hr.	10 minutes general
Cell IV	5000 mr./hr.	45 seconds at the door decreasing as tanks at north wall were approached. (Radioactive solution was accidentially discharged nto cell equipment during evaporator clean-up operation; this reading was taken after this occurence.)

The intitial clean-up of Cells I, IA, and IV necessarily proceeded without regard to the exact sources of radiation since these cells could not be entered for survey. Solutions were introduced into cell tanks, columns, lines, and jets from the relatively uncontaminated roof and gallery areas until radiation levels were reduced to a general background of 500 mr./hr. or less. Surveys were then made to determine the specific area of each equipment item which required further intensive treatment and how the decontaminating solution could best be brought into contact with that area. The clean-up of Cells II and III, which were accessable for limited periods from the inception of the clean up program, was guided throughout by detailed radiation surveys.

It became necessary during the course of cell decontamination work to depart occasionally from the policy of removing activity without removal or alteration of equipment. When small pieces of equipment such as jets and valves failed to respond to the normal chemical treatment, it became necessary to remove them from the cells by unbolting flanged connection or cutting connecting lines, or to shield them in place with lead. When removed, efforts were made to clean them for return to the cell if the particular items were intended to be parts of the converted facility. In some instances, hot lines and valves

were cut out and discarded because the cost of clean-up appeared unjustifiable. Spot shielding with lead was employed on two tanks where local areas of retained activity resisted chemical treatment. Removal of tanks from the cells for clean-up was not economically feasible except in one instance where a change of tank location also was involved.

As adjuncts to the primary program of rapid Pilot Plant clean-up, studies were made of the efficiency of various decontaminating reagents and techniques. The objectives were: (1) to find a decontaminant which combined effectiveness with low cost, low corrosion rate, and ready availability, (2) to develop standard techniques for future decontamination work. Decontamination results were followed by the analysis of discharged solutions for gross beta activity and by the use of frequent H.P. surveys of equipment.

The different types of equipment and their locations precluded the use of a single decontamination technique. For reference purposes, each equipment category is discussed separately.

3.1 Tank Decontamination

Cell I tanks had become contaminated by contact with hot feeds or waste products from the A column. Activity levels of the empty tanks when decontamination was started were estimated to range from 5 R./hr. (A-9 displacement fluid catch tank) to 500 R./hr. (A-1 dissolver).

Cell II tanks were employed for 1st and 2nd uranium cycle catch tanks (except the IAW catch tank) and 2nd uranium cycle feed adjustment. Radioactivity levels had reached a maximum of 1000 mr./hr.

Cell III tanks and associated Solvent Room tanks were contaminated by activity carried in the process solvent. Contamination in Cell III averaged

700 mr./hr. except for D-14 (spent carbonate separator) which had activity of 15 R./hr. at contact; Solvent Room tanks had activities up to 300 mr./hr. at contact.

In Cell IV, the Acid Recovery Unit tanks were contaminated by accidental discharge of hot decontaminating solution from the fission product evaporator (E-1) into the distillation column and other tanks in the cell. A survey made immediately after this occurrence showed activities to be 600 R/hr. in the evaporator, 65 R/hr. in E-4, the reboiler, and 30 R/hr. in E-8 catch tank. Prior to this discharge, Cell IV activity was confined to the evaporator.

Tank decontamination was accomplished by washing internal surfaces with chemical solutions to remove contaminating ions. Generally these solutions were introduced from the Operating Gallery through regular solution addition lines. Economies in the quantities of chemicals required were made possible by passing one wash through several tanks in series. There were limitations to the economies that could be obtained by this means; all tanks in any one cell were not inter-connected. In Cell II for instance, one group of four tanks could be washed in series, all others were connected only to the waste catch tank and had to be washed individually.

Heat was applied to the tanks containing decontaminating solutions by means of steam jackets or steam spargers. Where the solution contained more than 1 per cent hydrofluoric acid, temperatures were not raised above 60° C in order to avoid excessive corrosion.

The facilities provided for the decontamination of most Cell I tanks were particularly effective. A 300 gallon stainless steel tank (C-17) located on the roof and equipped with agitator, steam jacket and pump permitted the preparation of a quantity of decontaminating solution which could then be discharged through stainless steel lines into Cell I tanks. Valving permitted the solution to be directed to any tank except A-1 (dissolver) and A-11 (filter). At the end of the solution line inside each tank, a spray nozzle was attached and directed upward. By this means decontaminant could be sprayed over the inside of the tanks. Contact time between one small portion of solution and a particular area on top or side of the tank was short. Since, during the period of spraying, the surfaces were being continually exposed to fresh solution with presumably a maximum decontaminating potential, the short exposure was not thought to be disadvantageous. Tanks with sprays were cleaned with the use of approximately one-half the total volume of wash required for comparable sized tanks not spray equipped.

Generally, detailed radiation surveys showed tank activity levels to be highest at the bottom. Where this held true, final tank clean-up was accomplished with sufficient solution to cover only the contaminated area.

Certain of the vessels in Cells I and II served to catch decontaminating solutions which had been pumped through the extraction columns. Some clean-up was accomplished by these solutions during their time of residence in the tanks.

The A-1 dissolver in Cell I was more difficult than other tanks in the cell to decontaminate because it was not equipped with a spray line leading from the solution make-up tank (C-17) on the roof and was initially much more

highly contaminated. In addition, the dissolving operations carried out in this vessel required the application of heat (up to 105° C) for extended periods of time which may have had the effect of causing the contaminating ions to adhere more tenaciously. Decontaminants were run into the dissolver (A-1) from the solution make-up tank (C-17) through the dissolver off-gas pipe and entrainment separator. Since A-1 did not have a spray, it was necessary to fill the tank for each wash to insure clean-up at the top. Hot spots at welds remained at the conclusion of the program despite intensive efforts to eliminate them. These localized hot areas contributed significantly to radiation background in the region of the dissolver but were not shielded since they did not interfere with cell conversion.

The outlet from A-11, filter tank, a short length of 3-1/2 inch pipe, did not respond to any solution employed. Intense activity was apparently held up in weld flaws at either end of this pipe. Lead shielding 1-1/2 inches thick lowered apparent radiation from this source to a point which permitted adequate working time for completion of the minor amount of pipefitting proposed for this cell location. This shielding was planned for easy removal so that it would not provide a possible point for activity hold-up during subsequent hot operation of Cell I.

Because of leaking welds, it was necessary to cut off exterior piping from the fission product evaporator (E-1) Cell IV. Considerable contamination had seeped into and through weld fissures; it could not be removed either by internal or external washing of the tank. Since changes were planned, this piping and the associated tank flange connections were removed and discarded.

The Cell IV reboiler (E-4) was removed in order to retrieve corrosion samples which had been exposed in it. This tank, at the time of removal, had a 10 R/hr. level of contamination. It was unbolted from the column and removed from the cell. Corrosion samples were removed and the tank was then treated separately and the activity brought down to a level which would not hamper re-installation or give misleading results when used again in the acid recovery process. For this particular vessel, hydrochloric acid inhibited with a petroleum derivative (trade name "Acryl") was used as the decontaminant. Details are given in Section 4.4.

3.2 Column Decontamination

Citric, nitric and periodic acids were used for the preliminary treatment of all columns in Cells I and II. The solutions were fed from process head tanks through head pumps to the columns.

Later in the program it became apparent that A, B and C column decontamination was not proceeding at a satisfactory rate. Solutions containing from 0.3 per cent to 1.0 per cent hydrofluoric acid were then employed, but difficulty was experienced with pump check valve failures from corrosion and weld leaks in pipe lines from pumps to columns. This method of feeding the columns was abandoned in favor of delivering solutions through sampler suction lines which originated at the top of the columns and terminated at roof samplers. A 3 per cent hydrofluoric - 20 per cent nitric acid mixture was used as the availability of hydrofluoric permitted. Solutions were added to the empty columns, held in the columns for one hour, discharged completely, and the columns were then rinsed and again discharged.

Activity retention in column flanged connections proved troublesome and it was necessary to use lead to shield a column hot spot that did not yield to chemical treatment. The box built onto the bottom of C column continued to emit radiation at a level of 10 R/hr. after the vertical extraction sections were well cleaned. Heavy layers of lead were placed around this box as a temporary protection.

The distillation column in Cell IV had become highly contaminated (65 R/hr.) as the result of accidental discharge of activity from the evaporator (E-1). Decontamination procedures initially involved flooding of the column with 10 per cent citric and 10 per cent nitric acid solutions. These reagents proved ineffective; 5 per cent nitric - 0.3 per cent hydrofluoric acid was then employed. This opened a weld at the base of the column forcing abandonment of column treatment. Activity levels had been reduced to an average of 200 mr./hr. This was adequate since no maintenance work was planned for this part of the cell.

3.3 Pump Decontamination

Centrifugal pumps used to transfer both solvent and displacement fluid had become contaminated. They were partially cleaned by circulating washes through the impeller chamber. For a thorough job it was necessary to dismantle and perform a piece-by-piece decontamination in order to get at activity retained in packing and close-fitting parts.

3.4 Sampler Decontamination

All sample-taking assemblies associated with hot tanks or streams required extensive decontamination. Table II gives radiation levels before and after treatment.

TABLE II

Sampler Decontamination Results

Sampler Number	Location	Sample Origin	Internal Sampler Activity Levels	
			Before Clean-up	After Clean-up
A-1	Roof	Dissolver	10 R/hr.	75 mr./hr.
A-4	"	Feed Preparation tank	10 R/hr.	100 mr./hr.
A-12	"	Feed adjustment tank	10 R+/hr.	400 mr./hr.
A-13	"	Filter wash catch tank	5 R/hr.	35 mr./hr.
A-17	"	Fission product catch tank	7 R/hr.	50 mr./hr.
B-12	"	Fission product flowing stream - A Column	5 R/hr.	200 mr./hr.
B-13	"	Uranium stream - A column	35 R/hr	75 mr./hr.
E-1	Pipe tunnel	Fission product evaporator	10 R/hr	200 mr./hr.

The samplers were cleaned by dismantling and swabbing all exposed surfaces with concentrated nitric acid. Three treatments using this techique reduced internal activities to the levels noted. These levels were considered adequate since the contamination was within the sampler shields and would permit the taking of samples without operator over-exposure. Sampler lines from tanks to roof were effectively decontaminated in the course of taking the considerable number of samples required for the program of reagent evaluation. The E-1 sampler parts located in Cell IV but accessible from the Pipe Tunnel had failed to operate during the latter period of the Cell IV operation; circulation of solution from E-1 through this sampler was not possible. It was necessary to cut the sampler assembly loose and decontaminate it piece by piece.

3.5 Decontamination of Pipe Lines

Most of the pipe lines were cleaned adequately in the process of pumping solutions through the columns into catch tanks and in jetting solutions from tank to tank. Particular care was taken to flush all possible jet, pressure discharge, or overflow lines. Some lines needed more intensive treatment. This was done by filling the lines for a short time with 3 per cent hydrofluoric - 20 per cent nitric acid decontaminant, discharging the solution and rinsing with water. Where this technique was unsuccessful or could not be applied, the line was suitably shielded or removed.

3.6 Decontamination of Fittings (Jets, Valves, Flanges)

This type of equipment frequently proved difficult to decontaminate in place. The narrow internal clearances favored activity retention which was particularly hard to remove with reagents. Several jets had to be removed from the cells and disassembled for clean-up. Wherever possible, hot flange connections were unbolted and the faces and gaskets swabbed with concentrated nitric acid to obtain a quick, effective decontamination. Valves which could not be decontaminated in place with several internal washes had to be removed and disassembled for effective treatment.

3.7 Decontamination of Concrete and External Stainless Steel Surfaces

All cell floors and lower parts of the walls were lined at a previous time with thin gauge stainless sheets to prevent absorption of activity spills into porous concrete. The removal of contamination from these stainless steel surfaces usually involved washing with versene, citric acid, dilute nitric acid or scrubbing with a detergent. Such treatment sufficed to remove contamination which was not tenaciously held. Some hot spots were found which

did not readily wash off. Those spots not associated with welded joints were swabbed with hydrochloric acid and then rinsed. Where hot spots were associated with welded joints, it was considered undesirable to employ hydrochloric or concentrated nitric acids at the risk of creating pinhole leaks in the floor covering. Citric acid or versene was used to give partial decontamination.

Exterior tank surface contamination, when not concentrated at a welded joint, was usually treated successfully with nitric acid swab and a moderate amount of rubbing. No effective technique was found for chemically removing contamination from very fine weld fissures.

Several areas of unprotected concrete floor had become badly contaminated. The worst instance involved the dissemination over the West Gallery floor of a radioactive citric acid solution. Chemical treatment with caustic and intensive scrubbing with PAX detergent subsequently reduced activity levels to a point that workmen could get at the floor to remove a thin layer of concrete with air blasted steel shot*. This shot blasting did not remove sufficient activity to justify its use. Finally, it proved necessary to use air hammers to remove the top 2-3 inches of concrete and imbedded activity. In another instance, a spill of slightly contaminated acid solution was promptly cleaned up with only a trace of activity being retained by the floor. Apparently the successful decontamination of concrete which had been contacted by a penetrating (acidic) radioactive solution depended upon promptness of action. After penetration had progressed the only recourse was to surface shielding or removal of the contaminated concrete.

* The "Vacublaster" was used. This unit shot-blasts, draws back dust and shot, exhausts filtered air and recycles shot.

3.8 Entrainment Separator (A-5 and E-2) Decontamination

The entrainment separator (A-5) which functioned as a part of the dissolving equipment in Cell I became contaminated during use (500 mr/hr. at the time the cell was opened). Washes discharged from the roof tank, C-17, through the off-gas system into the dissolver (A-1) passed through the separator and effected a partial decontamination. Final decontamination was accomplished by closing the separator to A-1 drain valve and flooding the separator for one hour periods with 3 per cent hydrofluoric - 20 percent nitric acid. This wash procedure was followed three times. During subsequent conversion work the separator was disassembled and it was found that the interior parts had been reduced to an activity level of about 25 mr./hr.

When it became possible to enter Cell IV for short periods of time, the entrainment separator (E-2) positioned over and bolted to the evaporator was found to emit activity of greater than 10 R/hr. intensity. After partial decontamination, the unit was unbolted by the use of long-handled tools and removed from the cell. Upon removal the separator was found to have a spot emitting 100 R/hr. radiation. This had been shielded when in place by a heavy concrete beam. The presence of this high level radiation required that the unit be conveyed to a remote area and disassembled with long-handled tools. The parts were then cleaned individually. Contamination on some areas was not removed by nitric-hydrofluoric acid mixtures; there appeared to be a covering film which prevented attack of the steel. A mixture of nitric-hydrochloric acid readily etched the metal and removed the contamination. Such a powerfully corrosive reagent is not recommended for general use. In this instance, the corrosion was not objectionable since

these parts were to be used in a way that was not adversely affected by a slight loss of metal.

3.9 Off-Gas Condenser Decontamination

The off-gas condenser system positioned on the roof above Cell I had become contaminated with spots at valves and flanges reading in excess of 10 R/hr. It was found possible to wash the internal piping and chambers by cutting into a line which had delivered water to a spray ring located in the top condensation chamber. Connections were made so that chemical solutions could be forced through the spray ring into the condenser to flood the assembly. Solutions were held in the system for one hour, then discharged and replaced by water rinses. Since the area was partially shielded, complete clean-up was not required. Radiation was reduced from 10 R/hr. at various points to a maximum reading of 200 mr./hr.

4.0 Reagent Evaluation

The effectiveness of chemical reagents in the decontamination of stainless steel equipment was evaluated during the Pilot Plant clean-up. These studies had to be fitted into the program as the overall schedule permitted.

Due to time limitations it was not possible to develop a broad range of information on optimum reagent concentrations, temperature, contact times, pH and corrosion rates. To meet the requirements of the conversion schedule, it was frequently necessary to abandon the evaluation of a particular compound.

In addition to schedule restrictions, availability and cost were also determining factors in defining the scope of a reagent study. For instance, the cost of periodic acid was $28.20/lb. and only small amounts were available at the times needed. Its use on a scale involving hundreds of pounds could have

been justified only by evidence of superior effectiveness. This evidence was not immediately forthcoming from the study and only a small quantity (158 oz.) was used.

The progress of decontamination had to be followed closely in order to determine the results achieved by a particular reagent. Two techniques were employed: sampling of washes prior to discharge for gross beta count and the maintenance of equipment activity level records as indicated by "cutie pie" surveys. Both techniques were subject to shortcomings. At times tank and column samplers were taken out of service. In addition, no direct correlation existed between the radioactivity of a particular wash sample and the effectiveness of the reagent used. The beta count of the sample was affected by the decontaminating effect of the washes used immediately prior to the one sampled. Analytical results therefore showed only a general trend. Instrument measurements were useful within limits; during the initial periods of Cell I and IV clean-up the cells could not be entered to make surveys; as cell clean-up progressed, activity readings of particular pieces of equipment were obscured frequently by background effects.

4.1 Reagents Studied

The following reagents were used in Pilot Plant studies:

1. Nitric Acid
2. Hydrochloric Acid
3. Hydrofluoric Acid
4. Citric Acid
5. Oxalic Acid
6. Periodic Acid
7. Potassium Periodate
8. Ammonium Bifluoride
9. Caustic
10. Versene

These had been used previously in the Pilot Plant for decontamination work. Ammonium bifluoride and potassium periodate were used in acid solution as sources of hydrofluoric and periodic acids respectively when the latter were unavailable.

4.2 Analysis of Results

Table V in the Appendix presents a summary of data taken during decontamination of B-40. This tank had been used as a boildown tank in the concentration of product from the C column; its activity was approximately 500 mr./hr. initially. Decontamination from 500 mr./hr. to 50 mr./hr. was accomplished by washing the tank 30 times. The general trend of beta activity in solution samples showed a decrease as decontamination progressed. Activity of the first 3 washes, all 10 per cent nitric acid, averaged 5.8×10^3 beta counts/min/ml. A solution of 10 per cent nitric - 0.1 per cent hydrofluoric which followed analyzed 1.8×10^4 beta counts/min/ml. This increase of discharge solution activity presumably was due to the hydrofluoric acid content. When tank activity had been reduced to 100 mr./hr., three washes, one of 10 per cent nitric acid, one of 10 per cent citric acid, and one of 8 per cent nitric - 0.05 per cent potassium periodate were put into B-40 in the order noted. Each was heated to 100° C and held in the tank for 1 hour. Sample beta activity decreased and then increased again. Tank activity remained at 100 mr./hr. The succeeding solution, however, of 20 per cent nitric - 1.0 per cent hydrofluoric acid held in the tank for 1 hour at approximately 30° C showed an increased sample beta count and a sharp lowering of tank activity to 70 mr./hr. The final 3 washes containing 1.0 per cent hydrofluoric acid brought tank activity from 100 mr./hr. to 50 mr./hr.

The A-4 decontamination summary is presented in Table VI, Appendix. This tank had contained feed solutions of beta activities which exceeded 8×10^9 counts/min/ml. Preliminary feed adjustments were made in the tank usually without any application of heat. At the time decontamination treatments were started the activity level of A-4 was estimated to be in the range of 100-300 R/hr. After a series of 30 washes of various reagents, activity of the tank was reduced to 150 mr./hr. The second wash used was one containing 0.3 M citric acid - 0.5 M hydrochloric acid; it removed an unusual amount of contamination as indicated by the solution sample analysis of 2.5×10^7 beta counts/min/ml. Except for the reboiler (E-4) no further use of hydrochloric acid was made for tank decontamination because of the risk of corrosion damage. The last washes of 3 per cent hydrofluoric - 20 per cent nitric originated in the dissolver (A-1) and were passed through A-4 enroute to the waste tank (A-8). Despite the short residence time (10 minutes) of the washes in A-4, the tank was brought from an activity of 300 mr./hr. to 150 mr./hr.

It is interesting to note that A-4 and B-40 were each washed 30 times. A-4 was decontaminated from 100-300 R/hr. to 150 mr./hr., while B-40 was brought from an activity of 500 mr./hr. to 50 mr./hr. in the same number of treatments. There are several factors which may explain this apparent difference in amount of decontamination per wash. Approximately 70 per cent more hydrofluoric acid was used in the treatment of A-4 than B-40. A spray was used in decontaminating A-4; B-40 had no such facility. Since B-40 had been used as a boildown tank for acidic solutions containing activity, conceivably the contamination was more tenaciously held than in A-4 which had been heated infrequently.

Table VIII in the Appendix presents the B column decontamination history. Samples of the solutions were taken for 11 of the first 13 washes; subsequently the sampler was dismantled and inoperative. During this initial period covering 266 hours exposure, the column was washed with the following: 2-10 per cent nitric acid (210 hours), 2 per cent versene (8 hours), 5 per cent nitric - 0.1 per cent hydrofluoric acid (16 hours), 0.05 per cent perfodate (32 hours). Solution beta counts showed a decrease from 3.7×10^5 counts/min/ml. to 3.9×10^3 counts/min/ml.

A sudden increase in B column activity occurred after 314 hours of exposure to reagents. This resulted from an accidental spill-over of hot decontaminating solution from the A column. Activity which had dropped from an initial 700 mr./hr. to 345 mr./hr. rose to 6000 mr./hr. At this point, the technique for washing the column was changed from one which involved the constant flow of decontaminant through the column to one of static washing. In the latter technique solutions were poured into the column, held for one hour, and discharged. Final column clean-up from 6000 mr./hr. to 160 mr./hr. was accomplished in a total exposure time of 11 hours with 11 washes, 4 of which contained hydrofluoric acid. The reduction in apparent column activity from 1000 mr./hr. to the final 160 mr./hr. resulted in part from shielding placed on an adjacent wall and the removal of a hot pipe line from the area.

4.3 Evaluation of Inhibited Hydrochloric Acid

A solution containing hydrochloric acid and inhibitor was evaluated early in the program and appeared effective. The reboiler (E-4) was used for this test. The interior of the tank had become excessively contaminated by accidental discharge of hot solution from the evaporator (E-1). The tank was

Drawing # 14145

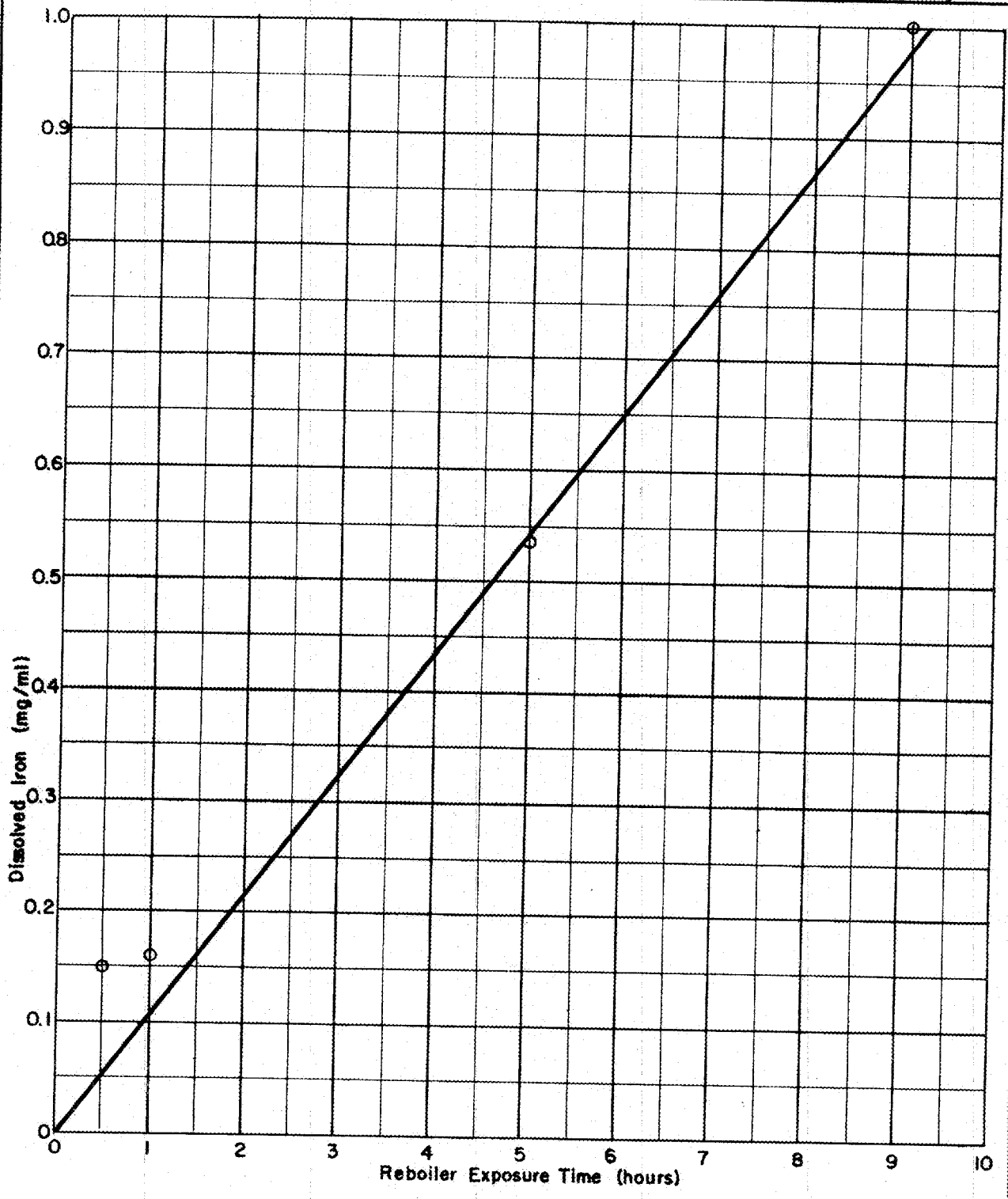

Fig. VI

DISSOLVED IRON IN DECONTAMINATING SOLUTION

vs

REBOILER EXPOSURE TIME

(Inhibited Hydrochloric Acid)

RBW 1-15-52

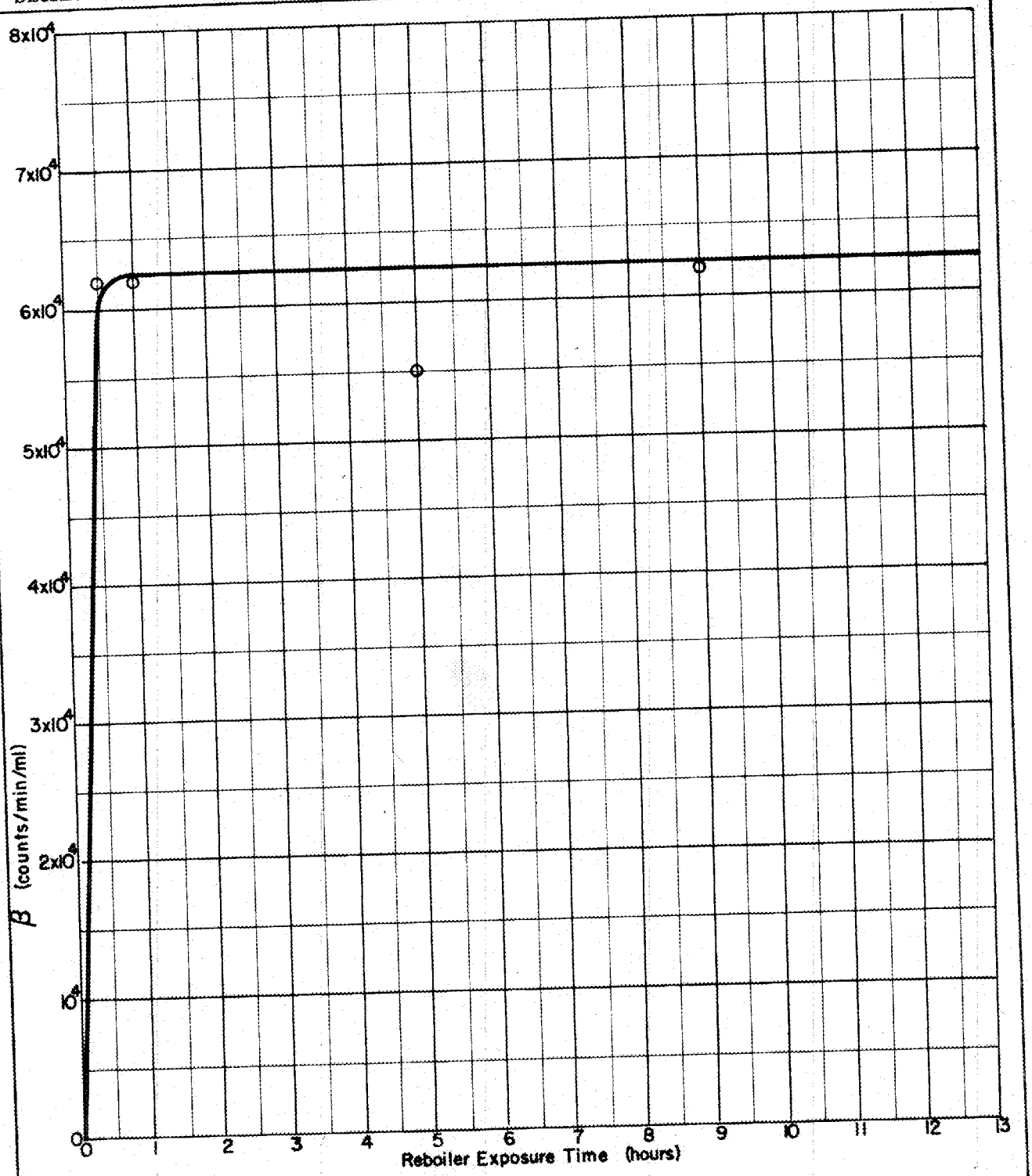

Fig. VII

TOTAL BETA ACTIVITY IN DECONTAMINATING SOLUTION

vs

REBOILER EXPOSURE TIME

(Inhibited Hydrochloric Acid)

RBW 1-15-52

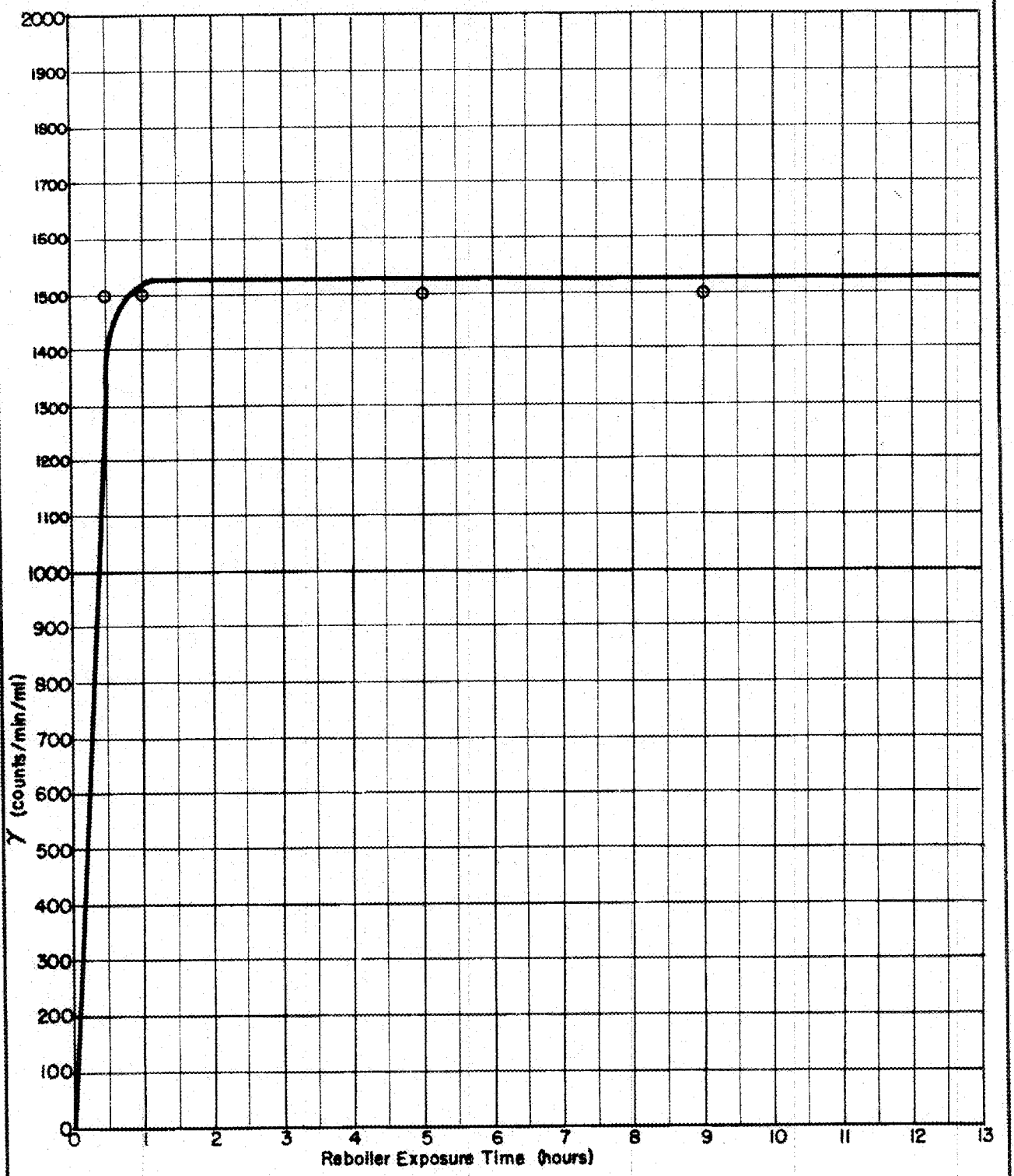

Fig. VIII

TOTAL GAMMA ACTIVITY IN DECONTAMINATING SOLUTION

vs

REBOILER EXPOSURE TIME

(Inhibited Hydrochloric Acid)

RBW 1-15-52

placed in an isolated area and filled with 22 l. of 38 per cent hydrochloric acid and 500 ml. of "Acryl" inhibitor. Samples of the solution were taken at frequent intervals for analysis of iron content, gross beta and gross gamma activities. Curves of beta and gamma count vs. exposure time (Figures VII, VIII) indicate that all decontamination by the solution apparently occurred within the first 15 minutes of treatment. Corrosion, as indicated by iron content of the samples, continued at a fairly constant rate during the period of exposure (Figure VI). It may be concluded that maximum decontamination with least corrosion can be obtained by limiting hydrochloric acid-stainless steel (Type 347) contact time to 30 minutes. The inhibited hydrochloric acid reduced tank activity internally from 500 mr./hr. to 200 mr./hr; the residual activity was apparently buried in weld fissures which could not be reached by the reagent.

No further use was made of inhibited hydrochloric acid for general equipment clean-up because of inadequate knowledge concerning the long-range performance of the inhibitor.

4.4 Conclusions

The mixture of 3 per cent hydrofluoric acid (by weight) and 20 per cent nitric acid (by weight) was found to be a promising reagent; it decontaminated rapidly without excessive corrosion of stainless steel equipment. The limited supply of hydrofluoric acid prevented its more extensive use. The quantities which could be obtained were largely responsible for the successful decontamination of the Pilot Plant to the reported low activity levels.

Nitric, citric and oxalic acids as used in these studies gave approximately equal decontamination results. The conditions of application for each reagent were not necessarily optimum. However, they proved generally satisfactory for use during the early stages of this particular decontamination assignment. Nitric acid was preferred since it was cheapest and most readily available.

As the activity level of a particular item of equipment was reduced by repeated treatment, the contamination became more difficult to remove. As this stage was reached, more aggressive decontaminants were required. Both hydrochloric acid and hydrofluoric acid met this requirement. Except for use in spot decontamination of exposed stainless steel surfaces, hydrochloric acid was not acceptable because of its corrosiveness.

Best decontamination results were obtained with the 3 per cent hydrofluoric - 20 per cent nitric acid solution, although its corrosiveness required that particular care be observed in its use. The corrosion of sound stainless steel was apparently not significant. However, faulty welds which had resisted concentrated nitric acid failed quickly when exposed to hydrofluoric acid concentrations of 0.3 per cent and greater. No tanks were thus affected but several small diameter pipe lines had to be rewelded because of hydrofluoric attack at welds. This reagent should be used at room temperature (approximately 25°C) <u>not heated</u>, to minimize corrosive attack.

Data indicated caustic and versene to be relatively ineffective. The latter was used without attention to pH adjustment and may consequently not have given optimum results.

Potassium iodate and periodic acid were used in very dilute solutions less than 0.5 per cent - and did not give results, in light of their high cost, warranting further use for general decontamination.

Ammonium bifluoride in nitric acid solution was used as a source for hydrofluoric acid. There was no evidence that the ammonium radical contributed toward decontamination.

PAX detergent found application in loosening superficial contamination from stainless steel cell floors and concrete floors in operating galleries and roof. It was not used in tank and column treatment.

Extensive investigation was not made of optimum contact time for the various reagents. Results obtained with inhibited hydrochloric acid indicated that all decontamination occurred within the first 15 minutes of exposure. Conceivably, the same would hold true for 3-20 reagent washes. If so, the total time required per wash could be reduced substantially over that specified for the recent decontamination program (1 hour exposure).

5.0 Decontamination Costs

Decontamination involved the expenditure for chemicals of an estimated $5400. The costs and quantities of the various items used were:

TABLE III

Reagent Costs

Chemical	Amount Used	Cost ($)
Nitric acid (tech. and C.P.)	64,000 lbs.	3220
Hydrochloric acid	522 lbs.	78
Hydrofluoric acid	560 lbs.	690
Citric acid	2165 lbs.	610
Oxalic acid	100 lbs.	73
Periodic acid	158 oz.	278
Potassium periodate	118 oz.	112

TABLE III (Cont'd)

Chemical	Amount Used	Cost ($)
Ammonium bifluoride	58 lbs.	32
Caustic	1500 lbs.	30
Versene	340 lbs.	238
PAX detergent	150 lbs.	39
Total chemical cost		$5400

Approximately 6000 man hours of operator labor were required to handle the decontamination work. In addition, charges for special maintenance work such as cutting and removing lines, removing and replacing jets and valves, loosening flanges, and welding cost an additional $2500 for labor and materials. Total direct costs were:

Chemicals	$ 5,400
Labor, 6000 man hours at $1.70	10,200
Maintenance	2,500
Total	$18,100

The use of 3 per cent hydrofluoric - 20 per cent nitric acid solution wherever possible for a decontamination program comparable to the one noted above offers economies of labor and chemical costs. Table IV shows estimated present and future (using 3-20 reagent) decontamination labor and chemical requirements.

TABLE IV

Estimated Present and Future Decontamination Cost Elements

Type Decont.	Relative Amounts of Labor Req'd. for Decontamination		Relative Chemical Costs	
	Present	Future	Present	Future
Tanks	50	15	75	60
Columns	25	12	15	12
Samplers	10	10	5	5
Other	15	15	5	5
Total	100	52	100	82

The types of decontamination listed involve different clean-up techniques and are not equally adapted to the use of 3-20 reagent. Estimates of future labor and chemical requirements reflect considerations of safety, adaptability of technique, and resistance of material to be treated to hydrofluoric acid. Based on the figures in Table IV, direct costs of a future decontamination using 3-20 reagent are thus estimated to be:

Chemicals	$4500
Labor	5300
Maintenance work	2500
	$12,300

This represents a reduction of 32 per cent from the cost ($18,100) previously noted and a reduction from 75 to 38 days time required to accomplish the decontamination. In addition, substantial indirect savings would accrue from decreased Pilot Plant downtime during decontamination.

The advantage of having a built-in decontamination spray system, as in Cell I tanks, and the additional cost of treating a tank of high activity vs. one of low activity became evident in an analysis of cost data for the treatment of the feed adjustment tank (A-4) and the CU boil-down tank (B-40). These tanks were of 300 gallon and 237 gallon capacities respectively. Initial A-4 activity was 100 R/hr., B-40 was 500 mr./hr; A-4 was equipped with an internal decontamination spray. The average volume per A-4 treatment was 350 liters; for B-40, 450 liters.

TABLE V

A-4, B-40 Actual Decontamination Cost Summary

Chemicals Used	A-4 Wt. (Lb.)	A-4 Cost ($)	B-40 Wt. (Lb.)	B-40 Cost ($)
Nitric Acid, tech., at $0.03/lb.	5000	150.00	5500	165.00
Hydrofluoric acid, tech., at $0.23/lb.*	100	23.00	60	13.80
Caustic, 50%, tank car, at $0.02/lb.	240	4.80	700	14.00
Periodic acid, C.P., at $1.76/oz.	12(oz.)	21.00		
Potassium periodate, C.P. at $0.93/oz.			16(oz.)	15.00
Citric acid, C.P. at $0.28/lb.	40	11.20	40	11.20
Oxalic acid, C.P. at $0.73/lb.	45	33.00		
Versene, at $0.70/lb.	28	20.00	20	14.00
Total Costs		263.00		233.00
Operator labor required:				
A-4 - 1 man hour per tank at $1.70/ man hour 30 tank treatments		51.00		
B-40 -2 man hour per tank at $1.70/ man hour (lack of spray system doubled labor required over A-4 requirements) 30 tank treatments				102.00
Total Direct Costs		$314.00		$335.00

* C.P. hydrofluoric acid cost is $1.23/lb; the tech. grade cost of $0.23/lb., is more realistic.

Pilot Plant decontamination in the past had followed the practice of using first one reagent and then another in an effort to remove activity. Thus, the cost estimate, although based on a list of chemicals reflecting more the requirements of the reagent evaluation program than an expeditious decontamination, is felt to be reasonably representative of past Pilot Plant practice.

The principal difference in A-4, B-40 decontamination costs was in operating labor required; the A-4 decontamination spray system permitted each treatment to be carried out with one-half the labor needed for B-40 which was not equipped with a spray. The A-4 chemical cost was higher primarily because of the greater amounts of hydrofluoric and oxalic acids used to reduce the higher level of activity in A-4.

6.0 Fission Product Distribution in Column Decontamination Solutions

During the time that decontaminating solutions were pumped from head tanks to columns, samples of wash solutions were taken over a two day period from A, B and C columns and submitted for determination of gross beta, ruthenium beta, and niobium beta. The results are summarized in Table IX, Appendix. Information was desired on the deposition of niobium on stainless steel surfaces during Purex Process operation.

Decontamination of the A column removed niobium and ruthenium in a ratio of approximately 60 to 1; the average niobium - ruthenium ratio in the A column during processing of hot feeds was 0.10 to 1. Decontamination solutions from the B and C columns showed a somewhat lesser preponderance of niobium.

Since the data was taken after some decontamination of column surfaces had been accomplished, the possibility existed that earlier treatment had

removed ruthenium preferentially and that the niobium-ruthenium ratio changed, as the decontamination progressed, to the values noted. The chemicals used initially were not known to be specific for ruthenium; evidence favors the theory that niobium deposited on stainless steel during process operation.

Information was not developed concerning the equilibrium concentrations of niobium in process solutions and that on column walls. However, it was determined on the basis of known total beta curies fed to the A column during Purex Process operation and estimated beta curies removed during decontamination, that deposited beta activity represented only 0.002 per cent of the total fed (calculations are in Appendix, Table X). Although the estimate of activity stripped could be in error considerably, the range of values is such that the amount of deposited activity can be considered insignificant in so far as Purex operation is concerned.

7.0 Electrodecontamination

A brief investigation was made of electrodecontamination as a possible technique for Pilot Plant use. D-14 in Cell III, which had been used as an aqueous decantation tank in the solvent recovery system, was selected for trial because of its high activity level (15 R at the bottom) and accessability for making the necessary electrical connections. Over a period of 24 hours of continuous treatment at a current density of 0.04 amperes per square inch of internal tank area and using a 1 inch x 4 foot stainless pipe as the cathode, tank activity was reduced to 700 mr./hr. This residual activity was lodged in flanged connections at the bottom of the tank and was not further reduced by additional electrodecontamination effort.

Despite the successful removal of tank activity by this technique, further use was abandoned for these reasons: (1) Since activities in lines, flanges, and valves were not touched by electrostripping, it would have been

necessary to use reagents for these items. (2) Pulse columns were not adaptable to electrodecontamination because of their length and difficulty of withdrawing pulse plates for electrode insertion. (3) Only a few tanks in the Pilot Plant were equipped with properly located top openings for the insertion of the cathode. (4) The extension of numerous pipe lines into the body of the tank interferred with efficient stripping. (5) Many tanks could not be reached until a preliminary chemical decontamination had been completed.

8.0 Materials of Construction

Types 347 and 309 Cb stainless steel had been employed in general equipment fabrication. Inspection of internal tank and column surfaces showed no evidences of serious attack by the reagents employed for decontamination. All surfaces were bright and clean. Since hydrofluoric acid in the 0.5 - 4.0 per cent range of concentrations was the most corrosive chemical employed, its judicious use for decontamination can be considered feasible. The nitric acid content of the 3-20 reagent is essential in minimizing corrosion. Exposure times of stainless steel to hydrofluoric acid should not exceed one hour.

Hydrofluoric acid solutions attack faulty welds which had resisted concentrated nitric acid. Every instance of weld failure occurred in small diameter pipe lines which were difficult to weld without inclusion of flux. Sound welded joints were not attacked. Normally, welding of the best quality has been required for fabrication of stainless steel facilities intended for the processing of hot solutions. This becomes a doubly important consideration if exposure to hydrofluoric acid is contemplated.

The Milton Roy positive displacement metering pumps used during a part of the decontamination program to pump reagents through the columns did not show evidences of chemical attack on any of the stainless steel working parts.

Solutions handled included 10 per cent nitric acid, 2 per cent versene, 10 per cent citric acid, 0.3 per cent hydrofluoric acid, 10 per cent oxalic acid, 10 per cent caustic, 0.05 per cent potassium periodate. Leakage of hydrofluoric acid solutions from pump pistons resulted in some severe local corrosion of cast pump bases. Non-stainless steel pump ball checks were destroyed by 0.3 per cent hydrofluoric acid solution; stainless balls served without evidence of corrosive attack.

9.0 Recommendations

1. The work of Pilot Plant tank decontamination would be greatly facilitated by the installation of spray lines from a centrally located solution makeup tank to all tanks which are expected to pick up radioactivity. Cells II, III and IV tanks are not now so equipped.
2. New process equipment for the Purex Pilot Plant should be installed only after due consideration has been given to means of decontamination.
3. The reagent mixture of 3 per cent hydrofluoric - 20 per cent nitric appears to be very promising as an effective, economical decontaminant. It is strongly recommended that further broad studies be made of its properties.

APPENDIX

DECONTAMINATION - PUREX PILOT PLANT

APPENDIX

TABLE VI

Decontamination Record

B-40, ICU Evaporator Tank, Cell II

Date	Decontaminating Solution	Contact Time	Sample Analysis Gross Beta (counts/min/ml)	*Tank Activity (mr./hr.)
8/28/51	10% HNO_3	2 hr. at 100°C	6.5×10^3	500
8/29/51	10% HNO_3	1 hr. at 100°C	9.0×10^3	
8/30/51	10% HNO_3	1 hr. at 100°C	2.0×10^3	
8/31/51	10% HNO_3 - 0.1% HF	1 hr. at 100°C	1.8×10^4	
	10% HNO_3	1 hr. at 100°C	-	
	10% HNO_3	1 hr. at 100°C	-	
9/1/51	10% HNO_3	2 hr. at 100°C	8.0×10^3	
9/3/51	10% HNO_3	1 hr. at 100°C		
	10% HNO_3	1 hr. at 100°C		
9/7/51	10% HNO_3	1 hr. at 100°C		
	10% HNO_3	1 hr. at 100°C	Sampler	
9/8/51	10% HNO_3	1 hr. at 100°C	inoperative	345
	10% HNO_3	1 hr. at 100°C		
9/9/51	8% HNO_3 - 0.05% KIO_4	1 hr. at 100°C		325
9/10/51	2% citric	1 hr. at 100°C		
9/10/51	2% versene	1 hr. at 100°C		
9/11/51	10% NaOH	1 hr. at 100°C		
9/11/51	7% HNO_3 - 0.5% HF	1 hr. at 100°C		180
9/12/51	10% HNO_3	1 hr. at 100°C		
9/13/51	10% HNO_3	1 hr. at 100°C	6×10^2	130
9/14/51	10% HNO_3	1 hr. at 100°C	Sampler inoperative	
9/15/51	8% HNO_3 - 0.4% HF	2 hr. at 100°C	Sampler	
9/17/51	10% NaOH	1 hr. at 100°C	inoperative	100
9/18/51	10% HNO_3	1 hr. at 100°C	9.0×10^2	
9/19/51	10% citric	1 hr. at 100°C	1.2×10^2	
9/20/51	8% HNO_3 - 0.05% KIO_4	1 hr. at 100°C	5.0×10^2	100
9/24/51	20% HNO_3 - 1.0% HF	1 hr. at Rm.T.	2.0×10^3	70
9/25/51	20% HNO_3 - 1.0% HF	10 min. at Rm.T.	-	60
9/27/51	20% HNO_3 - 1.0% HF	10 min. at Rm.T.	-	50

* "Cutie Pie" reading at tank bottom - 6" from tank surface.

APPENDIX

TABLE VII

Decontamination Record

A-4, Preliminary Feed Adjustment Tank, Cell I

Date	Decontaminating Solution	Contact Time	Sample Analysis Gross Beta (counts/min/ml)	*Tank Activity (mr./hr.)
8/23/51	10% NaOH	7 hr. at Rm.T.	2.0×10^6	100,000 (est.)
	(0.3 M citric acid and 0.5 M HCl)	1 hr. at Rm.T.	2.5×10^7	
8/27/51	5.0% HNO_3 - 0.3% HF	1 hr. at 100°C	5.0×10^5	
8/28/51	5.0% HNO_3 - 0.3% HF	1 hr. at 100°C	2.3×10^5	
	5.0% HNO_3 - 0.3% HF	1/2 hr. at 100°C	1.0×10^5	
9/1/51	5.0% HNO_3 - 0.3% HF	1 hr. at 100°C	-	
9/2/51	10% NaOH	2 hrs. at 100°C		
9/3/51	5.0% HF - 0.3% HF	1 hr. at 100°C	Sampler	
9/4/51	10% HNO_3	1 hr. at 100°C	inoperative	
9/7/51	10% HNO_3	2 hrs. at 100°C		
9/8/51	10% HNO_3	1 hr. at 100°C	2.0×10^3	
9/9/51	10% HNO_3	1 hr. at 100°C	3.3×10^4	
9/10/51	10% citric acid	1 hr. at 100°C	2.6×10^3	
9/11/51	10% HNO_3	1 hr. at 100°C	-	2000
9/12/51	5% oxalic acid	1 hr. at 100°C	3.5×10^3	
9/12/51	10% HNO_3	1 hr. at 100°C	-	1500
9/12/51	10% HNO_3	1 hr. at 100°C	-	
9/14/51	5% HNO_3 - .3% HF	1 hr. at 60°C	3.4×10^4	
9/15/51	10% HNO_3	1 hr. at 100°C	3.0×10^4	
9/17/51	10% citric acid	1 hr. at 100°C	1.8×10^4	
9/19/51	10% HNO_3 - 0.1% HF	1 hr. at 100°C	-	
9/22/51	10% HNO_3 - .25% H_5IO_6	1 hr. at 100°C	1.1×10^4	1000
9/25/51	15% HNO_3 - 1.5% HF	1 hr. at Rm.T.	-	800
9/26/51	10% HNO_3 - 0.025% H_5IO_6	1 hr. at 100°C	9.0×10^3	
9/27/51	15% HNO_3 - 1.5% HF	1 hr. at Rm.T.	-	500
10/1/51	20% HNO_3 - 3.0% HF	1 hr. at Rm.T.	-	300
10/8/51	20% HNO_3 - 3.0% HF	10 min. (A-4 intermediate in transfer of solution from A-1 to waste)	no samples taken	200
10/9/51	20% HNO_3 - 3.0% HF		"	225
10/10/51	20% HNO_3 - 3.0% HF		"	200
10/11/51	20% HNO_3 - 3.0% HF		"	150

* "Cutie Pie instrument determination - tank bottom at 6 inches.

TABLE VIII

Decontamination Record

B Column, Cell II

Date	Decontaminating Solution	Contact Time	Total Contact Time	Sample Analysis Gross Beta (counts/min/ml)	Column Activity* (mr/hr.)
8/28/51	5% HNO_3 - 0.1% HF	16 hr.		3.7×10^5	700
8/29/51	5% HNO_3 - 0.05% KIO_4	8 hr.	24 hr.	3.5×10^5	
8/30/51	2% HNO_3	12 hr.	36 hr.	1.3×10^4	
8/30/51	4% HNO_3	12 hr.	48 hr.	3.9×10^3	
8/31/51	2% versene	8 hr.	56 hr.	-	
9/1/51	10% HNO_3	24 hr.	80 hr.	1.0×10^4	
9/2/51	10% HNO_3	24 hr.	104 hr.	6.5×10^3	
9/3/51	10% HNO_3	18 hr.	122 hr.	6.4×10^3	
9/4/51	5% HNO_3 - 0.05% KIO_4	24 hr.	146 hr.	-	
9/5/51	10% HNO_3	72 hr.	218 hr.	3.8×10^4	
9/8/51	10% HNO_3	24 hr.	242 hr.	8.6×10^3	345
9/10/51	10% HNO_3	24 hr.	266 hr.	3.9×10^3	455
9/10/51	2% citric acid	48 hr.	314 hr.		400
9/12/51	3% HNO_3 - 1% HF	18 hr.	332 hr		1000
9/13/51	10% HNO_3	24 hr.	356 hr.		6000
9/14/51	5% HNO_3 - 4% HF	1 hr.	357 hr.	Sampler inoperative	
9/14/51	5% HNO_3 - 4% HF	1 hr.	358 hr.		
9/15/51	5% HNO_3 - 4% HF	1 hr.	359 hr.		
9/15/51	5% HNO_3 - 4% HF	1 hr.	360 hr.		
9/18/51	10% citric acid	1 hr.	361 hr.		1000
9/18/51	10% oxalic acid	1 hr.	362 hr.		
9/18/51	10% HNO_3	1 hr.	363 hr.		
9/21/51	0.4% KIO_4	1 hr.	364 hr.		500
9/22/51	10% HNO_3	1 hr.	365 hr.		
9/22/51	10% citric acid	1 hr.	366 hr.		
9/22/51	0.1% H_5IO_6	1 hr.	367 hr.		160

* "Cutie Pie" reading - 6 inches from column 8 feet from floor.

APPENDIX

TABLE IX

Niobium/Ruthenium Distribution in Pulse Columns

Column	Stream	Ruthenium counts/min/ml	Niobium counts/min/ml	Nb/Ru	Remarks
A	-	1.26×10^3	8.13×10^4	72.5	Sampled at 8 hrs.
A	-	1.65×10^3	9.07×10^4	55.0	Sampled at 8 hrs.
A	-	5.13×10^2	3.29×10^4	64.0	Sampled at 24 hrs.
A	-	3.06×10^2	1.71×10^4	56.0	Sampled at 24 hrs.
A*	AP	3.65×10^5	1.91×10^4	0.055	-
A*	AW	3.3×10^8	8.2×10^7	0.25	-
B	-	2.19×10^2	1.88×10^3	8.6	Sampled at 8 hrs.
B	-	3.55×10^2	2.17×10^3	6.1	Sampled at 8 hrs.
B*	BU	1.4×10^5	1.2×10^4	0.086	-
B*	BP	2.0×10^6	1.28×10^5	0.064	-
C	-	2.77×10^3	2.27×10^3	0.82	Sampled at 8 hrs.
C	-	6.53×10^3	9.3×10^3	1.42	Sampled at 8 hrs.
C	-	2.07×10^2	5.28×10^2	2.55	Sampled at 24 hrs.
C	-	6.92×10^2	5.62×10^2	0.81	Sampled at 24 hrs.
C	-	1.71×10^4	3.42×10^4	2.0	Sampled at 30 hrs.
C	-	2.11×10^4	3.62×10^4	1.7	Sampled at 30 hrs.
C	-	9.69×10^2	3.0×10^3	3.1	Sampled at 48 hrs.
C	-	3.92×10^2	2.3×10^3	5.8	Sampled at 48 hrs.
C*	CW	5.86×10^4	5.95×10^3	0.101	-
C*	CU	5.8×10^4	4.15×10^3	0.071	-

* Data from Pilot Plant Purex Run IHP-13

TABLE X

Calculation of A Column Beta Deposition

In the following calculation, total beta activity stripped from the A column during decontamination is estimated. Beta activity fed to the column in Runs #6 through #14 was calculated to be 7.85×10^4 curies. Since a new A Column was installed following Run #5, feeds for Runs #6-14 represent the total source of activity removed during decontamination.

Date	Solution	Vol. Sol'n. Pumped (L)	Analysis of Discharged Decont. Sol'n β c/m/ml	Total β Activity in Discharged Sol'n c/min.
8/29/51	0.0015 M potassium periodate	150	3.8×10^5	57.0×10^9
8/30/51	2.0 per cent nitric acid	300	8.0×10^4	24.0×10^9
8/30/51	4.0 per cent nitric acid	150	5.2×10^4	7.8×10^9
8/31/51	2.0 per cent versene	150	5.0×10^4*	7.5×10^9
8/31/51	10.0 per cent nitric acid	300	4.0×10^4	12.0×10^9
9/2/51	10.0 per cent nitric acid	400	7.0×10^4	28.0×10^9
9/3/51	10.0 per cent nitric acid	150	5.5×10^4	8.2×10^9
9/3/51	10.0 per cent citric acid	200	1.5×10^5	30.0×10^9
9/4/51	0.0015 M potassium periodate	300	7.2×10^4	21.6×10^9
9/5/51	10.0 per cent nitric acid	150	4.0×10^4*	6.0×10^9
9/5/51	10.0 per cent nitric acid 0.25 per cent hydrofluoric acid	300	2.0×10^5*	60.0×10^9
9/7/51	6.0 per cent nitric acid	600	7.0×10^4	42.0×10^9
9/10/51	2.0 per cent citric acid	150	2.5×10^5	37.5×10^9
9/12/51	3.5 per cent nitric acid 1.0 per cent hydrofluoric acid	130	5.0×10^4*	6.5×10^9
9/13/51	10.0 per cent nitric acid	800	1.0×10^4	8.0×10^9
9/15/51	6.0 per cent nitric acid 3.0 per cent hydrofluoric acid	27		
9/15/51	6.0 per cent nitric acid 8.0 per cent ammonium bifluoride	27		
9/15/51	6.0 per cent nitric acid 3.0 per cent hydrofluoric acid	27		
9/18/51	10.0 per cent citric acid	27	2.0×10^5*	38.4×10^9
9/18/51	10.0 per cent nitric acid	27		
9/18/51	10.0 per cent oxalic acid	29		
9/18/51	20.0 per cent nitric acid 3.0 per cent hydrofluoric acid	28		
			Total	394.5×10^9

* estimated - sample not available for analysis

$$394.5 \times 10^9 \text{ counts/min} \times \frac{10 \text{ disint./count}}{60 \text{ sec./min.}} \times \frac{1}{3.7 \times 10^{10} \text{ disint./sec./curie}} = 1.77 \text{ beta curies}$$

$$\frac{\text{curies beta activity removed} - 1.77}{\text{curies beta activity fed} \quad 7.85 \times 10^4} \times 100 = 0.002 \text{ per cent}$$

[redacted]

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