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PILOT PLANT SECTION

CORROSION DATA FROM THE ORNL PUREX PILOT PLANT ACID RECOVERY EQUIPMENT

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J. W. Landry and J. W. Ullmann

DATE ISSUED

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0.0 Abstract

This report summarizes the corrosion data obtained for nine construction materials located throughout the ORNL Purex Pilot Plant Acid Recovery Unit during seven hundred hours of operation.

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1.0 Introduction

The nitric acid used as a salting agent in the Purex process can be recovered for reuse by evaporation and fractionation with a resultant reduction of waste volume and increase in fission product concentration. Acid raffinate IAW, from the tributyl phosphate - hydrocarbon diluent system, containing from 300 to 25,000 beta curies of mixed fission products per run, has been processed in the ORNL Pilot Plant Acid Recovery Unit during the Purex runs made in the spring of 1951.

In order to determine the corrosion resistance of equipment in the presence of boiling nitric acid and fission product radiation, ninety-eight specimens of stainless steel (Types ELC-304-CS*, CS-309-Cb, ELC-316, and 329, plain; ELC-304 and 309-Cb, plain and welded; and 347, welded), tantalum, and titanium were exposed in the column packing and in the liquid, vapor, and liquid-vapor interfaces in the evaporator and reboiler. The specimens were exposed to seven hundred hours of steady state operation, seven hundred additional hours during which solutions of steady state concentration were being heated, cooled, or stored, and two series of decontamination treatments.

At the conclusion of six runs (made with the waste from uranium slugs irradiated to 70, 45, 120, 120, 450, 450 megawatt days per ton), the acid recovery unit was dismantled for inspection of the equipment and removal of the corrosion specimens.

Complete details are presented in this report for the metallurgical examination of the 309-Cb, CS-309-Cb, 329 and 347 stainless steel specimens. Because of the radiation involved, examination of the ELC-304, ELC-316 and titanium specimens will require several more months for completion.

* CS refers to commercially sensitized 1 hr. at 1250°F, water quenched.

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2.0 Summary

Materials of construction suitable for the Purex acid recovery system in the presence of kilocurie quantities of fission products have been determined.

All of the stainless steels tested are suitable for construction of the evaporator, vapor line, and column above the feed point. For the lower section of the column Type 329 or 309-Cb stainless steel is recommended. Although by visual inspection the Type 347 stainless steel reboiler evidenced good corrosion resistance, the specimens within the reboiler indicated that a tantalum lining may be desirable.

No direct effect of radiation upon metallic corrosion was observed, but Teflon gasketing should be avoided because of its embrittlement during exposure.

All the specimens evaluated in the evaporator (boiling 8N nitric acid) showed a penetration rate of four mils per year or less. In the reboiler (boiling 12N nitric acid) corrosion rates ranged from 0 (tantalum) to 70 mils per year (titanium) with the stainless steels in the region of 10 to 25 mils penetration per year. The specimens placed at the liquid-vapor interface in the reboiler generally showed the greatest attack; in the evaporator, this trend was not observed. In the presence of radiation, corrosion by the strong boiling acid in the reboiler is believed to have been aggravated by partial decomposition of the Teflon spacers which had been placed between the specimens.

In the fractionating tower the corrosion rate of 309-Cb stainless steel rings varied from less than 0.1 mil per year at the acid-depleted top section to 7 mils per year just above the reboiler. There was a peak of 0.7 mil per year in the penetration versus height curve (Figure 6) ten inches above the vapor feed point to the column. This probably was the 23 percent nitric acid region where

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Summary (continued)

chlorides may have accumulated to accelerate the corrosion.

Welding and sensitization of stainless steel specimens produced different effects upon the rate of corrosion. In the reboiler Type ELC-304 stainless steel welded specimens lost less weight than unwelded pieces by a factor of two; sensitized Type 309-Cb specimens lost twice as much weight as unsensitized specimens. In the evaporator no similar trend was observed.

The corrosion data reported include the effect of the decontamination treatments. Penetration rates corrected for the corrosion due to decontaminating solutions were from 30 to 70 percent lower.

3.0 Equipment

3.1 Acid Recovery Unit

The acid recovery unit concentrates the highly radioactive Purex IAW to a small volume and separates from it water and 57 percent nitric acid essentially free from radioactivity (Figure 1). The unit consists of an evaporator in which 95 percent of the acid and water are removed from the IAW, and a column in which the acid vapors from the evaporator are fractionated.

The evaporator is a twenty gallon conical bottom welded tank with steam jackets below the operating liquid level. Unannealed 309-Cb stainless steel is the material of construction. The wall thickness in the section exposed to liquid is 3/16 inch. Equilibrium evaporation at constant liquid level is conducted using continuous feed.

An entrainment separator of 309-Cb stainless steel is mounted over the evaporator and utilizes Raschig ring packing and a downdraft cyclone cone. The

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Acid Recovery Unit (continued)

separator is flanged to the evaporator and to the vapor pipe with Teflon ring type gaskets.

A three inch pipe of 347 stainless steel conducts the vapor from the separator to the fractionator which is a packed column with a reboiler at its base. The column is flanged to the vapor line, reboiler, and condenser with Teflon ring type gaskets. The vapor fed column consists of a five foot upper section of six inch diameter pipe and a ten foot lower section of four inch diameter pipe, both constructed of Type 347 stainless steel. The Raschig rings with which the column is packed were cut from half inch welded seamed pipe, Type 309-Cb stainless steel.

The reboiler is a three gallon conical tank with a steam jacket below the overflow liquid level and is welded from 3/16 inch unannealed Type 347 stainless steel. The submerged tubing and pipe within are also of Type 347 stainless steel with the exception of a tantalum bayonet heater and a bubble baffle of 40 mesh 80 gauge Type 316 stainless steel screen which is wrapped around the instrument dip tubes. The function of the reboiler is to maintain a concentration of 57 percent nitric acid by boiling at a fixed temperature. The product acid overflows at the constant operating liquid level.

The Type 347 stainless steel welded condenser contains seven four - foot lengths of one inch tubing in parallel within a water jacket. The acid recovery unit is vented at the condenser outlet. Air purge type instrumentation is used in the evaporator and reboiler.

General fabrication throughout the acid recovery unit is by welding without annealing, and joints are made with welded flanges gasketed with Teflon.

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3.2 Corrosion Specimens

3.21 Specimens

Table 6 shows the types of material and testing locations of the ninety-eight specimens which were prepared. Of the fifty-four rectangular billets used (average size 2 inches by 3/4 inch x 1/4 inch), sixteen bore weldments. For mounting, all billets had 1/4 inch holes, some chamfered and some not. Forty 7/8 inch by 7/8 inch Type 309-Cb stainless steel Raschig rings, spaced at an average interval of four inches, were used as part of the column packing. For inclusion in the evaporator and reboiler four rings were reduced to a length of 1/4 inch to conform with the billet mountings. Measured weights and dimensions of the rings and billets are included in Table 7. All specimens were stamped for identification.

3.22 Location and Mounting of Specimens

The general location of corrosion specimens within the acid recovery unit is shown in Figure 1, and specific locations are shown in Table 6 with details of the column specimens in Figure 3 and Column 2 of Table 7.

The evaporator specimens were mounted on a stainless steel staff flanged through a nozzle on the evaporator top. The staff and specimens could be removed without opening the evaporator. Teflon bushings and spacers in contact with the specimens prevented contact with the mounting bolts, staff, and adjacent specimens. Figure 3 illustrates this method.

The reboiler specimens were mounted similarly; however, interior space restrictions did not allow an assembly that could be removed through the 1-1/2 inch pipe nozzle at the top of the reboiler. Reboiler specimens were clamped

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Location and Mounting of Specimens (continued)

into jigs as illustrated in Figure 4. To prevent contact with vessel walls, tubing, screen, and tantalum heater, the specimens in each jig were surrounded by a Teflon cover. It was necessary to open the reboiler to remove the specimens.

The column specimens were spaced on a Teflon cord and suspended in the column while packing was poured around them. Figure 5 illustrates this method and Figure 3 and Column 2 of Table 7 show the grading of the specimens through the column packing. Several billets were placed around the point in the column at which the concentration of the liquid phase was 23 percent nitric acid because duPont experience indicated chlorides may accumulate there. The 50 percent nitric acid point was arbitrarily selected for locating a few billets in the lower section of the column.

4.0 Procedure

4.1 Purex Acid Recovery Runs- See Figure 1, Flow Sketch

The IAW from six Purex runs made from slugs varying in irradiation from 45 to 450 MWD/T was processed in the acid recovery unit. About 175 gallons of IAW was processed per run. A typical composition is given below:

Table 1

Typical Composition of Purex IAW

Water	87%	
Nitric Acid	13%	
Fission Products / Stable Isotopes	15-80	grams/run
Amsco 123-15	<0.6*	grams/run
Tributyl Phosphate	<0.6*	grams/run
Uranium	1.3	grams/run
Sodium Nitrite	550	grams/run

* lower limit of analysis

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Purex Acid Recovery Runs (continued)

The IAW was added to the evaporator at a rate of about four gallons per hour for approximately fifty hours per run.

By controlling the steam pressure in the evaporator jackets, the boiling rate was adjusted to maintain a constant liquid level in the evaporator. For steady state operation, the vapor rate and acidity equalled that of the feed; otherwise concentration changes occurred in the evaporator solution to adjust the vapor acidity until a balance was reached.

In the vaporization process, the evaporator liquid became adjusted to a concentration one theoretical stage different from the vapor. As a result of internal reflux due to heat losses in conducting the vapor from the boiling interface to the column an additional half stage was obtained. Based on a 2.2N feed, the evaporator solution would be adjusted to about 9.5N. Since an acidity above 9N was considered dangerous because of possible ruthenium volatilization, and subsequent contamination of recovered acid, the feed was diluted part for part with water. The evaporator solution then adjusted itself to an equilibrium concentration of 8N nitric acid. The radio-salts remained in the evaporator solution except for one part per million which escaped with the vapor.

The volume of solution in the evaporator was maintained at twenty gallons during the period in which the feed was on. When the feed was exhausted, the fission product concentrate was generally stored in the evaporator until it could be transferred to either the isotope processing facility or to waste storage. However, after the first run it was desired to investigate further concentration of the evaporator solution and the solution was evaporated to

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Acid Recovery Unit Runs (continued)

seven gallons before transferring. This was done by feeding water to the evaporator during boildown in order to maintain the same acidity in the evaporator solution as during the run. During this period the evaporator interface specimens were entirely in the vapor phase; all other conditions in the acid recovery unit were the same as during a regular run. At the conclusion of the fifth run, the evaporator solution was held and the feed resumed from the sixth run. Combining of the fifth and sixth runs doubled the radiation in the evaporator during the sixth run but resulted in no other changes in conditions of operation for the acid recovery unit.

The 1.1N acid vapors were fed to the packed column, one-third the distance down from the top of the column. The feed was fractionated with acid (liquid) leaving the reboiler at the base of the column and water vapor leaving from the top of the column. In order for the nitric acid to be reused for slug dissolving, 55 percent concentration is required. To provide for 2 percent dilution (through jetting) 57 percent nitric acid was set as the standard for the acid recovery unit. Since dilute acid is supplied to the reboiler at constant rate, controlling the reboiler boiling rate set the concentration of the acid produced. Hot water equal to 2/3 the amount separated from the evaporator vapor was added for column reflux.

For purposes of startup and otherwise gaining operating time when IAW was not available the unit was operated on a feed of the same acid concentration as the IAW feed but devoid of radioactivity.

Steam pressure required was 37 psig in the evaporator jackets and 60 psig in the reboiler jacket. Variations due to controlling were within + 5 psig, - 10 psig.

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4.2 Treatment of Specimens

4.21 Measurements and Normal Chemical Exposure

The dimensions of all specimens were measured after some of the ORNL specimens were repunched to give uniform mounting holes for all billets and after the burrs were polished off the packing ring specimens. Preparatory to weighing, the specimens were washed in acetone to dissolve wax and grease, rinsed in alcohol, and dried. The specimens were then weighed to the nearest milligram on an analytical balance. A number of representative specimens of each of the twelve types were weighed in water to determine the specific volume. The specimens were assembled and mounted in the acid recovery unit as described in Section 3.22.

The specimens were exposed for six Purex runs plus several synthetic runs in which non-radioactive feed was used. There was exposure also to decontamination solutions as described in Section 4.23. Radiation exposure is described in Section 4.22.

Advance data on corrosion in the acid recovery unit were necessary and therefore the reboiler specimens were removed after the second run. When the samples were installed they were shiny; when removed they showed considerable bronzing and film. To clean them, the samples were soaked in 60 percent nitric acid thirty minutes, rinsed, and then scrubbed with soap solution using a soft bristle hand-brush. After the specimens were degreased in acetone, rinsed in alcohol, and dried they were weighed to the nearest milligram. Some of the stains were retained and after the first weighing the specimens were scoured, using a cloth and slurry of "Bon Ami" until the stains were removed. A second weighing showed small weight losses mostly between one and three milligrams. The specimens were placed back in the reboiler and acid recovery operations continued.

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Measurements and Normal Chemical Exposure (continued)

Chemical exposure to the specimens from normal operation is shown in Table 8, which gives the total hours operation with standard feed concentrations of nitric acid and includes both Purex runs and synthetic runs. It does not include time during which concentrated nitric acid, hot citric acid, hydrofluoric acid, etc. were used for decontamination.

Samples of the recovered acid and the waste condensate from three runs were submitted for halide analysis. Chloride was reported as less than 10 ppm for the three samples of recovered acid, and 12, 13, and 15 ppm for the condensate. 10 ppm is the lower limit of analysis by the potentiometric method for chloride in the presence of strong nitric acid. The fluoride for the six samples of acid and condensate was reported as less than 5 ppm, the lower limit of analysis.

The acid recovery unit was dismantled to remove all corrosion specimens. After the specimens were decontaminated, they were degreased in acetone, rinsed in alcohol, dried, and weighed.

The specimens, reboiler, and tantalum heater were inspected by W. Myers and W. Lewellyn of duPont. No inspections have been made to date of the column interior, vapor line, or condenser. The specimens were sent to the ORNL Materials Laboratory where A. Olsen made the analysis presented in Section 9.5 of this report. His comments on the appearance of the reboiler, tantalum heater, and evaporator are also in the Appendix.

4.22 Radiation Exposure

The specimens were exposed to radiation during six Purex runs which were progressively more active, starting with 270 curies of beta activity

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Radiation Exposure (continued)

into the evaporator for the first run and 27,000 curies for the sixth. The number of curies and residence time in the evaporator for each run are given in Table 9.

The run normally started with the evaporator containing twenty gallons of 8N nitric acid. The 350 gallons of diluted IAW was added at a constant rate of about 7.5 gallons per hour and an equilibrium 8N evaporation conducted as described in Section 4.1.

As the run progressed, radioactivity of the evaporator solution gradually increased until the activity from the entire IAW of the run was contained in the twenty gallons of 8N evaporator solution. About one part per million of the activity escaped with the vapor to the column. The activity entering the column divided in the ratio of one part to the condensate and seven parts to the acid. The product acid flow was about one nineteenth the condensate flow so that the radioactivity of the acid (per milliliter) was about 120 times that of the condensate.

With respect to radiation then there were four regions in the equipment:

Evaporator

Stripping section of the column

Concentrating section of the column

Reboiler

On the basis of equations derived by T. H. J. Burnett (see Section 9.4), the total radiation in the various pieces of equipment in equivalent roentgens was estimated as follows:

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Radiation Exposure (continued)

Table 2

Total Radiation Exposure in Acid Recovery System

Unit	Total Beta Curie-Hours	Total Beta and Gamma Exposure in Equivalent Roentgens
Evaporator	5.6×10^6	10^8
Reboiler	1.6×10^3	10^5
Column Stripping Section	2	-
Column Concentrating Section	200	-

For each location the number of beta curies of radioactive substance was multiplied by the hours the particular amount was present and the total curie-hours obtained. This was done for the periods in which runs were in progress and also for intervening times when solution was cooled or stored. Calculations were based on radioactivity of solutions in these locations by radio analysis of samples. All radio analyses were for gross beta counts per minute per milliliter. 2.2×10^5 β counts per minute on a G. M. tube with second shelf geometry were taken to be equivalent to one microcurie. The volume of solution was measured for the evaporator and reboiler and estimated for the column locations. Since the activity level of the runs varied by a factor of one hundred, and the residual activity remaining in the equipment between runs was not known, the figures presented in Table 2 represent an order of magnitude estimate.

No radiation readings of the outside of the vessels were taken at the conclusion of the runs since cell entry was prohibited. After two weeks of operation with synthetic IAW, for decontamination purposes, the water filled evaporator read 70 R/hr. at three feet. The water filled reboiler read 65 R/hr. at one inch, due almost entirely to the activity which entered at the beginning

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Radiation Exposure (continued)

of the final run by accident. As this run progressed the activity of the product acid and column condensate decreased; this is shown in Figure 7.

4.23 Exposure to Decontaminating Reagents

During the first of the six hot runs, a blocked instrument control line resulted in the hot waste evaporator boiling excessively and driving some radioactivity through the vapor line into the column and reboiler. Following the run, this contamination was removed by chemical treatment. Details of the exposure to the decontamination reagents are given in Section 9.61.

Regular concentrations of nitric acid as normally used in the runs are not included as decontaminating reagents. Only reagents differing from the customary nitric acid solutions found in the equipment are listed. Operation with synthetic feed (regular acid concentration but no radioactivity) for purposes of decontaminating the equipment is counted in the list of operating hours (Table 8).

During startup of the sixth run, an increased evaporator liquid level caused a carryover of radioactivity by the entrainment which accompanied loss of freeboard. The activity was purged from the column and reboiler to a large extent during the balance of the run, but additional decontamination was required. Inadvertent boiling of the citric acid solution which was used caused further contamination of the column and reboiler. The decontaminating reagents to which the various sample groups were then exposed and the duration of exposure are included in Section 9.62.

The decontamination proceedings outlined above produced corrosion of the specimens beyond that due to normal operation. From past tests the members of

[redacted]

[redacted]

Exposure of Samples to Decontaminating Reagents (continued)

the ORNL Corrosion Group estimated a preliminary correction for decontamination on the three materials of primary interest to DuPont. This is reported in Section 9.5 together with corrected penetrations.

5.0 Results

5.1 Penetration Rates

The table below summarized the corrosion rate data for the ninety-eight specimens exposed. Detailed data are contained in Table 7.

Table 3

Summary of Corrosion Data
Penetration Rate (mils/year)

Material	Evaporator	Reboiler	Column*	
			Above Feed Point	Below Feed Point
ELC-304	3.2-3.9	8.8-25.0	0.5	0.8
ELC-304 CS	3.5	22.9	0.6	0.6
309-Cb	1.6-1.9	6.3-19.7	0-0.7	0.1-7.2
CS-309-Cb	1.6-3.9	26.7-33.3	-	-
ELC 316	3.6-4.2	10.6-14.9	-	-
329	1.7-1.8	6.2-6.4	0.3	0.7
347	4.2	19.5-21.5	-	-
Ta	0	0	-	-
Ti	0.1	59.6-73.3	-	-

* Entire column length contained 309-Cb specimens, other materials placed in the 23 percent and 50 percent nitric acid (liquid phase) regions only.

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Penetration Rates (continued)

The range of values in the evaporator and reboiler is due partly to the fact that some specimens had weldments while others did not.

It can be observed that the most severe conditions encountered were those in the reboiler (12N nitric acid) even though the radiation intensity in the evaporator (8N nitric acid) was higher by a factor of roughly a thousand. Since tantalum evidenced no measurable corrosion, a tantalum-lined reboiler may be preferred to a stainless steel vessel which is periodically replaced.

In the evaporator the stainless steels all showed penetration rates in the range of two to four mils per year. The choice of the type of stainless is therefore dependent upon cost, availability, and ease of fabrication.

Table 4 compares the corrosion rates for plain, welded, and sensitized Types ELC-304 and 309-Cb stainless steel flats.

Table 4

Effect of Welding and Sensitization on Penetration

Location	Penetration Rate (mils/year)					
	ELC 304			309-Cb		
	Plain	Welded	Sensitized	Plain	Welded	Sensitized
Evaporator Liquid	3.73	3.92	3.45	-	1.86	3.92
Evaporator Vapor	3.20	3.43	-	-	1.65	1.59
Evaporator Interface	3.63	-	-	1.80	-	2.37
Reboiler Liquid	15.1	10.6	22.9	-	10.2	31.9
Reboiler Vapor	25.0	8.78	-	-	9.58	26.7
Reboiler Interface	24.0	-	-	19.7	-	33.3

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Penetration Rates (continued)

In the evaporator no significant effect of welding or sensitization is evident. In the reboiler, sensitization appears roughly to have doubled the corrosion rate of both the ELC-304 and 309-Cb specimens. From the data available the corrosion rate of the welded ELC-304 was only half as great as for the unwelded billets. A comparison of the 309-Cb welded and unwelded specimens indicates the same effect, though the specimens were not at entirely comparable locations.

Figure 6 shows a plot of the corrosion rate of the 309-Cb column packing rings as a function of their position in the column. A peak in the curve is evident probably representing influence of chlorides at the 23 percent nitric acid liquid region. In general, the curve shows increasing penetration progressively down the column due to increased nitric acid concentration. The value of 7.2 mils/year quoted above in Table 3 represents the corrosion rate of the 309-Cb rings just above the reboiler and is therefore a maximum for the column.

At comparable positions in the lower section of the column, the resistance of 309-Cb ELC-304, ELC-304-CS, and 329 billets compared to the 309-Cb rings was:

Table 5

Comparative Corrosion of Materials in the Column

Billet		309-Cb
Type	Penetration (mils/year)	Rings Penetration (mils/year)
ELC 304	0.8	0.3
ELC 304-CS	0.7	0.4
329	0.7	1.0
309-Cb	0.6	0.6

[redacted]

[redacted]

Penetration Rates (continued)

The data show that of the steels listed in Table 5, Type 329 or 309-Cb stainless is best suited for corrosion resistance of the packing in the lower section of the column.

Penetration rate was calculated as weight loss sustained by the specimen while within the acid recovery unit (boiling acid, cold acid, decontamination solution, et al) divided by the time in which the specimen was exposed to regular Purex equilibrium temperatures and acid concentration (Purex runs plus synthetic runs). Sample calculations are shown in Section 9.3. Only operating time with boiling solutions, and not total calendar time was used. Since corrosion occurred also when unheated solutions were stored, the penetration results may be higher than would be anticipated for continuous operation. Including synthetic runs with Purex runs would normally provide less radiation exposure in the unit than operating on Purex runs exclusively; however, because of accidental contamination of the column and reboiler preceding the sixth run, radiation exposure of those specimens exceeded normal quantities and the combined operation on Purex and synthetic dummy runs produced as severe conditions as straight Purex operation. After the second run the reboiler specimens were weighed. The period for reporting penetration rates for these specimens begins with the third run.

5.2 Interface Corrosion

For reasons of: (1) variation in operating liquid levels and (2) boiling turbulence, the interface specimens were not semi-immersed in the liquid at all times during the runs.

Because of a temperature effect on the instrumentation used, the evaporator solution level in the first three runs was actually above the interface specimens. After this was discovered, allowance was made to permit operation at the desired level.

[redacted]

[redacted]

Interfacial Corrosion (continued)

The evaporator interface specimens were submerged during the first three runs for the following exposure time:

Hours of boiling 8N Nitric Acid 112°C	48
Hours of cool 8N Nitric Acid	72
Hours of Water	29
Hours Empty	104

The reboiler liquid level was not always at the same position. During column startup the reboiler overflow valve was closed to prevent reflux from purging the acid, and the liquid level rose about three inches. Boiling brought the liquid level back to the standard operating level, at which time the concentration was 57 percent nitric acid.

In the period for which reboiler specimen corrosion rates are reported, there were approximately twelve reboiler startups of thirty minutes each for a total of six hours immersion of the reboiler interface specimens in boiling 50 to 57 percent nitric acid. There was also an increase in the reboiler operating liquid level to one inch above the overflow, due to a vapor seal that was installed on the overflow line after the interface specimens had been placed. This put the interface at about the level of the specimen mounting holes.

The boiling interface in these two vessels was very turbulent and the vapor phase specimens undoubtedly received spatter although they were not immersed. The interface specimens were surrounded by a baffle and were precisely located at the room temperature interface level. Whether there was still a liquid-vapor demarcation line within the baffle on boiling could not be observed.

At the conclusion of the runs the interior surfaces of the reboiler and evaporator were inspected by A. R. Olsen. No apparent interface corrosion was detected.

[redacted]

[redacted]

5.3 Teflon Decomposition from Radiation

A consistent picture of the effect of Teflon decomposition upon the corrosion results was not obtained. This section includes the observations made and some possible explanations.

Teflon was used in the rods and bushings for stringers, spacers in the specimen mounting jigs, and as a gasketing material on the evaporator and reboiler. These pieces broke or crumbled during disassembly. Attack occurred chiefly where the Teflon was in the liquid phase; the Teflon in the vapor line and column was intact. Maximum specimen attack was observed at the areas in contact with Teflon, and the reboiler surface in contact with the gasket showed marked corrosion where seepage occurred.

Since Teflon ordinarily is stable in boiling nitric acid, radiation may have caused the decomposition observed in the acid recovery unit. The total radiation exposure was estimated in Section 4.22 at 10^8 R for the evaporator specimens and 10^5 R for the reboiler specimens. C. D. Watson has found that 5×10^6 R are necessary for incipient decomposition of Teflon (liberation of fluorine) and 10^8 R for its deterioration. These figures were for Teflon placed in dry vials at room temperature in a gamma field, while the acid recovery corrosion specimens were immersed in boiling acid solutions of mixed fission products. Watson estimated that 10^5 R could have been sufficient to cause fluorine evolution under the reboiler conditions.

The effect of radiation explains the fact that the evaporator solution (8 N nitric acid) caused greater Teflon deterioration than the reboiler solution (12N nitric acid). The radiation exposure in the evaporator was a thousand times that in the reboiler.

[redacted]

[redacted]

Teflon Decomposition from Radiation (continued)

On those specimens in the reboiler with highest penetration rates corrosion was mostly confined to the areas contacting Teflon, indicating that it was not direct radiation damage to the specimens that was responsible for the undue corrosion but rather the Teflon contact and resultant attack by evolved fluorine. However, in the evaporator where Teflon decomposition was greatest, specimen corrosion in the areas contacting Teflon was practically nil. The rate of fluorine corrosion therefore depended on acid concentration more than radiation. Since this corrosion was localized to the areas of Teflon contact it appears the fluorine was expended immediately by corroding the specimen areas and did not reach the exterior solution. It has been pointed out that fluoride ion unlike chloride ion will not recycle in corroding. Chemical analysis of the acid recovery unit solutions showed less than 5 ppm of fluoride, the lower limit of analysis.

Upon removal of the specimens from the equipment it was noticed the reboiler jigs had loosened and the evaporator jigs were tight. This would allow greater access of acid to the Teflon - to - specimen interfaces in the reboiler. Another explanation for the higher corrosion in the reboiler specimen Teflon areas was given by D. E. Waters of du Pont. There may have been galvanic action due to an oxygen deficiency in the areas where Teflon pressed against the specimens. Due to greater acidity this effect would have been greater in the reboiler than in the evaporator.

Small amounts of dissolved solvent from the IA extraction column passed into the acid recovery unit. This was not believed to have affected the Teflon deterioration. The air-purge type of instrumentation was used in the

[redacted]

[redacted]

Teflon Decomposition from Radiation (continued)

evaporator and reboiler; the oxygen is expected to have improved corrosion resistance of the vessels.

6.0 Program

The present corrosion specimens will be stored. A. R. Olsen will continue with further investigations of the ELC-304 stainless steel specimens. Following this, surface examinations will be conducted on all remaining specimens and sections will be made if indicated.

Olsen and Watson are preparing to test certain of the stainless steels when flanged with Teflon and the assembly exposed to acid recovery nitric acid concentrations under the influence of radiation. Currently, Watson is testing stainless steel specimens in the acid mixtures under the influence of radiation as well as testing nitric acid under the influence of radiation.

The data reported herein have been from processing IAW only. In the future IDW and IIAW are to be processed in addition to IAW, thereby introducing sulfate.

In view of the data available only four materials are of continued interest. Type 329 showed best resistance of the stainless steels. Type 309-Cb is comparable and has been used for the present evaporator. Type ELC-304 stainless steel is of general interest since it does not include critically short columbium. Since Type 347 stainless steel is used so extensively in the ORNL Pilot Plant it will be of continued interest in the corrosion studies planned. Further simplification of the corrosion program can be obtained by locating specimens at the interface positions only - not in all three phases as done to date.

[redacted]

[redacted]

Program (continued)

Teflon spacers and gaskets will be avoided and a means of mounting specimens utilized to eliminate galvanic effects. The specimens will be supported from inert materials by a method that will not appreciably restrict any specimen surface.

Integrating radiation dosimetric devices such as a radiation sensitive crystal, or cerium sulfate solution will be exposed with the specimens.

Specimens will be given a preliminary exposure in the unit, reweighed, and placed back. This procedure will ensure that results representative of long term operation, as well as the rapid initial corrosion, can be obtained.

The hours will be logged for the following exposures:

Boiling solutions of steady state acid concentration

Cool solutions of steady state acid concentration

Water

Empty

Decontamination treatments

7.0 Acknowledgements

The assistance of the following individuals is gratefully acknowledged: D. E. Waters for samples and suggestions, L. C. Peery for liaison, A. R. Olsen and J. L. English for specimen examination, C. D. Watson for Teflon radiation data, T. H. J. Burnett for radiation calculations and E. J. Weber for penetration calculations.

8.0 Bibliography

ORNL-1045 - Purex Pilot Plant Quarterly Report (February-April 1951)

ORNL-1115 - Purex Pilot Plant Quarterly Report (May-July 1951)

ORNL-1207 - Purex Pilot Plant Quarterly Report (August-October 1951)

In Prep. - Experience With Teflon in ORNL Purex Pilot Plant

[redacted]

NOT CLASSIFIED

9.0 Appendix

9.1 Specimen Consignment Memorandum (duPont)

To: Dewey Waters
Nemours Building

From: L. J. Clarke, Engineering Research Lab.

Date: December 20, 1951

Corrosion Test Specimens

Enclosed are the following corrosion test specimens as required for you by P. H. Permar, Explosives Department, Wilmington, Delaware.

6 - Type 309 CB stainless steel, welded, marked 309 CB

6 - Type ELC 304 stainless steel, welded, marked ELC 304

6 - Type 347 stainless steel, welded, marked, 347

4 - Titanium, heliarc welded, marked Ti.

NOT CLASSIFIED

[redacted]

9.2 Specimen Analyses (duPont)
(Letter from L. C. Evans to L. C. Peery)

SEPARATIONS - ACID RECOVERY CORROSION TESTS
Ref: Ltr. L. C. Peery to L. C. Evans, Same Title, September 27, 1951

The reference letter requested any analyses available for the corrosion test coupons being evaluated at ORNL.

The following analyses were supplied by the Engineering Research Laboratory in a letter Clarke to Permar, 25505 - Corrosion Test Coupons, October 5, 1951:

Metal	Heat	C	Ni	Cr	Mo	N_2	Mn	Si	P	S	Cb
ELC 304	FK1	0.021	10.88	17.84		0.033					
ELC 316	FH1	0.027	11.34	17.85	2.25	0.13					
329	GK7	0.07	5.48	27.15	1.49		0.36	0.48	0.013	0.013	
309-Cb	GA7	0.07	14.58	23.56			2.01	0.59	0.012	0.006	0.83

The 347 samples came from one of the following heats. The particular heat can be determined by analyzing for chromium or nickel:

Heat	C	Mn	S	P	Si	Cr	Ni	Cb
GF3	0.069	1.31	0.017	0.025	0.53	19.10	12.33	0.81
GF4	0.052	1.40	0.017	0.024	0.40	17.74	10.33	0.73

The samples were marked as follows:

Material	Marking: Annealed*	Marking: Sensitized**
ELC 304	ELC 304	ELC 304 CS
347	347	347 CS
309-Cb	309-Cb	309-Cb CS
ELC 316	ELC 316	ELC 316 CS
329	329	Not Sensitized

* Commercially annealed.
** Sensitized 1 hr. 1250°F., W.Q.

ATOMIC ENERGY DIVISION

/s/ L. C. Evans,
Technical Division

[redacted]

9.3 Sample Calculations

9.31 Surface Area of Specimens

Billets

L = Length
W = Width
T = Thickness

D_1 = Diameter of mounting hole

D_2 = Diameter of redundant mounting hole when present.

$$\text{Area} = 2\ [LW + T(L + W)] + \pi\left(D_1T - \frac{D_1^2}{2}\right) + \pi\left(D_2T - \frac{D_2^2}{2}\right)$$

ELC - 304 - 7:

$$\text{Area} = 2[5.1\text{x}2.5 - 0.25\ (5.1 - 2.5)] + \pi\left(0.7\text{x}0.25 - \frac{0.7^2}{2}\right) + \left(0.5\text{x}0.25 - \frac{0.5^2}{2}\right) = 29.1\ \text{cm}^2$$

Rings

D_o = Outside Diameter = 2.12 cm all rings

D_i = Inside Diameter = 1.60 cm

L = average length of ring

$$\text{Area} = 2\pi\left(\frac{D_o^2}{4} - \frac{D_i^2}{4}\right) + L\pi(Do - Di) = 3.01 + 11.7\ L$$

Column Ring No. 1:

$$\text{Area} = 3.01 + 11.7\text{x}2.35 = 30.5\ \text{cm}^2$$

9.32 Specific Volume of Specimens

$$\text{Specific Volume} = \frac{\text{wt in air - wt in water}}{\text{wt of 1 cc water x wt in air}}$$

wt of 1 cc water (weighing temperature) = 0.997 gm

$$\frac{1}{0.997} = 1.003$$

$$\text{Sp. vol.} = \frac{\text{wt in air - wt in water}}{\text{wt in air}} \text{ x } 1.003$$

Corrosion rates will be to three significant figures, hence, the $\pm \frac{0.003}{1.003}$ can be neglected

ELC - 304-2:

$$\frac{46.27 - 40.45}{46.3} = 0.126 \frac{cc}{gm}$$

ELC - 304-5

Before exposure $\frac{60.98 - 53.30}{61.0} = 0.126$

After first exposure $\frac{60.64 - 52.98}{60.6} = 0.126$

ELC - 304-6

Before $\frac{60.49 - 52.87}{60.5} = 0.126$

After $\frac{60.12 - 52.59}{60.1} = 0.125$

ELC - 304-9

Before $\frac{26.23 - 22.91}{26.2} = 0.127$

After $\frac{25.97 - 22.72}{26.0} = 0.125$

Average Sp. vol for ELC - 304 = $0.126 \frac{cc}{gm}$

9.33 Penetration Rate

Weight loss = initial weight - final weight

ELC-304 - 7

Wt. loss = 25.694 - 25.522 = 0.172 gm

$$\text{Penetration} = \frac{\text{wt. loss x sp. vol.}}{\text{surface area}}$$

ELC - 304-7:

$$\text{Penetration} = \frac{0.172 \times 0.126}{29.1} = 7.46 \times 10^{-4} \text{ cm}$$

$$\text{Penetration rate} = \frac{\text{Penetration x 1 year's time}}{\text{Exposure time}}$$

$$\text{Penetration rate} = \frac{7.46\text{x}10^{-4}\text{x}365\text{x}24}{687} = 0.00949 \text{ cm/year or } 3.73 \text{ mils per year}$$

[redacted]

9.4 Calculation of Radiation Exposure
(Portion of Memo of 10/19/51 to J. W. Ullmann from T. H. J. Burnett, ORNL Health Physics Division)

Introduction

It is desired to convert the corrosion data obtained in terms of "curie-hours" of beta and gamma to equivalent roentgens "exposure".

Working Data

The data available consisted of an approximate estimate of the gamma composition by energy as used by Hanford. Instead of basing the beta energy calculations on an approximate composite analysis of the constituent beta components, data on beta range were used.

An absorption series of beta counts was taken on a sample representative of activity of the solutions used for corrosion testing, and the values of counts and corresponding absorbers tabulated.

Beta Absorption Data and Extensions

Data Given		Derived Calculations			
Absorber thickness mg./cm^2,Al	Counts/ min. Sample	Equiv. Emax. Mev.	Average Emax. Mev.	Decrease Counts/ min.	Product Average E_m x c/m
0	11,288	0			
			0.009	685	6.2
1.79	10,603	0.019			
			0.022	364	8.0
2.72	10,239	0.026			
			0.028	104	2.9
3.28	10,135	0.030			
			0.033	408	13.5
4.33	9,727	0.037			
			0.041	306	12.5
5.88	9,421	0.046			
			0.057	904	51.5
10.32	8,517	0.069			
			0.080	599	47.9
15.01	7,918	0.091			
			0.103	603	62.2
20.58	7,315	0.115			
			0.134	587	78.6

[redacted]

[redacted]

Beta Absorption Data and Extensions (continued)

Data Given		Derived Calculations			
Absorber thickness mg./cm^2,Al	Counts/ min. Sample	Equiv. Emax. Mev.	Average Emax. Mev.	Decrease Counts/ min.	Product Average E_m x c/m
30.49	6,728	0.153			
			0.165	459	75.8
37.5	6,269	0.177			
			0.203	380	77.2
52.5	5,889	0.23			
			0.24	135	32.4
60.8	5,754	0.25			
			0.28	340	95.2
80.0	5,414	0.31			
			0.335	452	151.3
102.0	4,962	0.36			
			0.40	540	216.0
136.0	4,422	0.44			
			0.49	418	205.0
179.0	4,004	0.54			
			0.61	791	482.5
239.0	3,213	0.68			
			0.72	312	224.7
274.0	2,901	0.76			
			0.83	563	467.5
341.0	2,338	0.90			
			0.98	623	611.0
426.0	1,715	1.07			
			1.27	1,012	1,285.2
651.0	703	1.47			
			1.95	663	1,293.0
1,210.0	40	2.43			
			2.77	22	61.0
1,610.0	18	3.12			
(Pb. Sand.)	12	Total		11,270	5,561.1

Using the above extensions of the given absorber data the effective β energy (for immersion exposure) is obtained.

[redacted]

[redacted]

The total of the increments of counts/min. is $\Sigma\ \Delta c/m = 11{,}270$.

The total of the products of average maximum beta energy is $\Sigma\ \bar{E}_m \cdot \Delta c/m = 5{,}561$.

The effective beta energy of the mixture is then given:

$$E_{eff} = \frac{\Sigma \bar{E}_m \times \Delta\ c/m}{\Sigma\ \Delta\ cm} = \frac{5{,}561}{11{,}270} = 0.493 \text{ Mev.}$$

Calculations and Assumptions - Beta Exposure

The beta exposure is calculated on the assumption of equilibrium: the energy absorbed per cubic centimeter being equal to that emitted. This is valid since the range of a 5 Mev beta particle in aqueous solution is less than an inch. The size of the test specimen introduced is considered small enough not to affect this equilibrium significantly. The following equation can then be set up:

$$\text{(a)}\quad 1/2 \times \frac{Q \times 3.7 \times 10^4 \times 3.6 \times 10^3 \times E_{eff} \times 10^6}{5.93 \times 10^{13}} = \text{rep/hour}$$

(μc/cc) (dis/sec/μc) (sec/hr)(mev/dis)(ev/mev)

(ev/cc of tissue/rep)

(b) $1.12\ Q\ E_{eff}$ = rep/hr

based on the following assumptions and with these qualifications:

1. The factor of 1/2 was introduced because of thickness of sample tested and because of the incidence of β rays isotropic to sample surface.

2. The value of 95 ergs/gm tissue = 1 rep. is used.

3. No correction has been made for the difference in the relative mass stopping power of the sample compared to the solution, and the mass stopping power of the solution is assumed equivalent to that of tissue.

4. No correction has been made for the specific gravity of the solution.

5. No correction has been made for buildup of degraded radiation or for the difference in scattering in the material and in the solution.

[redacted]

[redacted]

6. No correction is added for the dosage in the medium due to the Brehmstrahlen from the β interaction at distant points of the solution, although its average energy is probably low enough for it to be in equilibrium.

The value of E_{eff} in equation (b) is evaluated by use of the absorption counting data in the previous section. The aluminum absorber thicknesses were converted to equivalent maximum beta energies and the counting increments used with an average maximum energy to compute the effective energy, which in this case came out 0.493 Mev.

Since the value for Q in microcuries/cc is computed

$$\text{(c)} \quad Q = \frac{\text{c/m/ml (at 10\% geometry) x 10}}{2.2\text{x}10^6 \text{ d/min/}\mu\text{c}} = \mu\text{c/cc}$$

the exposure can then be calculated directly from the reported activity analysis gross counting data using the value for E_{eff}

$$\text{(d)} \quad R_\beta = \frac{1.12 \; [\text{c/m/ml}] \, 0.493}{2.2\text{x}10^5} = 2.5 \text{ (c/m/ml) x } 10^{-6} \text{ rep/hour}$$

where (c/m/ml) is the counts reported at 10% geometry

Calculations and Assumptions - Gamma Exposure

The gamma exposure is calculated on the basis of a non-equilibrium condition with a correction term for the finite volume. For simplicity in calculation the test volume was assumed a sphere of radius, r, rather than the true conical shape. The sample was further considered as at the center rather than otherwise. A volume of 76 liters gives a radius of 26 cm, and for comparison the results using a volume of 11 liters with a radius of 14 cm are also shown.

The equation resulting is very similar:

$$\text{(e)} \quad \frac{\overset{(\mu\text{c/cc})(\text{dis/sec/}\mu\text{c})}{Q} \text{ x } \overset{(\text{sec/hr})}{3.7\text{x}10^4} \text{ x } \overset{(\text{mev/dis})}{3.6\text{x}10^3} \text{ x } \overset{(\text{ev/mev})}{E} \text{ x } \overset{(\text{eq. factor})}{10^6 \, (1-e^{-\mu r_1})}}{\underset{\text{(ev/cc of tissue/rep)}}{5.93 \text{ x } 10^{13}}} = \text{rep/hour}$$

$$\text{(f)} \quad 2.25 \; Q \; E \; (1-e^{-\mu E r_1}) = \text{rep/hour}$$

based on the following assumptions and with these qualifications:

7. Uses value of 95 ergs/gm tissue = 1 rep, since the γ exposure is in terms of energy absorption.

[redacted]

[redacted]

8. No correction has been made for the specific gravity of the solution, assumed 1 gm/cm^3 for attenuation calculations.

9. No correction has been made for the difference in the relative mass stopping power of the sample compared to the solution, and the mass stopping power of the solution is assumed equivalent to that of tissue.

10. Assume scattered and degraded radiation component in equilibrium because of its high attenuation although its source may not be. (This is improved if the photoelectric absorption predominates over the Compton effect).

Since the attenuation of the solution is a function of energy, the values for the gamma components given in the Hanford approximation are used separately:

E	%	μ_E	$26\mu_E$	$11\mu_E$
2.0 Mev	10	0.024 cm^{-1}	0.624	0.264
0.8 Mev	66	0.030 cm^{-1}	0.780	0.330
0.2 Mev	24	0.027 cm^{-1}	0.702	0.297

so that:

(g) $\sum_E 2.25\ Q\ E\ (1-e^{-\mu E r_1})$ rep/hr

$= 2.25 \times 0.10 \times 2.0\ (.465)$ for 26 cm: (.232), 11 cm.

$+ 2.25 \times 0.66 \times 0.8\ (.542)$ for " (.281) "

$+ 2.25 \times 0.24 \times 0.2\ (.505)$ for " (.257) "

(h) $= 0.209\ Q + 0.644\ Q + 0.055\ Q = 0.91\ Q$ rep/hr

and again, using the reported counts per minute data (at 10% geometry):

(i) $Q = \frac{[c/m/ml] \times 10}{2.2 \times 10^6\ d/m/\mu c}\ \mu c/cc$

$R_\gamma = \frac{0.91\ [c/m/ml]}{2.2 \times 10^5} = 4.1\ (c/m/ml) \times 10^{-6}$ rep/hour

where (c/m/ml) is at 10% geometry and the immersion volume is 76 liters. If the immersion volume is only 11 liters, the value in (h) is 0.47 Q so that the corresponding gamma exposure is only 2.1 (c/m/ml) x 10^{-6} rep/hr, or 51% of the value in the case of the larger solution.

[redacted]

[redacted]

Comments and Discussion

The values for "exposure" rate to the immersed samples are functions of the activity concentration and only in the case of gamma exposure does the volume affect the results (to the extent of equilibrium present).

If the corrosion processes are considered to be affected by the ionization from radioactivity, the reporting of exposure in terms of equivalent roentgens is a valid presentation of the value of a significant variable.

From the standpoint of effective estimates of immersion exposures for such corrosion studies, it is suggested that dosimeter of crystal or silver phosphate glass be used and the exposure evaluated photometrically by thermoluminescence or fluorescence upon ultraviolet irradiation. Details of these and other possible appropriate dosimetry methods can be found in Central Files Memorandum #50-10-184.

T. H. J. Burnett
Consultation and Special Problems

The comments of Dr. W. S. Snyder on the method used for beta E_{eff} calculations are gratefully acknowledged.

[redacted]

[redacted]

9.5 Comments on Equipment and Specimen Examination
(Memo of 10/18/51 to J. W. Landry from A. R. Olsen , ORNL Materials Laboratory)

The preliminary survey of the samples of stainless steel Types 309-SCb, 329, and 347 has been completed. The relatively high activity of the samples and the pressure of other work make it impossible to give a complete analysis of all samples at this time. The examinations will continue and a complete report of the extent and type of damage on all samples tested will be written as soon as possible.

Table 1 gives the data on three types of stainless steel examined, together with a description of the sample appearance. Figure 0 is a graphical representation of the corrected weight losses on all samples reported. It should be noted that the correction factor is limited only to those samples exposed to the mixed nitric acid-hydrofluoric acid decontamination solution and the exactness of this correction is subject to the same criticism as all control laboratory data when applied directly to field usage. A further complication in this case is the rather complex decontamination procedure involving solutions such as the nitric-periodic acid where the rate of metal attack is unknown. However, the corrected weight losses now fall in the expected order based on the apparent damage as shown by visual examination. Figures 1 through 5 are photographs of these samples which clearly show most of the observed surface phenomena. Figure 6 is a group of macro-photographs of areas where the nature and/or extent of attack was significant enough to warrant more careful examination.

Figures 7, 7A, 8 and 9 are micro-photographs of the samples shown in Figure 6. There are some scratches left on these samples because the hot polishing equipment at the Laboratory is not installed yet.

Figure 7 shows strong intergranular attack in the support area. This attack had proceeded in one spot to produce a pit approximately 0.010" in diameter and 0.040" deep. This support area is where the samples contacted the Teflon gaskets and spacers used for insultation against couple attack. Figure 7A shows the top edge of the sample where no Teflon was present. This area gives no indication of intergranular corrosion.

Figure 8 shows a very strong etching attack on sample 347-4 coupled with intergranular attack in the weld deposit. The base material was not subject to intergranular attack even in the severely corroded support area. However, the weld deposit was apparently not properly stabilized.

Figure 9 shows sample 309 CB-3 as not susceptible to intergranular attack in the unsensitized condition in reboiler solutions. There is, however, a distinct knife line corrosion at the weld edge, which may be due to either insufficient stabilization or more likely the formation of Sigma in this heat affected zone.

[redacted]

On October 1 the author had an opportunity to give the vessels in which the tests were conducted a visual inspection. As a matter of record, the observations on the boilers are included here.

Evaporator: Type 309 SCb Stainless Steel

All weldments and wall surfaces had the bright shiny appearance of normally pickled stainless steel. There was no indication of where the solution level may have been, nor was there any sign of concentrated corrosion in the area between the two heating jacket welds. As no heat treatment had been given the vessels after welding, it seems safe to assume that in the solutions encountered in these tests, Type 309 SCb stainless is not subject to stress corrosion cracking.

Reboiler: Type 347 Stainless Steel

The reboiler has a much more complicated interior and not all of the surface was visible through the access hole. However, the interior was in much better condition than any of the Type 347 samples which were exposed in it. The sides of the vessel had a strongly pickled appearance as did all weldments and piping, but all surfaces were bright and clean. There was no solution level indication except on the slosh suppressor at the overflow pipe. There was no concentration of attack in or at the edge of the heating jacket section. In addition to these observations on the Type 347 components, a fine wire screen of Type 316 stainless steel had been placed around the instrument thimbles and this material showed no signs of attack whatsoever. The tantalum heater was removed from the equipment and a careful examination showed that other than mechanical denting, all surfaces, including the seam and bottom weld, are apparently unchanged.

From the results of this survey of corrosion damage, it would appear:

(1) That the tests are of questionable value as a quantitative measure of the corrosion resistance of the various materials in actual operation.

This doubt is based on the unknowns involved in the decontamination procedure, the obvious poor quality of weld metal in the case of the 347 samples, and principally in the unknown effect of the radiation-decomposition products of the Teflon insulators. An attempt to evaluate the effects of the decontaminants and the insulators is being made now with the cooperation of Mr. C. D. Watson of the Chemical Technology Division.

(2) If one were to make a choice based on the three types of stainless steel so far evaluated, it would seem that Type 329 is far superior to the others under all conditions.

[redacted]

Corrosion Data on Types 309, 329, and 347 Stainless Steel Samples from the Evaporator and Reboiler Sections

Piece No.	Ident.	Area (cm^2)	Wt. Loss (gram) (29 days)	Correction* factor (grams)	Corrected Wt. Loss (grams)	MDD**	Penetration Rate (mpy)	Surface Appearance
4	309CB-1	42.9	0.125	-	-	9.98	1.81	Very thin film, clean spacer area, no weld attack, no pitting
5	CS309CB-1	28.9	0.109	-	-	13.00	2.36	Very thin film, clean spacer area, no pitting
7	329-1	33.6	0.094	-	-	9.67	1.75	Very thin blotched film, no pitting
8	347-1	27.7	0.0184	-	-	22.92	4.15	Lightbrown film, clean spacer area, no weld attack, no pitting
14	309CB-2	39.9	0.104	-	-	8.98	1.63	Very thin film, clean spacer area, minor pitting in heat affected zone
15	CS309CB-2	29.6	0.074	-	-	8.63	1.55	Very thin film, clean spacer area, no pitting
17	329-2	28.4	0.077	-	-	9.34	1.69	Thin brown film, spacer partially covered, no pitting
18	347-3	27.7	0.183	-	-	22.78	4.13	Heavy dark brown film, spacer area and part of back clean, no weld attack, no pitting
23	309CB-4	30.0	0.085	-	-	9.75	1.77	Very thin film, clean spacer area, no pitting
24	CS309CB-5	28.6	0.106	-	-	12.79	2.32	Very thin film, clean spacer area, no pitting
29	309CB-3	40.0	(20 days) 0.443	0.336	0.117	14.61	2.64	Thin gray-tan film, moderate etching in spacer area only
30	CS309CB-3	28.2	0.975	0.237	0.738		23.68	Heavy gray scale, moderate etching in spacer area only

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Piece No.	Ident.	Area (cm^2)	Wt. Loss (gram) (20 days)	Correction* factor (grams)	Corrected Wt. Loss (grams)	MDD**	Penetration Rate (mpy)	Surface Appearance
32	329-3	35.4	0.234	0.252	-	-	-	Gray brown film over all, no etching
33	347-4	28.0	0.657	0.176	0.481	85.86	15.52	Heavy granular brown scale, weld bead is pitted, possibly knife attack, spacer area very strongly etched
39	309CB-6	40.0	0.417	0.336	0.081	10.13	1.82	Thin gray-tan film, moderate etching in spacer area only
40	CS309CB-4	27.4	0.794	0.210	0.584	106.6	19.28	Heavy gray scale, moderate etching in spacer area only, soft rust colored blob on one corner
42	329-4	34.5	0.236	0.294	-	-	-	Gray-brown film over all, no etching
43	347-5	27.4	0.583	0.173	0.410	74.82	13.54	Heavy granular brown scale, weld bead is pitted, possibly knife line attack, spacer area very strongly etched
48	309CB-5	27.4	0.586	0.210	0.376	68.62	12.42	Heavy gray-brown film, very strong etching at spacer, no pitting
49	CS309CB-6	29.8	1.074	0.252	0.822	137.7	24.91	Heavy gray scale, moderate etching at spacer, two corners have thick dark brown scale patches

* Correction factor based on HNO_3-HF decontamination only because of known data on these solutions (8.4 mg/cm^2 for 309 and 6.3 mg/cm^2 for 347 and 4 mg/cm^2 estimated for 329 for six hours exposure)

** MDD - Milligrams per square decimeter per day

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Dwg. #13749

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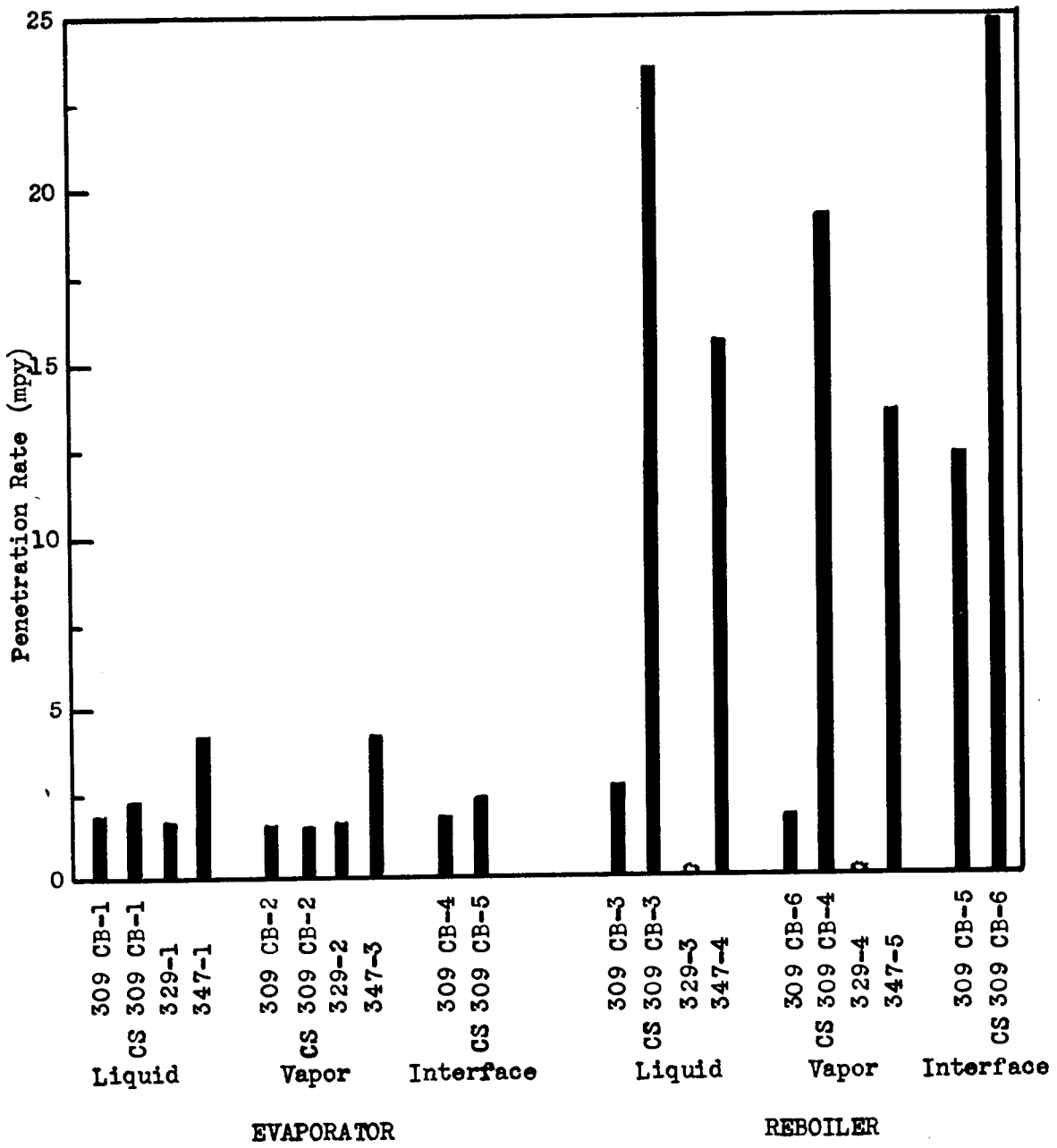
FIGURE O

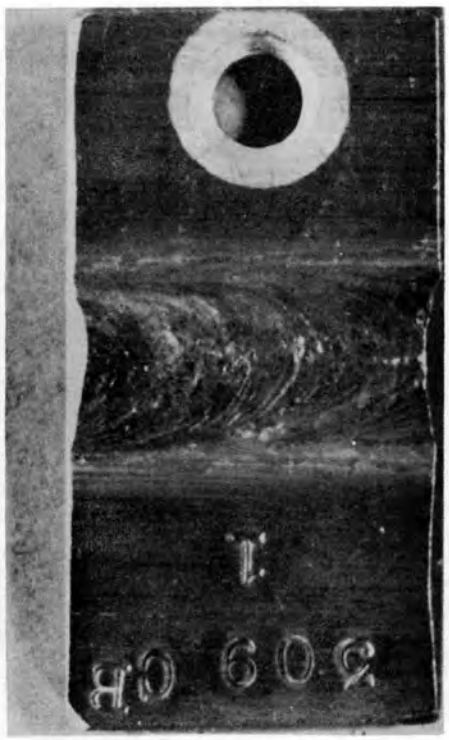
COMPARATIVE CORRECTED CORROSION RATES

of Stainless Steel Samples

in the Two Boilers



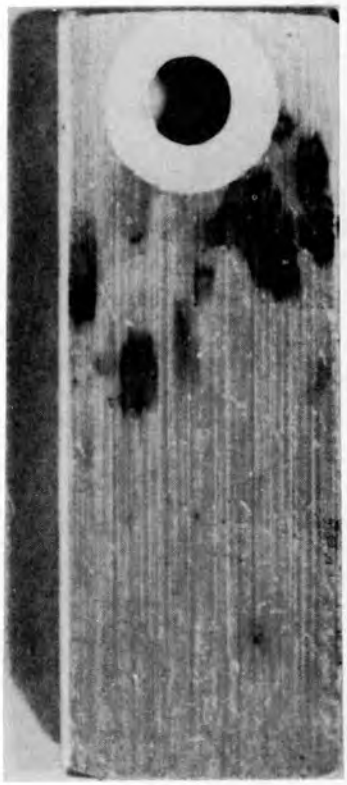
[redacted]



Sample No. 4

Sample No. 5

Sample No. 8



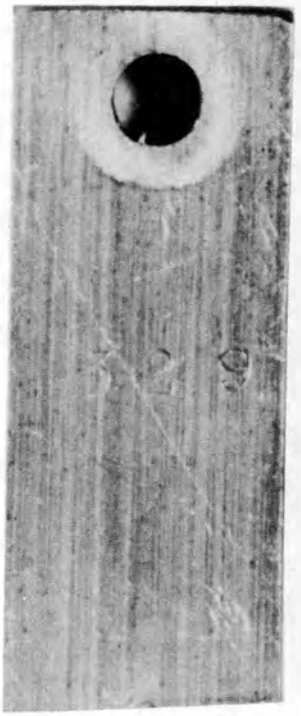
Sample No. 7

Fig. 1 TOTAL IMMERSION SAMPLES FROM EVAPORATOR

Sample No. 14

Sample No. 15

Sample No. 18



Sample No. 17

Fig. 2 SAMPLES EXPOSED TO THE VAPORS IN THE EVAPORATOR

Sample No. 23

EVAPORATOR

Sample No. 24

Sample No. 48

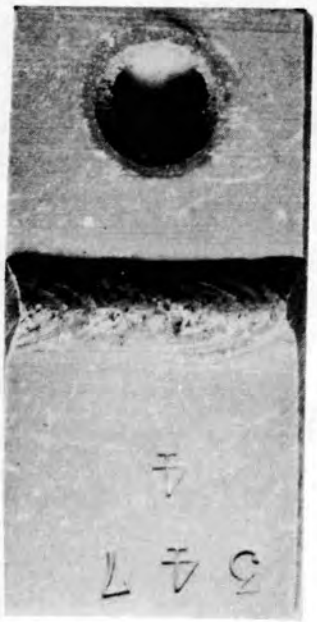
REBOILER

Sample No. 49

Fig. 3 SEMI-IMMERSION SAMPLES FROM BOTH VESSELS

Sample No. 29

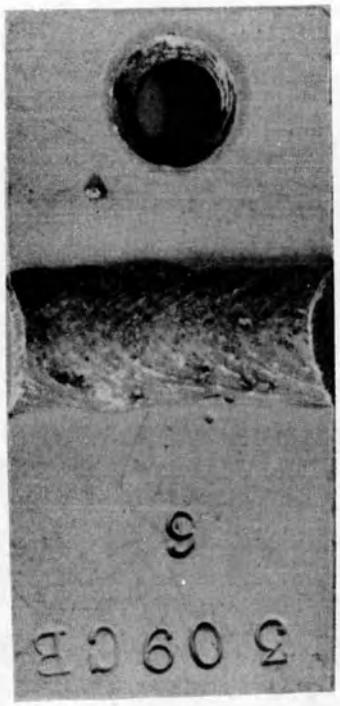
Sample No. 30



Sample No. 33

Sample No. 32

Fig. 4 TOTAL IMMERSION SAMPLES FROM THE REBOILER



Sample No. 39

Sample No. 40

Sample No. 42

Sample No. 43

Fig. 5 SAMPLES EXPOSED IN THE VAPOR REFLUX OF THE REBOILER

UNCLASSIFIED

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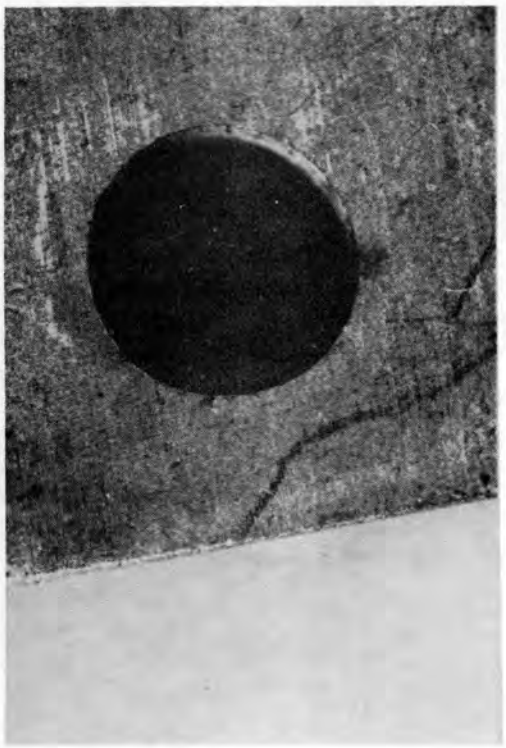
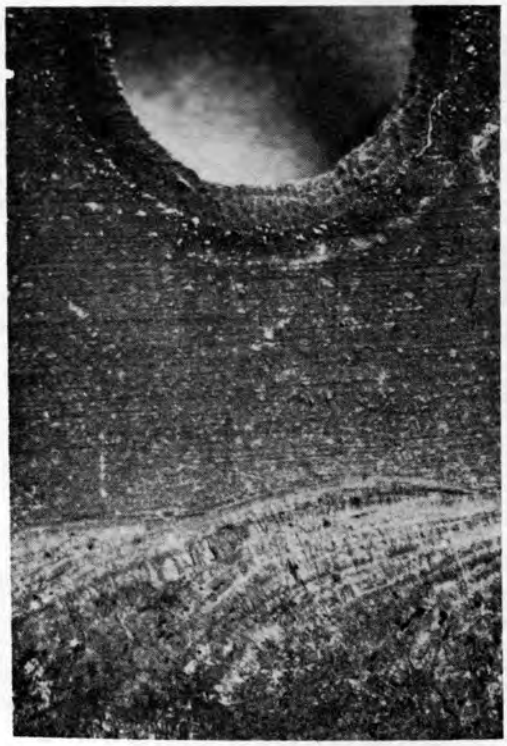
Weld Area Samples 309 CB-2 (10x)

Support Area Sample 309 CB-2 (10x)

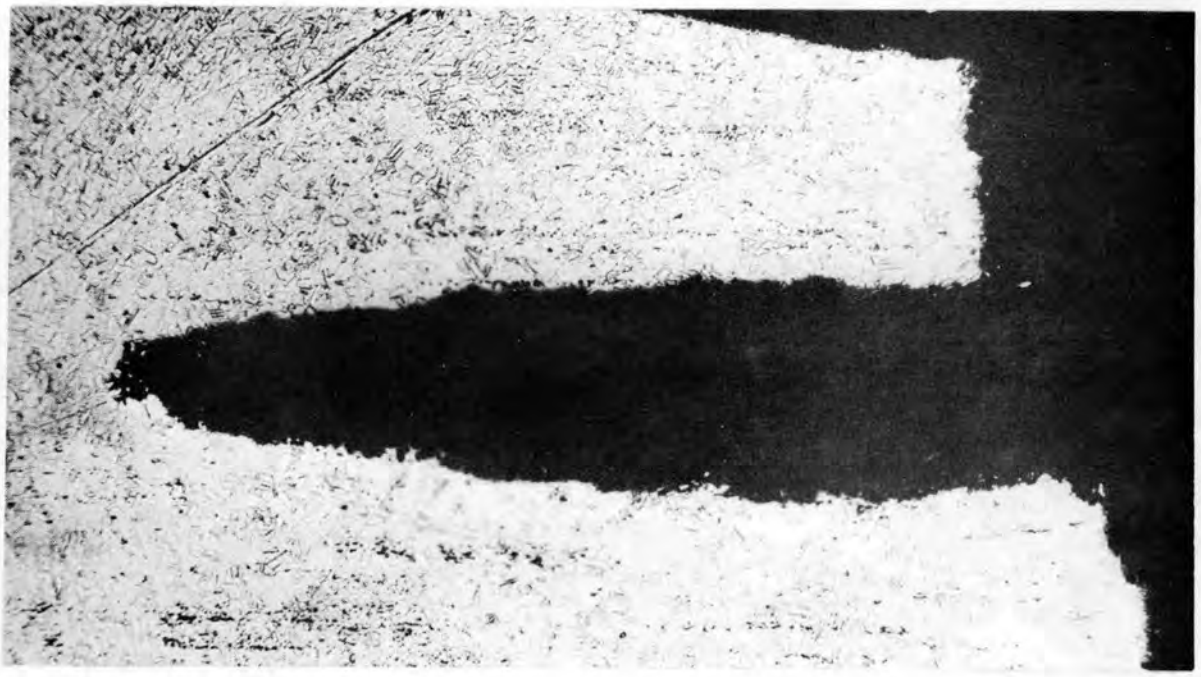


Support Area Sample 329-3 (10x)

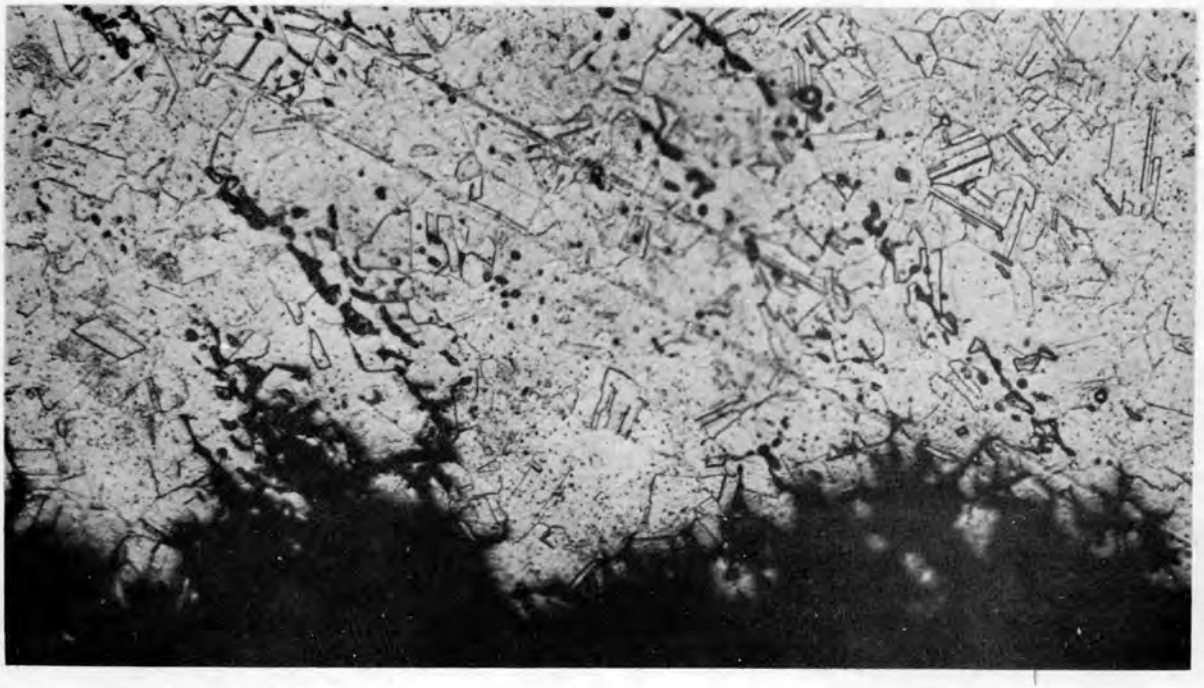


Support and Weld Areas Sample 347-4 (10x

Fig. 6 MACRO-PHOTOGRAPHS OF SAMPLE AREAS OF PRINCIPLE INTEREST



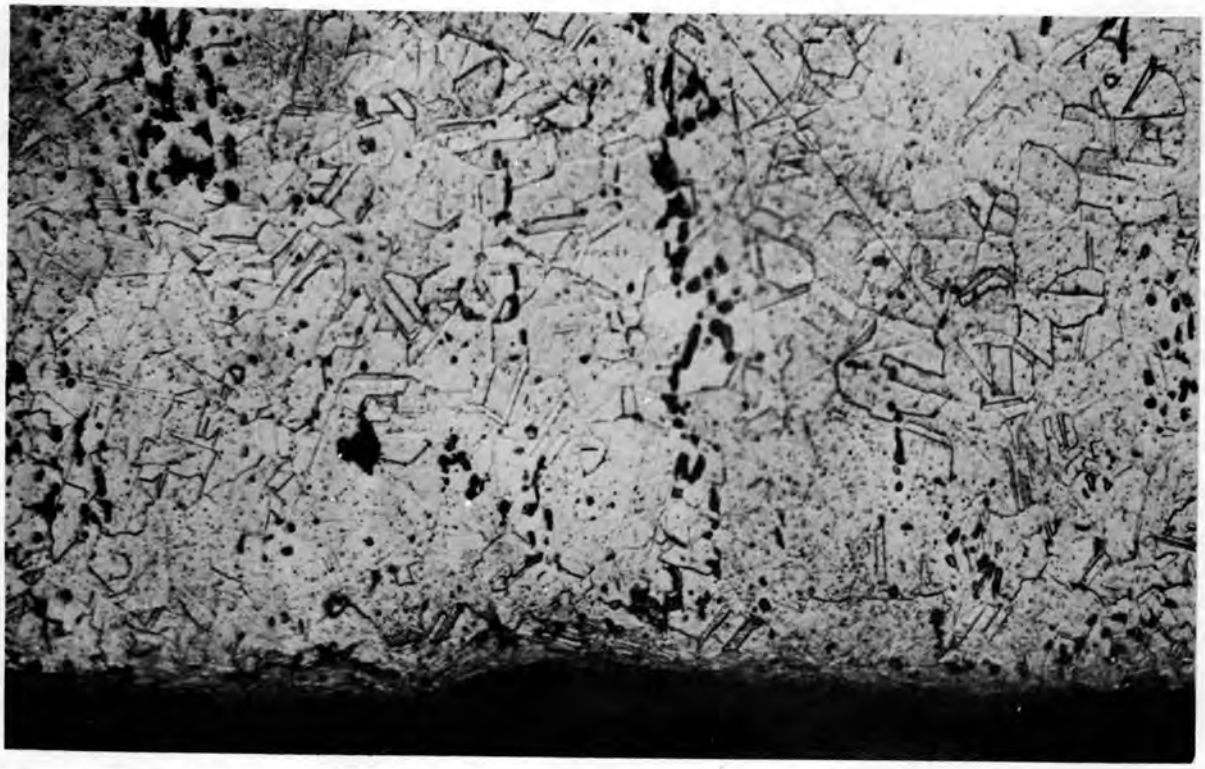
Support area showing pit formed. Magn 100x



Support area showing intergranular attack. Magn 500x

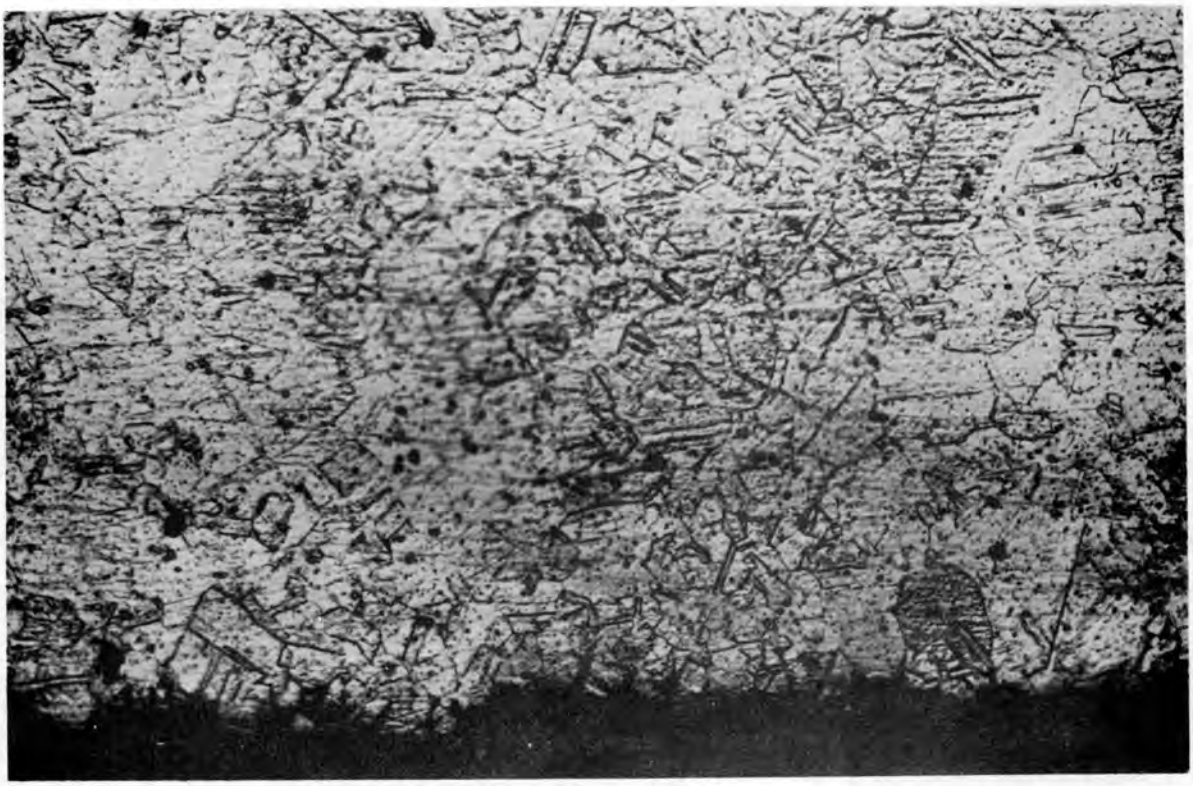
Fig. 7 SAMPLE 309-CB-2 EXPOSED TO THE VAPOR PHASE IN THE SOLUTION EVAPORATOR

UNCLASSIFIED

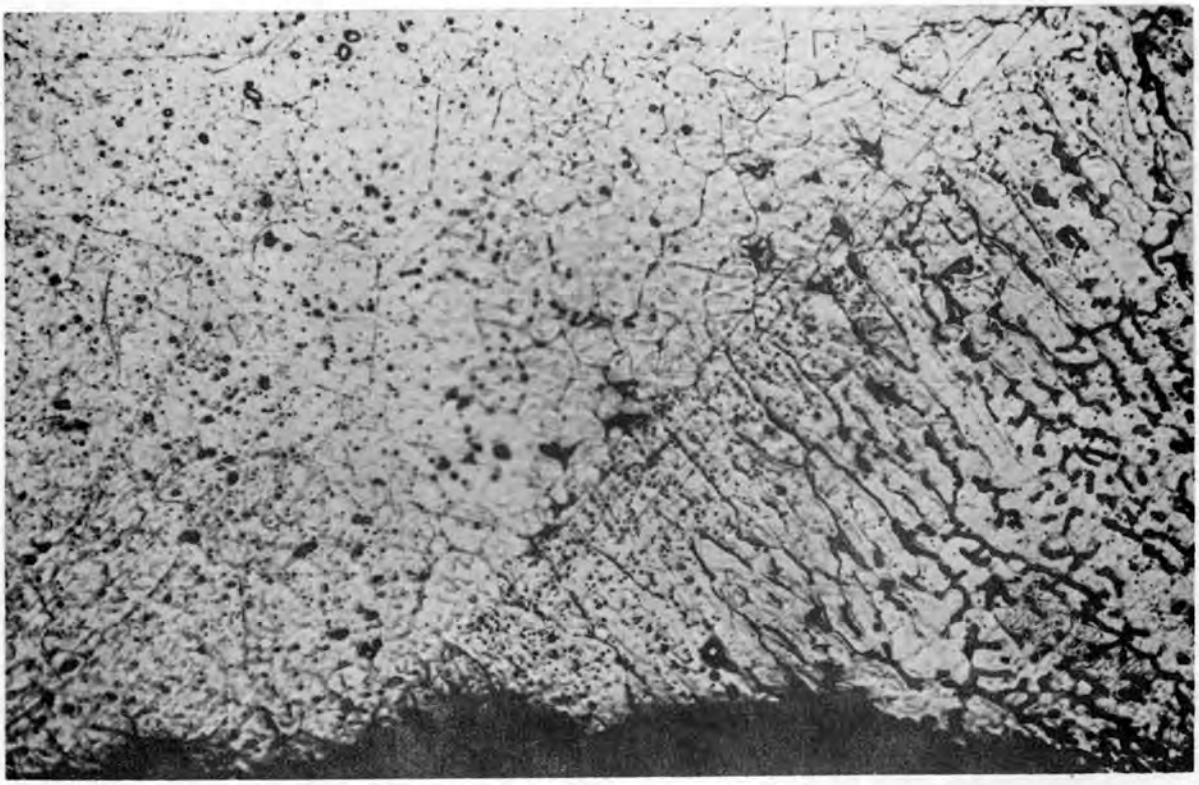


Top of sample showing mild overall corrosion attack. Magn 500x

Fig. 7A SAMPLE 309-CB-2 EXPOSED TO THE VAPOR PHASE OF THE SOLUTION EVAPORATOR



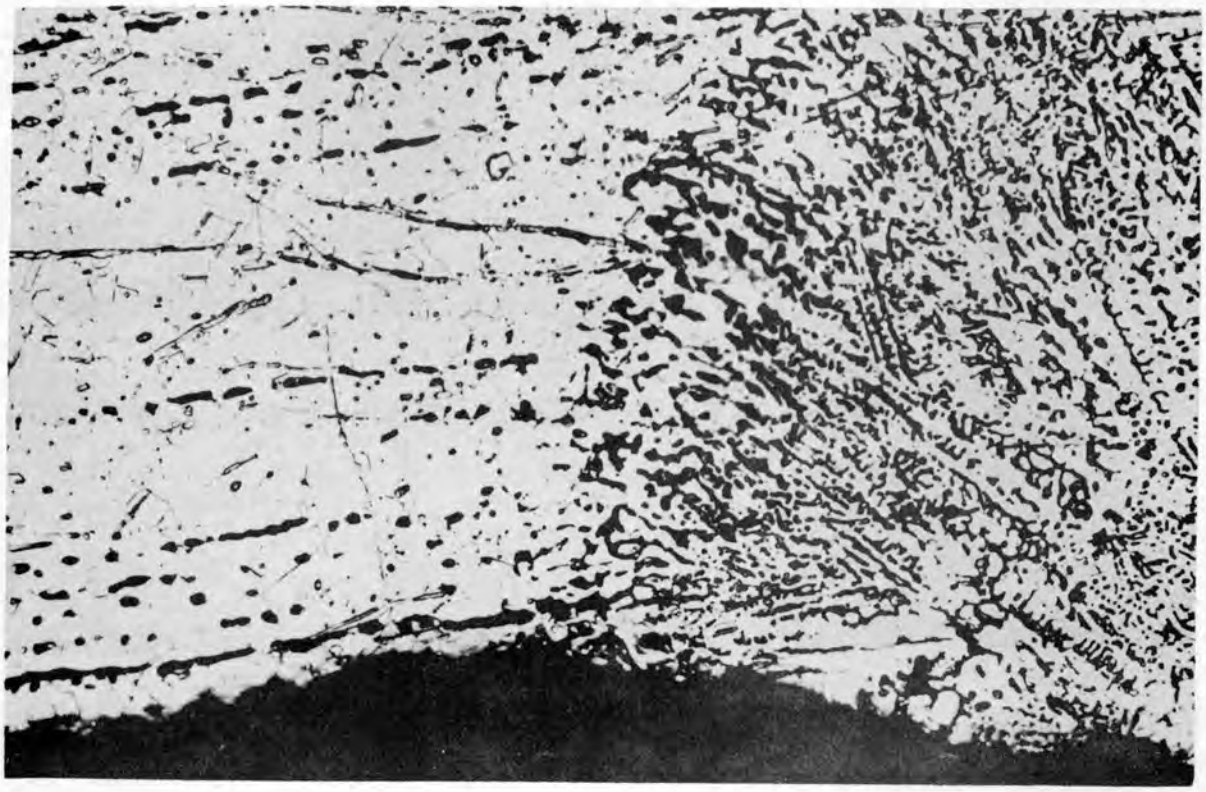
Top of sample showing typical strong overall corrosion attack. Magn 500x



Surface at weld line showing intergranular attack of weld and heat affected zone. Magn 500x

Fig. 8 SAMPLE 347-4 EXPOSED UNDER TOTAL IMMERSION IN THE REBOILER

Support area showing uniform corrosion attack. Magn 500x



Surface at weld line showing intergranular attack in weld metal and increased attack in heat affected zone. Magn 500x

Fig. 9 SAMPLE 309-CB-3 EXPOSED UNDER TOTAL IMMERSION IN THE REBOILER

9.6 Exposure of Specimens to Decontaminating Solutions

9.61 At the conclusion of the first hot run the specimens were subjected to the following solutions.

Evaporator

0.1 N nitric acid, 0.005 M periodic acid - room temperature, 1-1/2 hours
2% Versene, r.t., 1 hour
70% nitric acid, hot, 5 hours
1 M sodium carbonate, hot, 1 hour
2% citric acid, hot, 1/2 hour

Column

65-70% nitric acid refluxed from reboiler up into the column for one hour. Concentrations in upper regions of column probably were less than 65%.

Reboiler

0.1 nitric acid, 0.05 M periodic acid, r.t., 1 hour
70% nitric acid, hot, 3 hours
2% Versene, hot, 1/2 hour

9.62 At the conclusion of the sixth hot run the equipment and specimens were decontaminated with:

Evaporator

10% citric acid, hot, 4 hours; cold, 50 hours

Column

10% citric acid, hot, 1/2 hour; cold, 2 hours
10% nitric acid, 1/3% hydrofluoric acid, r.t., 1 hour
5% nitric acid, 1/3% hydrofluoric acid, r.t., 1 hour
2% Versene, r.t., 1 hour

Reboiler

10% citric acid, hot, 4 hours; cold, 48 hours
10% nitric acid, 1/3% hydrofluoric acid, r.t., 6 hours
2% Versene, r.t., 5 hours
5% nitric acid, 1/3% hydrofluoric acid, r.t., 1 hour until leaks occurred in asbestos gasket

After the above decontamination of equipment, the samples were removed and collectively treated with the following:

Soaked in 25% nitric acid, r.t., 4 hours
2% Versene, r.t., 4 hours
50% sodium hydroxide, r.t., 16 hours
10% citric acid, r.t., 16 hours
PAX soap solution, r.t., 16 hours
Scrubbed by bristle brush with PAX

Ring sample No. 19 was lost in the above treatments. The 97 remaining samples were rinsed with dilute nitric acid, then demineralized water, soaked in alcohol, dried with ether, and weighed.

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Table 6

CORROSION SPECIMENS AND LOCATIONS IN ORNL PILOT PLANT ACID RECOVERY UNIT

Location / Specimens By Type	ELC 304 Supplied as Shown each	ELC 304 Weldment duP	ELC 304 CS duP	309 CB duP	309 CB Weldment duP	CS 309 CB duP	ELC 316 duP	329 duP	347 Weldment duP	TA ORNL	TI Weldment duP	Column Packing Ring 309 CB
Evaporator Liquid	(1) ELC 304 7 (ORNL)	(2) ELC 304 1	(3) ELC 304 CS 1	None	(4) 309 CB 1	(5) CS 309 CB 1	(6) ELC 316 1	(7) 329 1	(8) 347 1	(9) 1 TA	(10) 1 TI	(11) (Ringlet) 1
Evaporator Vapor	(12) ELC 304 8 (ORNL)	(13) ELC 304 4	None	None	(14) 309 CB 2	(15) CS 309 CB 2	(16) ELC 316 2	(17) 329 3	(18) 347 3	(19) 2 TA	(20) 2 TI	(21) (Ringlet) 2
Reboiler Liquid	(26) ELC 304 9 (ORNL)	(27) ELC 304 5	(28) ELC 304 CS 2	None	(29) 309 CB 3	(30) CS 309 CB 3	(31) ELC 316 3	(32) 329 3	(33) 347 4	(34) 3 TA	(35) 3 TI	(36) (Ringlet) 3
Reboiler Vapor	(37) ELC 304 10 (ORNL)	(38) ELC 304 6	None	None	(39) 309 CB 6	(40) CS 309 CB 4	(41) ELC 316 4	(42) 329 4	(43) 347 5	(44) 4 TA	(45) 4 TI	(46) (Ringlet) 4
Evaporator Liquid-Vapor Interface	(22) ELC 304 11 (ORNL)	None	None	(23) 309 CB 4	None	(24) CS 309 CB 5	None	None	None	(25) 5 TA	None	None
Reboiler Liquid-Vapor Interface	(47) ELC 304 12 (ORNL)	None	None	(48) 309 CB 5	None	(49) CS 309 CB 6	None	None	None	(50) 6 TA	None	None
Approximate 23% HNO_3 Column	ELC 304 2 (duP)	None	ELC 304 CS 3	309 CB 7	None	None	None	329 5	None	None	None	11,12,13
Lower Section Column Approximate 50% HNO_3	ELC 304 3 (duP)	None	ELC 304 CS 4	309 CB 8	None	None	None	329 6	None	None	None	25,26,27
Remainder of Column	None	None	None	None	None	None	None	None	None	None	None	1-10 14-24 28-40
Sp vol. from weighings	0.126	0.126	0.126	0.127	0.127	0.127	0.126	0.129	0.126	0.0598	0.220	0.126

(1), (2), (3) etc., Piece No. 1, Piece No. 2 etc.

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[redacted]

TABLE 7
DETAILED CORROSION DATA
For
EVAPORATOR
687 Hours of Boiling 8N Nitric Acid, 112°C
716 Hours of 8N Nitric Acid at Temperatures Less Than Boiling
8.36×10^6 Curie-Hours of Radioactivity of Solution Contents

Piece(* Indicates Welded)	Phase In Contact	Specimen Label	Initial Wt. (gms.)	Final Wt. (gms.)	Wt. Loss (gms.)	Flats (cm.) Length	Flats (cm.) Width	Flats (cm.) Thickness	Flats (cm.) D_1	Flats (cm.) D_2	Rings (cm) Length	Surface Area (cm^2)	Sp. Vol. (cm^3/gm)	Penetration ($cm \times 10^{-4}$)	Penetration (mils/year)
1	Liquid	ELC-304-7	25.694	25.522	0.172	5.1	2.5	0.25	0.7	0.5		29.1	0.126	7.46	3.73
*2		ELC-304-1	54.642	54.427	0.215	5.1	2.6	0.5	0.8	0		34.5	0.126	7.85	3.92
3		ELC-304-CS-1	46.667	46.513	0.154	4.5	2.1	0.65	0.65	0		28.1	0.126	6.90	3.45
*4		309-CB-1	78.055	77.930	0.125	5.3	2.85	0.73	0.8	0		42.9	0.127	3.71	1.86
5		CS-309-CB-1	47.258	47.149	0.109	4.6	2.15	0.63	0.65	0		28.9	0.127	7.85	3.92
6		ELC-316-1	27.444	27.284	0.160	4.5	2.1	0.4	0.65	0		24.3	0.126	8.31	4.16
7		329-1	58.753	56.659	0.094	5.65	2.1	0.6	0.65	0		33.6	0.129	3.60	1.80
*8		347-1	29.010	28.826	0.184	4.9	2.4	0.3	0.75	0		27.7	0.126	8.37	4.18
9		TA-1	5.607	5.607	NWL	4.1	1.7	0.05	0.7	0		13.9	0.0598	0	0
*10		TI-1	12.340	12.339	0.001	5.45	2.5	0.2	0.7	0		30.1	0.220	0.08	0.04
11		Ring 1	7.882	7.848	0.034						0.62	10.3	0.126	4.16	2.08
12	Vapor	ELC-304-8	25.992	25.847	0.145	5.0	2.5	0.25	0.7	0.5		28.5	0.126	6.42	3.20
*13		ELC-304-4	60.542	60.345	0.197	5.45	2.55	0.5	0.8	0		36.1	0.126	6.87	3.43
*14		309-CB-2	78.573	78.469	0.104	5.3	2.8	0.6	0.8	0		39.9	0.127	3.31	1.65
15		CS-309-CB-2	49.558	49.484	0.074	4.8	2.15	0.6	0.65	0		29.6	0.127	3.18	1.59
16		ELC-316-2	27.934	27.796	0.138	4.5	2.1	0.4	0.65	0		24.3	0.126	7.16	3.57
17		329-2	58.842	58.765	0.077	5.8	2.15	0.68	0.65	0		28.4	0.129	3.50	1.75
*18		347-3	29.846	29.663	0.183	4.8	2.45	0.3	0.75	0		27.7	0.126	8.34	4.16
19		TA-2	5.869	5.869	NWL	4.1	1.75	0.05	0.7	0		14.3	0.0598	0	0
*20		TI-2	12.103	12.101	0.002	5.45	2.5	0.2	0.7	0		30.1	0.220	0.15	0.08
21		Ring 2	8.003	7.974	0.029						0.62	10.3	0.126	3.54	1.77
22	Liq. Vap. Interface	ELC-304-11	25.678	25.512	0.166	5.05	2.5	0.25	0.8	0.5		28.7	0.126	7.28	3.63
23		309-CB-4	49.486	49.401	0.085	4.8	2.15	0.63	0.65	0		30.0	0.127	3.60	1.80
24		CS-309-CB-5	46.886	46.780	0.106	4.5	2.15	0.64	0.65	0		28.6	0.127	4.72	2.37
25		TA-5	5.710	5.711	NWL	4.1	1.7	0.05	0.7	0		13.9	0.06	0	0

[redacted]

[redacted]

TABLE 7 (continued)
REBOILER
475 Hours of Boiling 57% Nitric Acid, 121°C
301 Hours of 57% HNO_3 at Temperatures Less Than Boiling
2.43×10^3 Curie-Hours Radioactivity of Solution Contents

Piece (*Indicates Welded)	Phase in Contact	Specimen Label	Initial Wt. (gms.)	Final Wt. (gms.)	Wt. Loss (gms.)	Flats (cm.) Length	Flats (cm.) Width	Flats (cm.) Thickness	Flats (cm.) D_1	Flats (cm.) D_2	Rings (cm) Length	Surface Area (cm^2)	Sp. Vol. (cm^3/gm.)	Penetration ($cm \times 10^{-4}$)	Penetration (mils/year)
26	Liquid	ELC-304-9	25.969	25.478	0.491	5.1	2.5	0.28	0.7	0.5		29.7	0.126	20.8	15.1
*27		ELC-304-5	60.636	60.177	0.459	5.5	2.6	0.63	0.75	0		39.4	0.126	14.6	10.6
28		ELC-304-CS-2	45.765	45.058	0.707	4.45	2.15	0.65	0.7	0		28.3	0.126	31.5	22.9
*29		309-CB-3	71.234	70.791	0.443	5.4	2.6	0.7	0.8	0		40.0	0.127	14.1	10.2
30		CS-309-CB-3	47.074	46.099	0.975	4.5	2.15	0.65	0.7	0		28.2	0.127	43.9	31.9
31		ELC-316-3	28.686	28.278	0.408	4.64	2.1	0.4	0.65	0		25.1	0.126	20.5	14.9
32		329-3	60.839	60.605	0.234	5.7	2.15	0.65	0.65	0		35.4	0.129	8.53	6.19
*33		347-4	29.832	29.175	0.657	4.85	2.45	0.3	0.8	0		28.0	0.126	29.6	21.5
34		TA-3	6.021	6.020	0.001	4.1	1.8	0.03	0.7	0		14.4	0.0598	0.042	0.030
*35		TI-3	12.269	10.902	1.367	5.4	2.5	0.2	0.75	0		29.8	0.220	101.0	73.3
36		Ring 3	7.831	7.739	0.092						0.62	10.3	0.126	11.3	8.20
37	Vapor	ELC-304-10	24.653	23.869	0.784	5.1	2.45	0.25	0.7	0.5		28.6	0.126	34.5	25.0
*38		ELC-304-6	60.122	59.745	0.377	5.5	2.6	0.62	0.8	0		39.2	0.126	12.1	8.78
*39		309-CB-6	71.907	71.490	0.417	5.4	2.6	0.7	0.8	0		40.0	0.127	13.2	9.58
40		CS-309-CB-4	45.392	44.598	0.794	4.5	2.1	0.63	0.7	0		27.4	0.127	36.8	26.7
41		ELC-316-4	26.446	26.172	0.274	4.4	2.1	0.38	0.65	0		23.6	0.126	14.6	10.6
42		329-4	57.751	57.515	0.236	5.65	2.1	0.65	0.65	0		34.5	0.129	8.81	6.40
*43		347-5	29.214	28.631	0.583	4.85	2.4	0.3	0.8	0		27.4	0.126	26.8	19.5
44		TA-4	5.738	5.737	0.001	4.1	1.8	0.03	0.7	0		14.4	0.0598	0.042	0.030
*45		TI-4	11.826	10.714	1.112	5.4	2.5	0.2	0.75	0		29.8	0.220	82.1	59.6
46		Ring 4	7.852	7.781	0.071						0.63	10.3	0.126	8.69	6.31
47	Liq. Vap. Interface	ELC-304-12	25.424	24.658	0.766	5.1	2.5	0.26	0.7	0.5		29.2	0.126	33.0	24.0
48		309-CB-5	45.111	44.525	0.586	4.55	2.05	0.62	0.65	0		27.4	0.127	27.2	19.7
49		CS-309-CB-6	49.638	48.564	1.074	4.7	2.2	0.65	0.7	0		29.8	0.127	45.8	33.3
50		TA-6	5.568	5.568	NWL	4.15	1.65	0.03	0.7	0		13.3	0.0598	0	0

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TABLE 7 (continued)
COLUMN
739 Hours of Boiling Liquid-Vapor Equilibrium Mixtures
Concentration Gradient from 0.03% HNO_3 at Ring No. 1 to 57% HNO_3
at Ring No. 40; Corresponding Temperatures are 100°C and 121°C
Radioactivity of Fluid Contents:
3×10^2 Curie-Hours Below Feed Point 2.3 Curie Hours Above Feed Point

Piece	Inches From Top "G"	Specimen Label	Initial Wt. (gms.)	Final Wt. (gms.)	Wt. Loss (gms.)	Flats (cm.) Length	Flats (cm.) Width	Flats (cm.) Thickness	Flats (cm.) D_1	Flats (cm.) D_2	Rings (cm) Length	Surface Area (cm^2)	Sp. Vol. (cm^3/gm)	Penetration ($cm \times 10^{-4}$)	Penetration (mils/year)
51	0.0	1	29.561	29.588	NWL						2.35	30.5	0.126	0	0
52	4.0	2	27.069	27.067	0.002						2.15	28.2	0.126	0.089	0.041
53	8.5	3	26.288	26.287	0.001						2.1	27.6	0.126	0.046	0.021
54	11.0	4	27.791	27.789	0.002						2.15	28.2	0.126	0.089	0.041
55	14.0	5	28.135	28.183	NWL						2.2	28.7	0.126	0	0
56	16.5	6	27.058	27.057	0.001						2.15	28.2	0.126	0.045	0.021
57	20.0	7	26.746	26.743	0.003						2.1	27.6	0.126	0.137	0.064
58	23.0	8	29.152	29.148	0.004						2.25	29.3	0.126	0.172	0.080
59	25.5	9	28.739	28.733	0.006						2.3	29.9	0.126	0.253	0.118
60	28.5	10	27.480	27.475	0.005						2.2	28.7	0.126	0.220	0.103
61	30.0	ELC-304-2	46.269	46.247	0.022	4.4	2.1	0.65	0.68	0		27.7	0.126	1.000	0.466
62	32.0	11	28.236	28.227	0.009						2.2	28.7	0.126	0.395	0.184
63	33.5	ELC-304-CS-3	48.831	48.802	0.029	4.45	2.2	0.65	0.68	0		28.9	0.126	1.26	0.587
64	35.5	12	28.587	28.578	0.009						2.25	29.3	0.126	0.387	0.180
65	37.0	309-CB-7	46.757	46.742	0.014	4.45	2.1	0.65	0.68	0		27.9	0.127	0.637	0.297
66	38.5	13	26.782	26.774	0.008						2.1	27.6	0.126	0.365	0.170
67	40.0	329-5	60.138	60.121	0.019	5.7	2.1	0.65	0.68	0		34.7	0.129	0.706	0.329
68	42.0	14	27.419	27.405	0.014						2.15	28.2	0.126	0.626	0.292
69	45.0	15	27.762	27.738	0.024						2.2	28.7	0.126	1.052	0.490
70	48.5	16	28.560	28.536	0.024						2.25	29.3	0.126	1.031	0.480
71	51.5	17	29.420	29.382	0.038						2.3	29.9	0.126	1.602	0.747
72	57.0	18	28.571	28.560	0.011						2.25	29.3	0.126	0.473	0.230
73	63.0	19	27.193								2.1	27.6	0.126	Lost	
74	69.0	20	27.840	27.833	0.007						2.2	28.7	0.126	0.307	0.143

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TABLE 7 (continued)
COLUMN
739 Hours of Boiling Liquid-Vapor Equilibrium Mixtures
Concentration Gradient from 0.03% HNO_3 at Ring No. 1 to 57% HNO_3
at Ring No. 40; Corresponding Temperatures are 100°C and 121°C
Radioactivity of Fluid Contents:
3×10^2 Curie-Hours Below Feed Point 2.3 Curie Hours Above Feed Point

Piece	Inches From Top "G"	Specimen Label	Initial Wt. (gms.)	Final Wt. (gms.)	Wt. Loss (gms.)	Flats (cm.) Length	Flats (cm.) Width	Flats (cm.) Thickness	Flats (cm.) D_1	Flats (cm.) D_2	Rings (cm) Length	Surface Area (cm^2)	Sp. Vol. (cm^3/gm)	Penetration (cmx10^{-4})	Penetration (mils/year)
75	75.0	21	26.875	26.867	0.008						2.2	28.7	0.126	0.351	0.163
76	81.5	22	27.642	27.634	0.008						2.15	28.2	0.126	0.357	0.166
77	88.0	23	27.526	27.516	0.010						2.15	28.2	0.126	0.447	0.208
78	93.5	24	27.372	27.360	0.012						2.15	28.2	0.126	0.536	0.250
79	95.5	ELC-304-3	46.513	46.478	0.035	4.35	2.15	0.65	0.68			27.2	0.126	1.621	0.755
80	97.0	25	26.816	26.802	0.014						2.1	27.6	0.126	0.639	0.298
81		ELC-304-CS-4	45.375	45.344	0.031	4.45	2.1	0.65	0.68			27.9	0.126	1.401	0.653
82	100.0	26	28.316	28.294	0.022						2.3	29.9	0.126	0.926	0.431
83	102.0	309-CB-8	46.720	46.690	0.030	4.55	2.1	0.65	0.68	0		28.4	0.127	1.342	0.625
84	103.5	27	29.516	29.487	0.029						2.3	29.9	0.126	1.221	0.569
85	104.0	329-6	58.600	58.560	0.040	5.7	2.1	0.65	0.68	0		34.7	0.129	1.487	0.693
86	106.5	28	28.129	28.062	0.067						2.15	28.2	0.126	2.99	1.39
87	111.5	29	29.022	28.990	0.032						2.25	29.3	0.126	1.375	0.641
88	117.5	30		27.535							2.25	29.3	0.126	Bad Weighing	
89	124.0	31	27.368	27.311	0.057						2.15	28.2	0.126	2.55	1.19
90	130.0	32	27.056	26.981	0.075						2.1	27.6	0.126	3.42	1.59
91	136.0	33	26.029	25.920	0.109						2.1	27.6	0.126	4.97	2.32
92	142.0	34	29.230	29.053	0.177						2.25	29.3	0.126	7.61	3.54
93	148.0	35	28.416	28.208	0.208						2.2	28.7	0.126	9.13	4.25
94	153.5	36	27.421	27.185	0.236						2.15	28.2	0.126	10.5	4.89
95	158.5	37	28.221	27.947	0.274						2.15	28.2	0.126	12.2	5.69
96	162.0	38	26.467	26.159	0.308						2.1	27.6	0.126	14.1	6.57
97	165.0	39	27.609	27.268	0.341						2.2	28.7	0.126	15.0	6.99
98	168.0	40	28.744	28.378	0.366						2.3	29.9	0.126	15.4	7.18

Outside diameter of the packing rings was 2.12 cm, wall thickness was 0.26 cm

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Table 8

Chemical Exposure of Corrosion Specimens in Acid Recovery Unit

Location		
Evaporator	April 7 to August 7	
Hours of boiling 8N Nitric Acid 112°C		687
Hours of cool 8N Nitric Acid		716
Hours of Water		558
Hours Empty		440
Column	March 22 to August 7	
Hours of boiling equilibrium mixtures of Nitric Acid 100-121°C		739
Hours of water		24
Hours empty		3250
Reboiler	March 22 to May 10	
Hours of boiling 57% Nitric Acid 121°C		
Purex runs	110	
Synthetic runs	154	
		264
Hours of cool 57% Nitric Acid		159
Hours of water		42
Hours empty		687
	May 10 to August 7	
Hours of boiling 57% Nitric Acid 121°C		475
Hours of cool 57% Nitric Acid		301
Hours of water		581
Hours empty		381

The above represents the total hours operation in which regular feed concentrations of nitric acid were used. It includes both hot runs and synthetic runs (with respect to radioactivity). It does not include time during which concentrated nitric acid, hot citric acid, hydrofluoric acid, etc. were used for decontamination. A separate list of this treatment is supplied in Section 9.6.

[redacted]

Table 9 (continued)
CORROSION DATA FOR ACID RECOVERY UNIT
ORNL PILOT PLANT
EXPOSURE OF SAMPLES IN RADIOACTIVE SOLUTION

FOR STRIPPING SECTION OF THE COLUMN, March 22 to August 7				
Period	Length of Period, Hours	Maximum Concentration $\mu c/ml$ (β)	Average Concentration $\mu c/ml$ (β)	Curie Hours (β)
1HP-3	23		$1x10^{-4}$	$2.3x10^{-6}$
1HP-5	87		$1x10^{-4}$	$8.7x10^{-6}$
1HP-6	55		$3x10^{-4}$	$1.8x10^{-5}$
1HP-7	64		$2x10^{-4}$	$1.3x10^{-5}$
1HP-8	67		$1.2x10^{-3}$	$8x10^{-5}$
1HP-9	87		$2.3x10^{1}$	1.5
Total	383		Total β	1.5
			γ (50% of β)	.8
			Total Radioactivity (Curie Hours)	2.3
FOR REBOILER, June 10 to August 7				
1HP-6	49		$2x10^{-2}$	$1.1x10^{-2}$
Post 1HP-6	73		$2x10^{-2}$	$1.6x10^{-2}$
1HP-7	45		$3x10^{-2}$	$1.49x10^{-2}$
Post 1HP-7	77		$3x10^{-2}$	$2.54x10^{-2}$
1HP-8	50		$1.2x10^{-1}$	$6.6\ x10^{-2}$
Post 1HP-8	24		$1.2x10^{-1}$	$3.2\ x10^{-2}$
1HP-9	63	$4.5x10^{6}$	$2.3x10^{3}$	$1.59x10^{3}$
Post 1HP-9	101	$3.2x10^{1}$	$1.82x10^{1}$	$2.02x10^{1}$
			Total β	$1.61x10^{3}$
			γ (50% of γ)	$.82x10^{3}$
			Total Radioactivity (Curie-hours)	$2.43x10^{3}$

[redacted]

Table 9
CORROSION DATA FOR ACID RECOVERY UNIT
ORNL PILOT PLANT
EXPOSURE OF SAMPLES IN RADIOACTIVE SOLUTION

IN EVAPORATOR, April 7 to August 7					
Period	Length of Period, Hours	Total Curies of Activity (β)	Maximum Concentration μc/ml (β)	Average Concentration μc/ml (β)	Curie Hours (β)
1HP-3	35	2.71×10^{2}	2.75×10^{3}	1.38×10^{3}	4.73×10^{3}
Post 1HP-3	18	2.71×10^{2}	2.75×10^{3}	2.75×10^{3}	4.87×10^{3}
1HP-5	61	6.65×10^{2}	5.46×10^{3}	2.73×10^{3}	2×10^{4}
Boildown 1HP-5	42	6.65×10^{2}	1.9×10^{4}	1.2×10^{4}	2.8×10^{4}
Post 1HP-5	146	6.65×10^{2}	1.9×10^{4}	1.9×10^{4}	9.71×10^{4}
1HP-6	49	1.96×10^{3}	2.06×10^{4}	1.03×10^{4}	4.8×10^{4}
Post 1HP-6	33	1.96×10^{3}	2.06×10^{4}	2.06×10^{4}	6.46×10^{4}
1HP-7	45	1.6×10^{3}	1.6×10^{4}	8×10^{3}	3.6×10^{4}
Post 1HP-7	137	1.6×10^{3}	1.6×10^{4}	1.6×10^{4}	2.19×10^{5}
1HP-8	50	1.00×10^{4}	1.28×10^{5}	6.4×10^{4}	2.5×10^{5}
Post 1HP-8	177	1.00×10^{4}	1.28×10^{5}	1.28×10^{5}	1.77×10^{6}
1HP-9	74	1.67×10^{4}	3.53×10^{5}	2.40×10^{5}	1.35×10^{6}
Post 1HP-9	63	2.67×10^{4}	3.53×10^{5}	3.53×10^{5}	1.68×10^{6}
Total	930		Total β		5.57×10^{6}
			γ (50% of β)		2.79×10^{6}
			Total Radioactivity (Curie-Hours)		8.36×10^{6}

IN CONCENTRATING SECTION OF THE COLUMN, March 22 to August 7					
1HP-3	23		2×10^{-2}	1×10^{-2}	2.3×10^{-4}
1HP-5	87		2×10^{-2}	1×10^{-2}	8.7×10^{-4}
1HP-6	55		4×10^{-2}	2×10^{-2}	1.1×10^{-3}
1HP-7	64		6×10^{-2}	3×10^{-2}	1.9×10^{-3}
1HP-8	67		2.4×10^{-1}	1.2×10^{-1}	8×10^{-3}
1HP-9	87		4.5×10^{6}	2.3×10^{3}	2×10^{2}
Total	383		Total β		2×10^{2}
			γ (50% of β)		1×10^{2}
			Total Radioactivity (Curie-Hours)		3×10^{2}

Dwg. #13760

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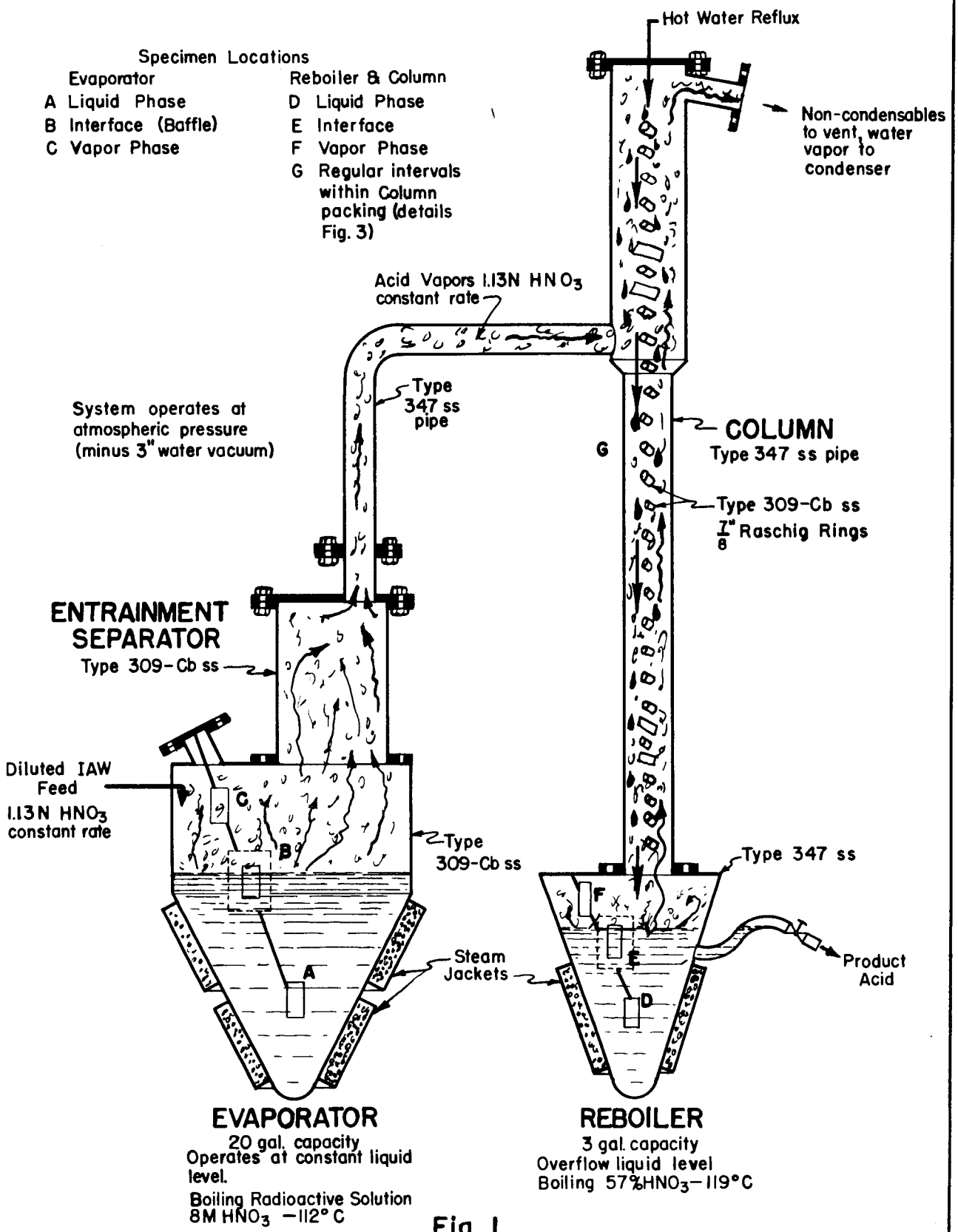

Fig. 1

EQUIPMENT & FLOW SKETCH

ACID RECOVERY UNIT & CORROSION SPECIMENS

ORNL PILOT PLANT

1-8-52

[redacted]

Dwg. #13761

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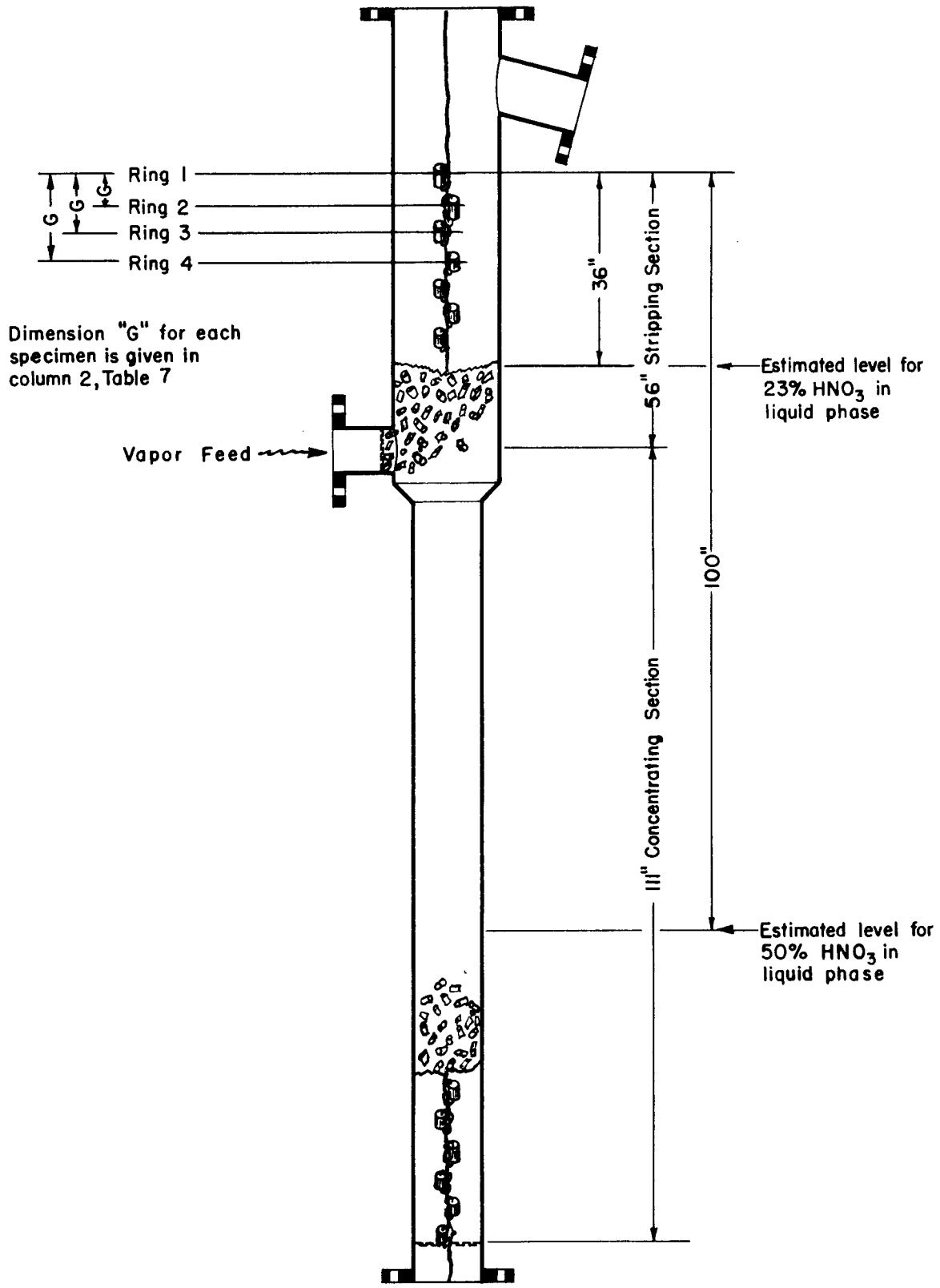

Fig. 2

LOCATION OF CORROSION SPECIMENS IN FRACTIONATION COLUMN

ACID RECOVERY UNIT — ORNL PILOT PLANT

1-8-52

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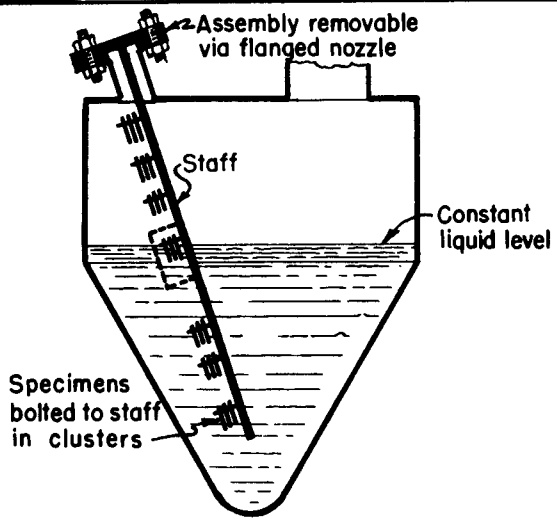
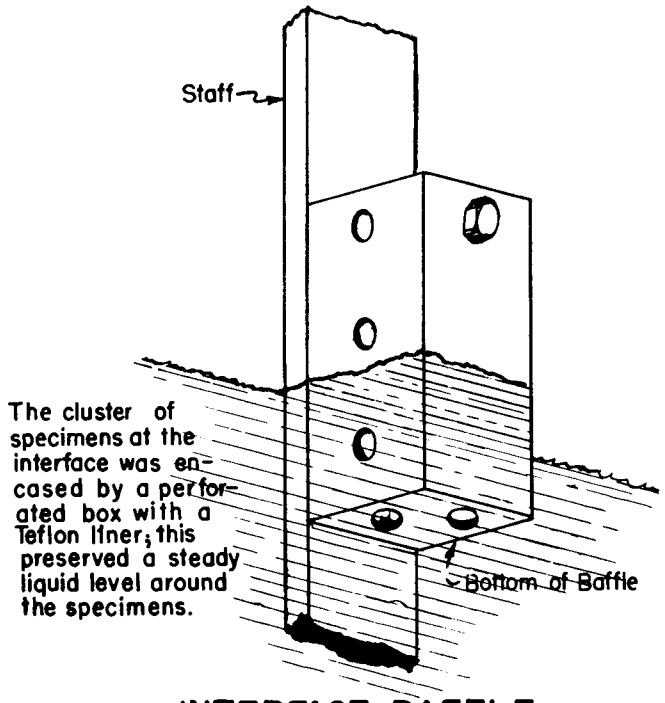
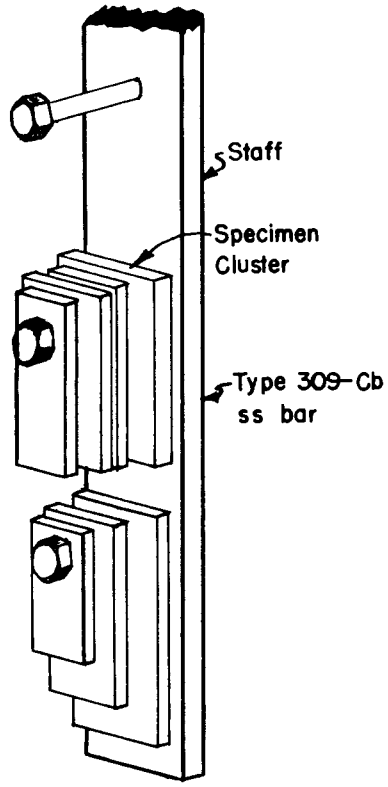


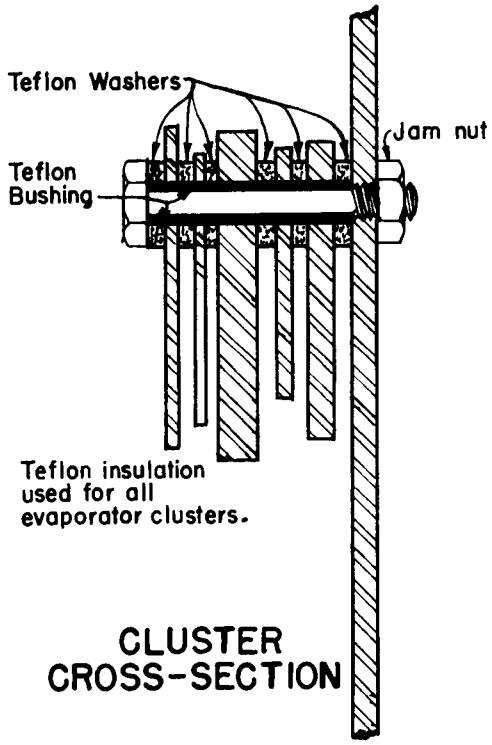
LOCATION IN EVAPORATOR



INTERFACE BAFFLE



CLUSTERS



CLUSTER CROSS-SECTION

Fig. 3

MOUNTING OF CORROSION SPECIMENS IN EVAPORATOR

ACID RECOVERY UNIT—ORNL PILOT PLANT

1-8-52

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Dwg. #13763

[redacted]

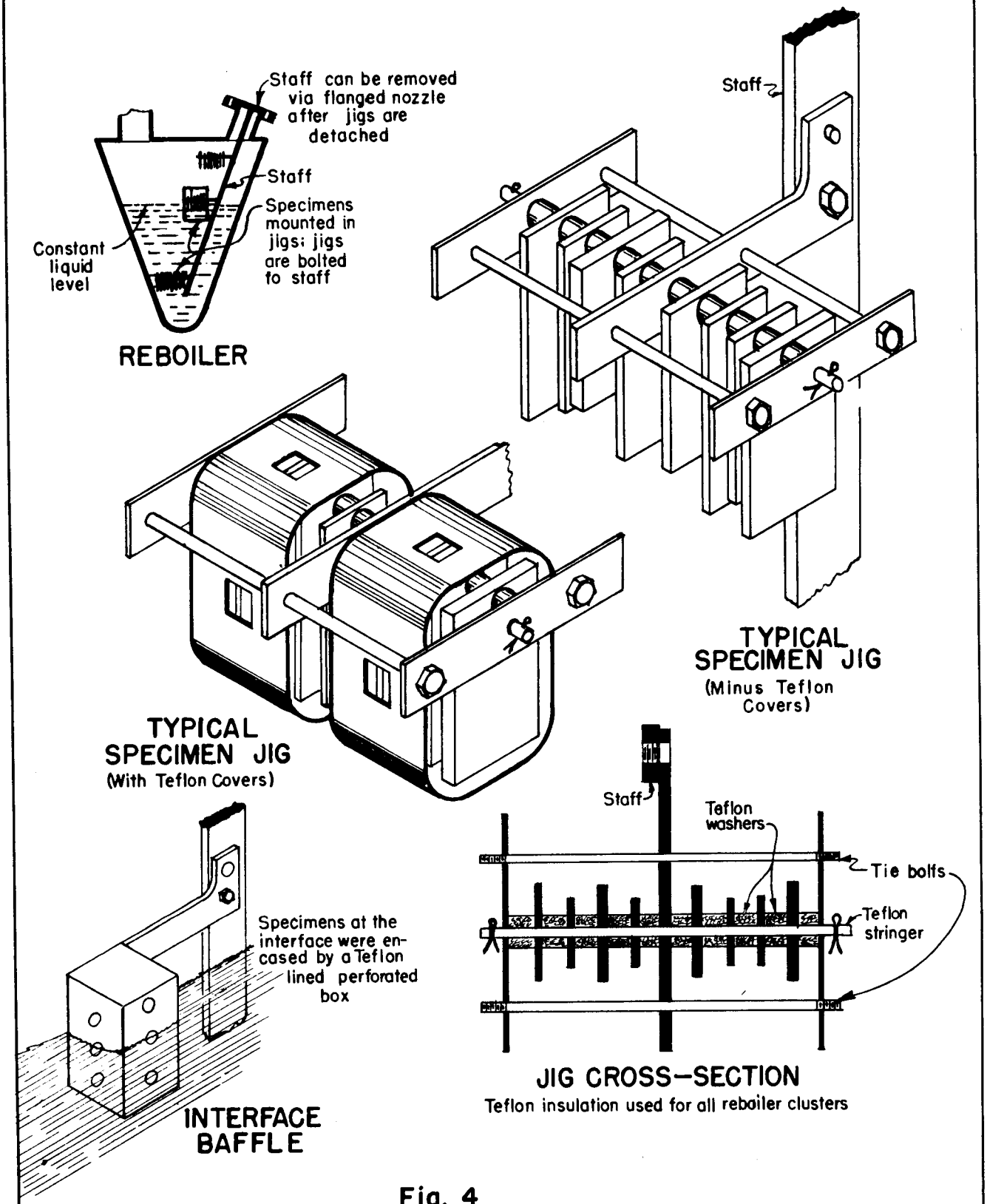

Fig. 4

MOUNTING OF CORROSION SPECIMENS IN REBOILER

ACID RECOVERY UNIT-ORNL PILOT PLANT

1-8-52

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Dwg. #13764

[redacted]

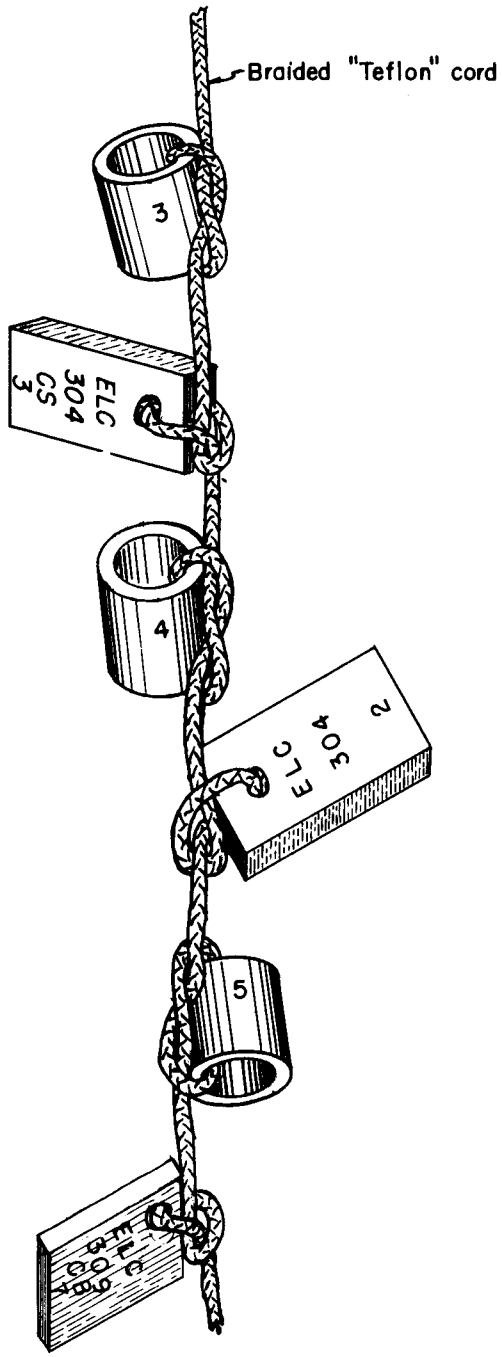

Fig. 5

MOUNTING OF CORROSION SPECIMENS IN ACID FRACTIONATING COLUMN

ACID RECOVERY UNIT ORNL PILOT PLANT

1-8-52

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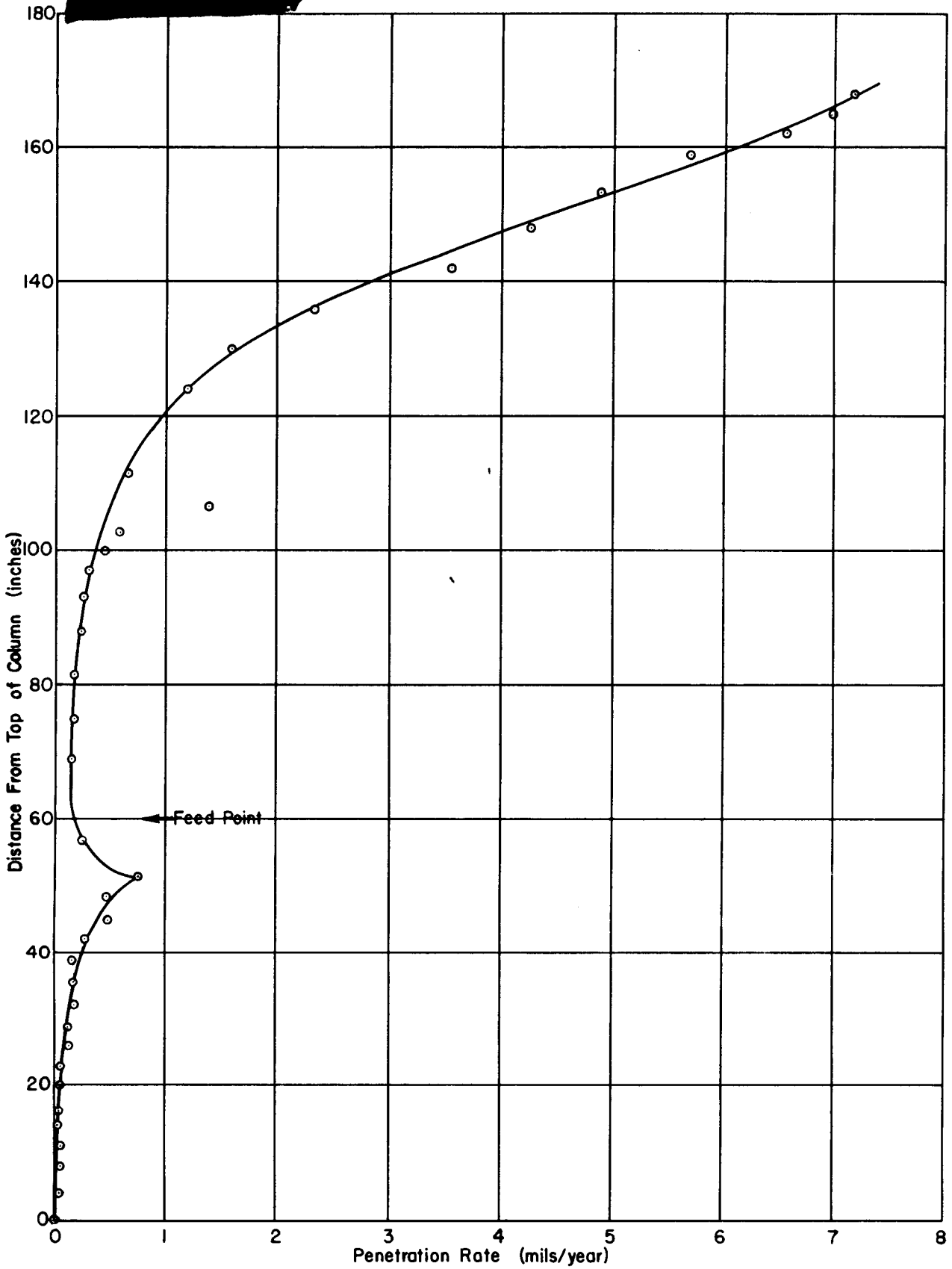

Fig. 6
PENETRATION RATE vs COLUMN LOCATION
for
TYPE 309-Cb STAINLESS STEEL RINGS

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1-8-52

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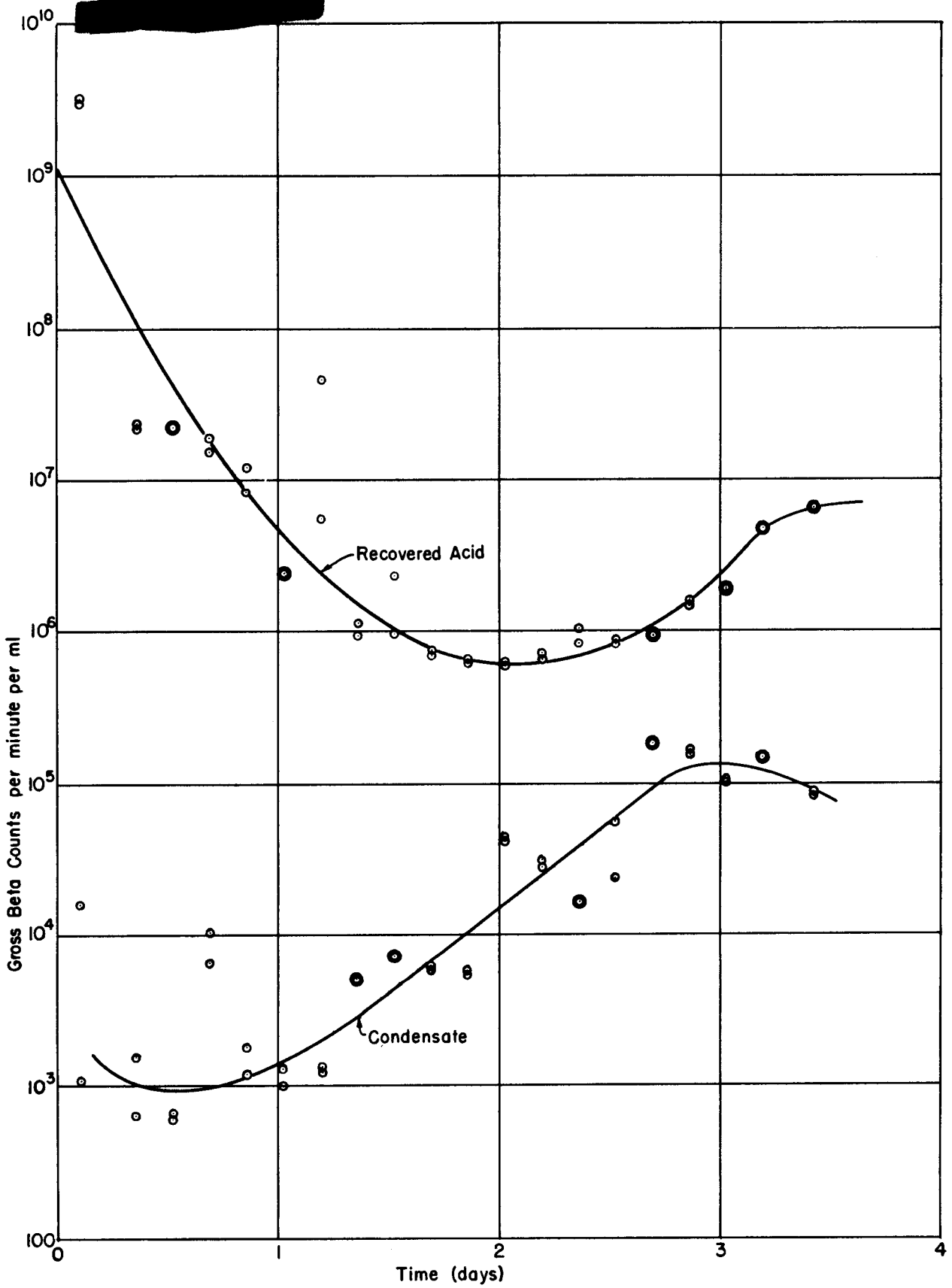

Fig. 7

ACTIVITY OF PRODUCT ACID & CONDENSATE DURING FINAL RUN

1-8-52

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