MASTER

CORROSION OF ALLOYS
IN VARIOUS ICPP DECONTAMINATING SOLUTIONS

T. L. Hoffman
G. S. Adams

April 14, 1961

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION

**NATIONAL REACTOR TESTING STATION
US ATOMIC ENERGY COMMISSION**

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

PRICE \$1.25

Available from the
Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Printed in USA

IDAHO CHEMICAL PROCESSING PLANT

IDO-14506
AEC Research and Development Report
Chemical Separations
Processes for Pu and U
TID-4500, Ed. 16
Issued: April 14, 1961

CORROSION OF ALLOYS IN VARIOUS ICPP DECONTAMINATION SOLUTIONS

T. L. Hoffman
G. S. Adams

Chemical Development Branch

PHILLIPS
PETROLEUM
COMPANY

Atomic Energy Division

Contract AT(10-1)-205

Idaho Operations Office

U. S. ATOMIC ENERGY COMMISSION

PAGES 2 to 4
WERE INTENTIONALLY
LEFT BLANK

CORROSION OF ALLOYS IN VARIOUS ICPP DECONTAMINATION SOLUTIONS

T. L. Hoffman
G. S. Adams

ABSTRACT

Corrosion studies were conducted on stainless steel types 347 and Carpenter-20, Monel, titanium 55A, and tantalum in decontamination solutions. These solutions are: 10% nitric acid, 10% citric acid, 10% sodium hydroxide - 2.5% tartaric acid, 10% oxalic acid, 0.003M periodic acid in 0.05M nitric acid, 3% sodium fluoride - 20% nitric acid, Turco 4501 and Turco 4502, and 0.25M phosphoric acid. Boron stainless steel type 304L was studied in 10% sodium hydroxide - 2.5% tartaric acid, 10 and 60% nitric acids, Turco 4501 and Turco 4502.

The two austenitic stainless steels are acceptable construction materials for handling each of the decontamination solutions except 3% sodium fluoride - 20% nitric acid. Special limitations are defined for Monel, titanium 55A, and tantalum when exposed to decontamination reagents.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

CORROSION OF ALLOYS IN VARIOUS ICPP DECONTAMINATION SOLUTIONS

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT.	5
I. SUMMARY	11
II. CONCLUSIONS	12
III. INTRODUCTION.	13
IV. EXPERIMENTAL.	14
A. Materials Employed.	14
1. Metals and Alloys	14
2. Decontaminating Solutions and Conditions.	14
B. Procedure	15
1. Cyclic Corrosion Experiments.	15
2. Corrosion Studies of Individual Decontamination Solutions	16
V. RESULTS	19
A. Cyclic Corrosion Experiments.	19
B. Individual Decontamination Solution Corrosion Studies .	21
VI. ACKNOWLEDGMENT.	28
VII. REFERENCES.	29
VIII. APPENDICES.	31

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Decontamination Solutions Used in the Cyclic Corrosion of Stainless Steel Type 347	15
2	Average General Corrosion of Stainless Steel Type 347 During the Decontamination Cycles	19
3	Predicted General Penetration of Unwelded Type 347 During 62-Hours Decontamination Cycles	20
4	Corrosion Rates of Various Alloys in Boiling Decontaminating Solutions	22
5	Corrosion of Boron Stainless Steel 304L in Selected Refluxed Boiling Decontaminating Solutions at Atmospheric Pressure	27

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Weld Deposit Pit	21
2	Penetration <u>vs</u> Time Curve of Welded Stainless Steel 347 in Boiling 10% Oxalic Acid	23
3	Penetration <u>vs</u> Time Curve of Unwelded Stainless Steel 347 in Boiling 10% Oxalic Acid	23
4	Penetration <u>vs</u> Time Curve of Welded Titanium 55A in Boiling 10% Oxalic Acid.	23
5	Penetration <u>vs</u> Time Curve of Unwelded Titanium 55A in Boiling 10% Oxalic Acid.	23
6	Penetration <u>vs</u> Time Curve of Monel (Combined Welded and Unwelded Data) in Boiling 0.003M Periodic Acid in 0.05M Nitric Acid	23
7	Penetration <u>vs</u> Time Curve of Welded Stainless Steel 347 in 3% Sodium Fluoride - 20% Nitric Acid at 144°F ± 10°F	23
8	Penetration <u>vs</u> Time Curve of Unwelded Stainless Steel 347 in 3% Sodium Fluoride - 20% Nitric Acid in 144°F ± 10°F	24
9	Penetration <u>vs</u> Time Curve of Stainless Steel Carpenter- 20 (Combined Welded and Unwelded Data) in 3% Sodium Fluoride - 20% Nitric Acid at 144°F ± 10°F.	24
10	Penetration <u>vs</u> Time Curve of Monel (Combined Welded and Unwelded Data) in 3% Sodium Fluoride - 20% Nitric Acid at 144°F ± 10°F	24
11	Penetration <u>vs</u> Time Curve of Titanium (Combined Welded and Unwelded Data) in 3% Sodium Fluoride - 20% Nitric Acid at 144°F ± 10°F.	24
12	Penetration <u>vs</u> Time Curve of Unwelded Tantalum in 3% Sodium Fluoride - 20% Nitric Acid at 144°F ± 10°F	24
13	Penetration <u>vs</u> Time Curve of Monel (Combined Welded and Unwelded Data) in Boiling Turco 4501	24
14	Penetration <u>vs</u> Time Curve of Titanium (Combined Welded and Unwelded Data) in Boiling Turco 4501	25
15	Penetration <u>vs</u> Time Curve of Titanium (Combined Welded and Unwelded Data) in Boiling Turco 4502	25
16	Surface of Boron Stainless Steel 304L.	27

CORROSION OF ALLOYS IN VARIOUS ICPP DECONTAMINATION SOLUTIONS

T. L. Hoffman
G. S. Adams

I. SUMMARY

Corrosion studies were conducted on welded and unwelded wrought stainless steel types 347 and Carpenter 20, Monel, titanium 55A, and unwelded commercial tantalum in several fission-product decontamination solutions. These solutions currently being used for decontamination in the Idaho Chemical Processing Plant (ICPP) are: 10% nitric acid, 10% citric acid, 10% sodium hydroxide - 2.5% tartaric acid, 10% oxalic acid, 0.003M periodic acid in 0.05M nitric acid, 3% sodium fluoride - 20% nitric acid, Turco 4501 and Turco 4502. In this study, 0.25M phosphoric acid was included as a potential decontaminant. The effect of 10% sodium hydroxide - 2.5% tartaric acid, 10% nitric acid, 60% nitric acid, Turco 4501 and Turco 4502 upon boron stainless steel was also studied. Test conditions were $144 \pm 10^\circ\text{F}$ for the sodium fluoride-nitric acid and the solution boiling point at about 650 mm pressure for all the others.

Repeated exposures to a series of typical ICPP decontaminants, under simulated plant conditions, revealed that welded 347 specimens corroded at a rate of 0.0031 ± 0.0012 inches penetration per month and unwelded specimens at a rate of 0.0039 ± 0.0012 ipm, stated at the 95% confidence level. Four cycles, each of a total of 62 hours duration, were used in the tests. The sodium fluoride-nitric acid reagent was responsible for the major portion of the corrosion.

All of the alloys exhibited suitable corrosion resistance toward boiling solutions of 10% citric acid. Monel dissolved in boiling 10% nitric acid, while the other alloys were resistant. Titanium is unsuited for handling 10% sodium hydroxide - 2.5% tartaric acid where concentration of these salts can occur as in heat transfer service. This decontaminant failed to attack any of the other alloys. Considerable corrosion attack was observed on all alloys when immersed in 3% sodium fluoride - 20% nitric acid at $144^\circ\text{F} \pm 10^\circ\text{F}$. The alloy best suited at this temperature for fluoride-nitric acid service appeared to be tantalum. Austenitic stainless steel exhibited relatively good resistance to boiling 10% oxalic acid, while both Monel and titanium 55A were severely attacked. Boiling solutions of 0.003M periodic acid in 0.05M nitric acid and 0.25M phosphoric acid attack both Monel and, to a lesser degree, titanium 55A. Boiling Turco 4501 dissolves tantalum.

Stainless steel 304L containing about 1% boron showed resistance to several of the decontaminating reagents similar to that for 304L containing no boron.

II. CONCLUSIONS

On the basis of current ICPP decontamination practice, the corrosion resistance of the various plant construction materials is encouraging. However, this study suggests that the Monel dissolver, designed for hydrofluoric acid service, and the titanium heat exchange surfaces of the aluminum raffinate evaporator, require special consideration.

With this knowledge of the corrosion sensitivity of the materials of construction of the plant vessels to the various decontaminants, it will be possible to establish plant decontamination procedures involving optimum contact times with firm knowledge of the resultant corrosion damage.

As new materials of construction and new reagents are introduced into plant use, it will be desirable to expand this work to include the new materials.

III. INTRODUCTION

The Idaho Chemical Processing Plant is designed for direct rather than remote maintenance. Accordingly, maintenance personnel must enter the cells containing the process vessels to perform certain repairs to equipment or to make major changes in the process vessels or lines. In order to obtain tolerable radiation levels in the cells for this work the process equipment must be decontaminated quite thoroughly. This involves the treatment of the vessels and lines with a group of chemical reagents which are proven decontaminants. Several of these reagents are known to be corrosive to the materials of construction of the plant equipment. Others are suspected of such corrosiveness. Since in several cases the factors which enhance decontamination, elevated temperature and prolonged contact time, also are likely to increase the corrosion of equipment, it is particularly important to have some impression of the corrosion penetration which will be experienced on the materials so that a proper balance can be obtained between equipment life and decontamination.

It is the purpose of the present work to provide this penetration data for the materials of construction at the Chemical Processing Plant with the well known decontaminating reagents used either singly or in one very common cycle. With these data available it will be possible to plan a decontamination program with full knowledge of the consequences of the effects of the reagents on the surfaces of the equipment.

IV. EXPERIMENTAL

A. Materials Employed

1. Metals and Alloys

The head-end process for aluminum-containing fuels requires vessels for handling a wide range of concentrations of nitric acid-aluminum nitrate mixtures at boiling temperatures. Type 347 (Cb and Ta) austenitic stainless steel is used extensively throughout the system for piping of process vessels. Minor quantities of type 304L were substituted for type 347 (Cb and Ta) when an improved delivery schedule from vessel fabricators could be obtained. The waste thermosyphon evaporator usually operates from a 35 psig steam chest at 277°F. However, on occasions, 50 psig steam (300°F) is used. For the latter service, titanium 55A is more suitable than type 347 (Cb and Ta) stainless steel, and is being used in the plant.

Monel and Carpenter-20 alloys are used as construction materials for other head-end batch processing vessels. In the zirconium fuel recovery process, an aqueous mixture of hydrofluoric and nitric acids is the fuel dissolution medium, and Monel is used as the batch dissolver vessel construction material. Following dissolution, the dissolver solution is transferred batchwise to a Carpenter-20 coarse feed adjustment vessel containing aluminum nitrate. This salt complexes excess fluoride and also forms uranyl nitrate which can be recovered by solvent extraction with tributyl phosphate.

The use of boron-containing Raschig rings has been proposed to poison large-diameter process equipment vessels. Corrosion rates are required on these ring materials to determine if there is any possibility of ring corrosion or of preferential leaching of boron into the process stream. The Superior Steel Company processed this alloy of surplus billets obtained from Knolls Atomic Power Laboratory.

The alloys used in this work were austenitic stainless steel type 347 (Cb and Ta), boron stainless steel type 304L, Carpenter-20, Monel, titanium 55A, and tantalum. Unwelded specimens of these alloys were studied in the as-received annealed metallurgical condition. Welded austenitic stainless steel types 347 (Cb and Ta), Carpenter 20, Monel, and titanium 55A specimens were tested in the as-welded condition. All coupons were taken from parent sheets of the appropriate alloy. Analyses of the metals are given in Appendix 1.

2. Decontaminating Solutions and Conditions

Chemical decontamination procedures are based upon experience gained at this plant and at Oak Ridge National Laboratory where the "25" Recovery Process was developed^(2,3). The first step in decontamination is a thorough flushing with water or steam of process solutions from the process vessels. Chemical solutions are then used to further decontaminate the process vessels.

The decontamination agents used in this corrosion study were 10% nitric acid, 60% nitric acid, 10% citric acid, 10% sodium hydroxide-2.5% tartaric acid, 10% oxalic acid, 0.003M periodic acid in 0.05M nitric acid, Turco 4501, Turco 4502, 3% sodium fluoride - 20% nitric acid, and 0.25M phosphoric acid. Turco 4501 and Turco 4502 are commercially-available decontaminating compounds⁽⁴⁾. Turco 4501 is marked as a viscous, high-boiling, alkaline liquid. The specific gravity at 25°C is 1.415. This material was used in the as-received condition, with frequent additions of water to replace evaporation losses. Turco 4502 is a granular free-flowing solid. It was used at a concentration of 2 pounds per gallon of water. Salts which meet ACS specifications and demineralized water were used to prepare the other solutions. Standard ICPP operations' practices were employed in the preparation of solutions, with the exception that purer salts were used.

B. Procedure

1. Cyclic Corrosion Experiments

Welded and unwelded coupons of type 347 stainless steel were exposed in turn to nine common decontamination solutions in a simulated decontamination cycle. The order of exposure, the solution, and the conditions are shown in Table 1. The coupons were totally immersed. Four complete cycles were used.

TABLE 1
DECONTAMINATION SOLUTIONS USED IN THE
CYCLIC CORROSION OF STAINLESS STEEL TYPE 347

<u>Solution Number</u>	<u>Medium</u>	<u>Temperature</u>	<u>Time Hours</u>
1	10% Citric Acid	Boiling	8
2	10% NaOH-2.5% Tartaric	Boiling	8
3	10% Nitric Acid	Boiling	8
4	10% Oxalic Acid	Boiling	8
5	10% Nitric Acid	Boiling	8
6	0.003M Periodic Acid 0.05M Nitric Acid	Boiling	4
7	10% Nitric Acid	Boiling	8
8	3% Sodium Fluoride in 20% Nitric Acid	144°F ± 10°F	2
9	10% Nitric Acid	Boiling	8
Total Continuous Exposure			62

Specimens were withdrawn after each of the first three cycles as follows: the specimens with the most shallow and least number of pits were withdrawn, leaving the more aggressively pitted specimens of the group for succeeding cycles. This method favored maximum pit penetration for the longest experiment.

Initially, five samples of the same metal or alloy were placed in each of four flasks. Two flasks contained welded samples and two flasks unwelded samples. The ratio of corrodent to exposed metal was 15 ml per square inch. The flasks were placed on a hot plate and fitted with reflux condensers. At the end of each period the solution was poured out and replaced by the next solution. Flasks and samples were not rinsed between solution changes in the same cycle. Because the sodium fluoride-nitric acid solution attacks glass, the samples were transferred to polyethylene beakers and placed in a hot water bath ($144^{\circ}\text{F} \pm 10^{\circ}\text{F}$). Following the sodium fluoride-nitric acid solution exposure period, the samples were replaced in their original flasks on the hot plates.

At the end of each 62-hr. cycle, the specimens were cleaned, dried, weighed, and examined microscopically at 30X. Specimens having the most shallow pits were withdrawn and the remainder used in the next cycle.

In this work, careful specimen control was exercised and variation in solution concentrations for various cycles was avoided. Flasks contained the specimens of one metal for each cycle. A sufficient quantity of each solution was prepared, initially, so that the entire study could be completed without subsequent batch makeup.

2. Corrosion Studies of Individual Decontamination Solutions

Stainless steel 347 and Carpenter-20, Monel, titanium 55A, and tantalum specimens were corrosion-evaluated in 10% nitric acid, 10% citric acid, 10% sodium hydroxide - 2.5% tartaric acid, 10% oxalic acid, 0.003M periodic acid - 0.05M nitric acid, 3% sodium fluoride - 20% nitric acid, 0.25M phosphoric acid, Turco 4501, and Turco 4502. Evaluation of these alloys in the 3% sodium fluoride - 20% nitric acid mixture was conducted at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$. Corrosiveness toward each different alloy of the other eight decontaminants was determined at the boiling temperature. Welded and unwelded specimens of these alloys were studied in the above solutions, with the exception of tantalum, which was evaluated in the unwelded condition only.

Specimens were totally immersed in each of the decontamination solutions for varying time intervals up to 360 hr. Permanent withdrawals of specimens were made at the end of 24, 72, 168, and 360 hr. term exposures. At the same time, periodic observations were made at the end of successive 24, 48, 96, and 192-hr. intervals, on specimens which were exposed for the entire 360-hr. study. Thus, 7 different test observations were made for each of the 5 different alloys in 9 decontaminating solutions.

Each of 4 different flasks initially contained specimens of the same alloy, with 5 welded specimens in each of 2 flasks and 5 unwelded specimens in each of the others. One of the specimens in each flask was assigned for repeated periodic observations, and the other 4 for observations after 1 term of corrosion. Specimens used for repeated observation were cleaned, dried, weighed, and re-immersed in the corrodent after each period. For term observations, 1 of the assigned specimens was cleaned, dried, weighed, and retired after each of the 4 different exposure periods. Accordingly, 3 specimens remained for term immersion

study after 24 hours, 2 after 72 hours, and 1 after 168 hours. Meanwhile, the specimen assigned for repeated study was periodically observed and retired after 4 periods of intermittent immersion. Ninety welded and 90 unwelded specimens of each alloy were required.

The ratio of decontamination solution to exposed metal was 15 ml per square inch.

Glass flasks and condensers were used to observe the effects of 10% nitric acid, 10% citric acid, 10% oxalic acid, 0.003M periodic acid in 0.05M nitric acid, 0.25M phosphoric acid on specimens of the various alloys. Flasks were placed upon the hot plates at boiling temperatures.

To study the effect of sodium fluoride-nitric acid solution on the various alloys, polyethylene beakers with covers were used. These vessels were placed in a hot water bath at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$.

For observing the corrosion behavior of various alloys in Turco 4501 and Turco 4502 decontaminants, special procedures were employed. Since the Turco solutions contain high caustic concentrations which rapidly dissolve glass at elevated temperatures, containment vessels of glass could not be used. Pots for stainless steel coupons were made of type 347, Monel coupons were immersed in a Monel vessel, and tantalum and titanium 55A coupons were immersed in Teflon beakers. Water-cooled condensers for these beakers were made from round-bottom boiling flasks which were covered with sheets of polyethylene paper. The metallic vessels with condensers were heated on hot plates, and the Teflon beakers with condensers were heated in a 240°F circulating oil bath.

To estimate the corrosiveness of a 2.5% tartaric - 10% sodium hydroxide toward the various alloys, Teflon beakers and polyethylene paper-covered water-cooled condensers were used. These beakers were heated in a 240°F circulating oil bath.

The use of boron-containing stainless steel Raschig rings has been proposed to poison large diameter vessels. The effect of 10% sodium hydroxide - 2.5% tartaric acid, 10% nitric acid, 60% nitric acid, Turco 4501 and Turco 4502 on boron stainless steel 304L coupons was observed. Nine 1-5/16 in. x 1 in. x 30 mils-thick specimens were fabricated from metal stock. These were exposed to solutions for varying time periods. Because of a shortage of material, the number of specimens per solution was limited. The arrangement of specimens in the solutions was as follows: 10% nitric acid, 1 specimen; 60% nitric acid, 2 specimens; 10% sodium hydroxide - 2.5% nitric acid, 2 specimens; Turco 4501, 2 specimens; and Turco 4502, 2 specimens.

The evaluation of boron stainless steel 304L was conducted in the decontaminants at boiling temperatures. The nitric acid studies were conducted in glass Erlenmeyer flasks fitted with glass condensers. Three 48-hr. periods constituted the corrosion time. Specimens were removed at the end of each 48-hr. period and examined under a wide field microscope. The samples were then cleaned, acetone-rinsed, weighed, rinsed with new solution, and immersed in the new solution. Specimens

exposed to sodium hydroxide-tartaric acid and the two Turco solutions, were immersed for a total of 10 days in Teflon beakers which were fitted with polyethylene-covered condensers. Removals were made on the 3rd, 5th, and 10th days. A visual inspection was given to each specimen under a wide field microscope prior to cleaning. This was followed by weighing, rinsing, and then re-immersing in a new solution.

V. RESULTS

A. Cyclic Corrosion Experiments

The data for the general corrosion during the cyclic tests are summarized in Table 2. The detailed data, including the rate data for each coupon, are shown in Appendix 2. Although some scatter of the data is shown between cycles and between duplicate experiments in Table 2, the average rates for the first cycle agree very well with the cumulative average rates through the subsequent cycles. Thus, for welded stainless steel 347, the average rate for the 1st cycle and the cumulative rates through the 2nd, 3rd, and 4th cycles are 3.17, 3.12, 3.12, and 3.11 mils per month. For the unwelded material, the corresponding values are 4.32, 4.07, 4.00, and 3.94 mils per month. Because of the high corrosion rate of the nitric acid-sodium fluoride solution, the temperature dependency of that rate, and the experimental difficulties experienced in maintaining a constant temperature with this test mixture, it is assumed that the largest portion of the experimental error is due to this portion of the cycle. The temperature-rate observation for Cycles III and IV of Table 2 does not support this assumption quantitatively, however.

TABLE 2
AVERAGE GENERAL CORROSION OF STAINLESS STEEL TYPE 347 DURING THE DECONTAMINATION CYCLES

Specimen Type	Flask No.	Cycle I		Cycle II		Cycle III		Sol'n No. 8 Temp. °F Av.	Cycle IV		Sol'n No. 8 Temp. °F Av.
		Av. Rate IPM	No. of Samples in Av.	Av. Rate IPM	No. of Samples in Av.	Av. Rate IPM	No. of Samples in Av.		Av. Rate IPM	No. of Samples in Av.	
Unwelded	1	0.0049	5	0.0045	4	0.0033	2	139	0.0035	2	140
Unwelded	3	0.0037	5	0.0027	3	0.0040	2	150	0.0037	1	136
Welded	2	0.0035	5	0.0034	4	0.0030	3	143	0.0033	1	136
Welded	4	0.0028	5	0.0027	4	0.0033	2	144	0.0036	1	142

It has been concluded that there was no evidence of accelerated corrosion with repeated cycles.

For practical application, the results for this particular 62-hr. cycle have been calculated in terms of mils penetration per cycle. The cumulative penetration for up to 4 cycles is given in Table 3, with estimates of the uncertainties associated with the prediction of corrosion from these data. The rates are based on 3.9 mils per month - the rate for unwelded stainless steel 347, which was the greater of the two. The uncertainties were derived from the pooled data, 48 values, and assume that a plant vessel or group of equipment represents a very large number of samples. The statistical analysis also makes it possible to estimate the penetration to be anticipated for the areas where, by chance, the

TABLE 3.

PREDICTED GENERAL PENETRATION OF UNWELDED STAINLESS STEEL 347
DURING 62-HR. DECONTAMINATION CYCLES^(a)

<u>Cycles</u>	Average ^(b) Cumulative Penetration (mils)	Penetration is Predicted ^(b) to Exceed These Values on	
		2.5% of the Surface Area (mils)	0.1% of the Surface Area (mils)
1	0.34	0.46	0.56
2	0.68	0.85	0.99
3	1.02	1.2	1.4
4	1.36	1.6	1.8

(a) This is the standard decontamination cycle described in Table 1.

(b) Only general penetration is considered.

corrosion will be greater than the average. Thus, after 4 cycles the average cumulative penetration will be 1.36 mils, but it would be predicted that 2.5% of the surface area would experience a penetration in excess of 1.6 mils and 0.1% of the area would experience a penetration in excess of 1.8 mils. Using the data of Table 4 (which will be discussed in detail later), it was possible to verify the results of the cyclic corrosion tests by summing the values for the data of the individual reagents. This was effectively 4 times the 2-hr. penetration for nitric acid-sodium fluoride and 4 times the 8-hr. penetration for oxalic acid, each on stainless steel 347, with the other components of the cycle not contributing to the penetration. This summation was 1.38 mils for 4 cycles, which compares favorably with the cumulative penetration for 4 cycles of Table 3. The uncertainties associated with this summation at the 95% confidence level were +1.9 and -0.5. These uncertainties are regarded as unrealistically high, since they involve fewer observations than the cyclic data, and the nitric acid-sodium fluoride data are undoubtedly influenced by high and erratic results at the longer contact times. Although there is no evidence that repeated cycles of decontamination are leading to accelerated corrosion rates, it is not recommended that these data be extrapolated beyond the 4 cycles for which there is experimental verification.

Pit depth on weld deposits increased with the number of cycles. Ten and 15-mil deep penetrations were present after the 3rd and 4th cycles respectively. Fig. 1 is a micrograph of a 15-mil deep weld deposit penetration. Unwelded specimens were generally found to be pit free.

Intergranular attack was found to be absent by metallographic examination on both welded and unwelded specimens after 4 cycles.

FIGURE 1

Etch: Aqua Regia

250X

WELD DEPOSIT PIT

Stainless steel type 347 (Cb and Ta) after immersion in four 62-hr. CPM decontamination cycles.

B. Individual Decontamination Solution Corrosion Studies

A summary of the results of the tests with individual decontaminating solutions is presented in Table 4. Actual general corrosion rates are given in the table for those metal-reagent combinations which show either very low attack or extremely high attack. Many of the former can be ignored in the planning of a decontamination program. The latter combinations, where the rates of attack are very high, probably must be avoided or extreme care exercised in their use. Where moderate to fairly heavy attack is expected, the data have been plotted to show total penetration as a function of contact time, to allow rapid estimation of the corrosion. These plots are given in Fig. 2 through 15. They were prepared by statistical analysis of the experimental results. All of the detailed data are given in Appendices 3 through 11.

TABLE 4

CORROSION RATES OF VARIOUS ALLOYS IN BOILING DECONTAMINATING SOLUTIONS^(a,b)

Decontaminating Solutions	Alloys								
	Stainless Steel 347 Welded	Stainless Steel 347 Unwelded	Carpenter-20 Welded	Carpenter-20 Unwelded	Monel Welded	Monel Unwelded	Titanium Welded	Titanium Unwelded	Tantalum Unwelded
Rates are in mils per hour									
10% Nitric Acid	0.000	0.000	0.000	0.000	1.94	2.22	0.000	0.000	0.000
10% Citric Acid	0.000	0.000	0.000	0.000	0.001	0.004	0.000	0.000	0.000
10% Sodium Hydroxide 2,5% Tartaric Acid	0.000	0.000	0.000	0.000	0.000	0.001	0.002 ^(c)	0.002 ^(c)	0.000
10% Oxalic Acid	Figure 2	Figure 3	0.002	0.001	0.032	0.038	Figure 4	Figure 5	0.000
0.003M Periodic Acid 0.05M Nitric Acid	0.000	0.000	0.000	0.000	-----Figure 6-----		0.000	0.000	0.000
0.25M Phosphoric Acid	0.000	0.000	0.000	0.000	0.006	0.026	0.005	0.008	0.000
20% Nitric Acid 3% Sodium Fluoride	Figure 7	Figure 8	----Figure 9-----		-----Figure 10----		-----Figure 11-----		Figure 12
Turco 4501	0.000	0.000	0.000	0.000	-----Figure 13----		-----Figure 14-----		0.276
Turco 4502	0.000	0.000	0.000	0.000	0.000	0.000	-----Figure 15-----		Not Studied

(a) The data for 20% nitric acid - 3% sodium fluoride are at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$.

(b) Where very low corrosion rates were evidenced in a system, a nominal value in mils per hour is given for the rate. Where intermediate to relatively high rates were experienced, there are references to the Figures in the text. These give the penetration to be expected as a function of contact time. High corrosion rates are marked for added emphasis, and a nominal value in mils per hour is given.

(c) Although the general rate of attack was low, serious pitting was observed. See text.

Fig. 2 - Penetration vs Time Curve of Welded Stainless Steel 347 in Boiling 10% Oxalic Acid

Fig. 3 - Penetration vs Time Curve of Unwelded Stainless Steel 347 in Boiling 10% Oxalic Acid

Fig. 4 - Penetration vs Time Curve of Welded Titanium 55A in Boiling 10% Oxalic Acid

Fig. 5 - Penetration vs Time Curve of Unwelded Titanium 55A in Boiling 10% Oxalic Acid

Fig. 6 - Penetration vs Time Curve of Monel (Combined Welded and Unwelded Data) in Boiling 0.003M Periodic Acid in 0.05M Nitric Acid

Fig. 7 - Penetration vs Time Curve of Welded Stainless Steel 347 in 3% Sodium Fluoride - 20% Nitric Acid at 144°F ± 10°F

Fig. 8 - Penetration vs Time Curve of Unwelded Stainless Steel 347 in 3% Sodium Fluoride - 20% Nitric Acid at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$

Fig. 9 - Penetration vs Time Curve of Stainless Steel Carpenter-20 (Combined Welded and Unwelded Data) in 3% Sodium Fluoride - 20% Nitric Acid at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$

Fig. 10 - Penetration vs Time Curve of Monel (Combined Welded and Unwelded Data) in 3% Sodium Fluoride - 20% Nitric Acid at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$

Fig. 11 - Penetration vs Time Curve of Titanium (Combined Welded and Unwelded Data) in 3% Sodium Fluoride - 20% Nitric Acid at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$

Fig. 12 - Penetration vs Time Curve of Unwelded Tantalum in 3% Sodium Fluoride - 20% Nitric Acid at $144^{\circ}\text{F} \pm 10^{\circ}\text{F}$

Fig. 13 - Penetration vs Time Curve of Monel (Combined Welded and Unwelded Data) in Boiling TURCO 4501

Fig. 14 - Penetration vs Time Curve of Titanium (Combined Welded and Unwelded Data) in Boiling Turco 4501

Fig. 15 - Penetration vs Time Curve of Titanium (Combined Welded and Unwelded Data) in Boiling Turco 4502

In the preparation of Fig. 2 through 15, the functional form which most reasonably describes the relationship between penetration and time was selected as:

$$P = at^b \quad (1)$$

where P = penetration depth (mils)

t = exposure time (hours)

and a and b are constants for given conditions of solution composition, metal and temperature.

An analysis of variance was conducted on the data obtained from the study of all metal-solution combinations, both welded and unwelded. A test of significance was made of the effect of welding on corrosion. In the cases where this effect was not significant, the data for welded and unwelded samples were combined. For certain combinations of reagent and metal, the rates were either essentially zero or extremely high. No analysis was made for these cases, but rate estimates were made by inspection of the data and are reported in Table 4. Each of the figures shows the best value for the specific data in the form of Equation 1 and the limits of uncertainty (at the 95% confidence level) due to the experimental error. It is not recommended that the data be used beyond the 360-hr. period covered by experiment. It should also be noted that, except for the nitric acid-sodium fluoride, these observations were made at the boiling point of the reagents at about 650 mm of mercury. The boiling points of aqueous solutions are generally about 4 or 5°C lower at this pressure than at 760 mm of mercury.

Detailed observations of the results of certain of the individual tests are given below:

In boiling Turco 4502, all the alloys studied suffered less than 2.0 mils per month corrosion. Welded Carpenter-20 and 347 suffered more attack than did the corresponding unwelded specimens. Weld areas on all the AISI 347 specimens were moderately etched. A notch decay was observed in the weld on one Carpenter-20 alloy specimen. These effects are due, in part, to "sensitization" associated with the welding process.

Titanium is unsuited for service in boiling 2.5% tartaric acid - 10% sodium hydroxide. This material suffered pits up to 20 mils deep and extensive general attack after 48 hours immersion in the boiling solution. The other alloys completely resisted the tartaric acid-caustic boiling mixtures.

Stainless steel types Carpenter-20 and 347 are resistant to 0.25M phosphoric acid, while Monel and titanium suffered excessive attack.

With the exception of Monel, alloys studied were completely resistant toward 0.05M nitric acid containing 0.003M periodic acid.

All the alloys suffered excessive attack when exposed to 20% nitric acid containing 3% sodium fluoride. The most resistant metal was tantalum.

When immersed in boiling 10% oxalic acid, welded and unwelded Monel and titanium suffered excessive general attack.

All alloys except tantalum and titanium resist boiling Turco 4501 up to 360 hours exposure. Tantalum dissolves slowly when air is above the solution, with an observed rate of 6 to 7 mils per day or 200 mils per month. Titanium suffered a lower rate of about 70 mils per month corrosion. Boiling Turco 4501 attacks titanium 55A. The amine present may contribute to the attack. Wrought titanium resisted all concentrations of caustic up to 73% sodium hydroxide at 240°F for the period of 162 days. However, titanium immersed in alkali which contained small amounts of ammonia suffered excessive attack⁽⁵⁾.

Table 5 lists the periodic and cumulative corrosion rates of boron stainless steel 304L toward 10 and 60% nitric acid, 10% sodium hydroxide - 2.5% tartaric acid, Turco 4501 and Turco 4502. Under these test conditions, the boron stainless steel 304L has acceptable corrosion rates which are comparable to those of boron-free stainless steel. A metal-lurgical examination revealed the absence of preferential leaching of boron from stainless steel, Fig. 16.

TABLE 5

CORROSION OF BORON STAINLESS STEEL 304L IN SELECTED REFLUXED
BOILING DECONTAMINATING SOLUTIONS AT ATMOSPHERIC PRESSURE

Decontaminating Solution	Average Penetration (mils/month)					Cumulative Penetration Mils/Month
	24 Hrs	48 Hrs	72 Hrs	120 Hrs	240 Hrs	
10% Nitric Acid	0.08	0.09	0.09	----	----	0.09
60% Nitric Acid	1.69	1.65	1.68	----	----	1.67
60% Nitric Acid	1.62	1.57	1.65	----	----	1.61
10% Sodium	----	----	0.02	0.01	0.01	0.01
Hydroxide - 2.5%	----	----	0.00	0.01	0.01	0.01
Tartaric Acid	----	----	----	----	----	----
Turco 4501	----	----	0.02	0.01	0.03	0.02
Turco 4502	----	----	0.02	0.01	0.04	0.04
Turco 4502	----	----	0.09	0.03	0.04	0.05
Turco 4502	----	----	0.06	0.02	0.04	0.04

Fig. 16 - Surface of Boron Stainless Steel 304L;
Etch: 5% Picral and 2% HCl in Methanol

250X

The dark laminae are iron borides. Preferential leaching was absent after 144 hr. exposure to boiling 60% nitric acid.

VI. ACKNOWLEDGMENT

Acknowledgment is made to D. L. Weaver, Laboratory Technician, for performing many of the corrosion experiments. Appreciation is expressed to Lewis B. Hansen, Mathematical Statistics, Operations Evaluation, for the statistical analysis of portions of the corrosion data.

VII. REFERENCES

1. Phillips Petroleum Company, "Chemical Processing of Reactor Fuel Elements at the Idaho Chemical Processing Plant", Chemical Processing and Equipment, United States Government Printing Office (1955)
2. Stevenson, C. E., "Technical Progress Report for April Through June 1958, Idaho Chemical Processing Plant", IDO-14453, p. 16
3. Watson, Clyde D., A General Decontamination Manual for the Idaho Chemical Processing Plant, Oak Ridge National Laboratory, ~~XXXX~~ IDO-26081
4. Specialized Chemical Processing Compounds for Radioactive Decontamination, Turco Products, Inc., Newark, New Jersey
5. Gegner, Paul J., and W. L. Wilson, "Corrosion Resistance of Titanium and Zirconium in Chemical Plant Exposure", Corrosion, Vol. 15, July, 1959 (P341t-350t)

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

VIII. APPENDICES

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX 1

NOMINAL COMPOSITION OF ALLOYS USED IN CORROSION STUDIES
OF VARIOUS ICPP DECONTAMINATING SOLUTIONS

Alloy	% C	% Cr	% Ni	% Mo	% Cu	% Mn	% Cb Ta ₂ O ₅	% N ₂	% H ₂	% Si	% Fe	% Ti	% B	% Ta
TYPE 347	0.08 Max.	17-19	9-13			2.50 Max.	1.0 Max. 10XC				Bal.			
Carpenter- 20	0.07 Max.	19-21	24-30	2-3	3-4						Bal.			
Titanium 55A	0.15 Max.							0.05 Max.	0.015 Max.	0.05 Max.	0.12 Max.	Bal.		
Tantalum	0.10 Max.									0.01	0.01			99.8
Monel	0.30		60.0 Min.		Bal.	3.5 Max.				0.50 Max.	3.5 Max.			
Monel 60 INCO Wire	0.30		60.0 Min.		23.0 Min.	3.5 Max.				0.50 Max.	3.5 Max.			
Monel 130X INCO	0.40 Max.		62-70		Bal.	4.0 Max.				1.00 Max.	2.5 Max.	4.00 1.75		
E 347 Weld Wire	0.08 Max.	18-21	9-11			1.0-2.5	8XC Min. 1.00 Max			0.90 Max.	Bal.			
Boron SS Type 304L	0.035	18.9	15.02			0.8				0.08	Bal.		1.35	

APPENDIX 2

PERIOD CORROSION RATES OF STAINLESS STEEL TYPE 347 IMMERSED IN FOUR DECONTAMINATION CYCLES

Cycle No. 1		
Flask No.	Sample No.	Period Rate (Inches/Month)
4	DD 266	0.0027
	DD 267	0.0028
	DD 268	0.0029
	DD 269	0.0029
	DD 270	0.0028
2	DD 271	0.0035
	DD 272	0.0036
	DD 273	0.0036
	DD 274	0.0035
	DD 275	0.0034
1	D 448	0.0049
	D 449	0.0052
	D 450	0.0049
	D 451	0.0049
	D 452	0.0048
3	D 453	0.0035
	D 454	0.0037
	D 455	0.0039
	D 456	0.0035
	D 457	0.0039

Cycle No. 2		
Flask No.	Sample No.	Period Rate (Inches/Month)
4	DD 267	0.0031
	DD 268	0.0025
	DD 269	0.0027
	DD 270	0.0025
2	DD 271	0.0034
	DD 272	0.0035
	DD 274	0.0034
	DD 275	0.0034
1	D 449	0.0045
	D 450	0.0045
	D 451	0.0043
	D 452	0.0047
3	D 455	0.0019
	D 456	0.0027
	D 457	0.0033

Cycle No. 3		
Flask No.	Sample No.	Period Rate (Inches/Month)
4	DD 268	0.0032
	DD 269	0.0034
2	DD 272	0.0028
	DD 274	0.0032
	DD 275	0.0030
1	D 450	0.0035
	D 451	0.0033
	D 452	0.0031
3	D 456	0.0042
	D 457	0.0038

Cycle No. 4		
Flask No.	Sample No.	Period Rate (Inches/Month)
4	DD 269	0.0036
2	DD 272	0.0023
1	D 451	0.0031
	D 452	0.0038
3	D 456	0.0037

DD: Welded Samples
D: Unwelded Samples

APPENDIX 3

INCHES. PENETRATION PER MONTH CORROSION RATE OF
VARIOUS METALS AND ALLOYS IN BOILING 10% NITRIC ACID

Specimen No.	6 1/2 Hr Period Rate	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 311		0.0000	0.0000		0.0000		0.0000	
319								0.0000
320						0.0000		
323				0.0000				
334		0.0000						
D 298		0.0000	0.0000		0.0000		0.0000	
299								0.0000
340						0.0000		
343				0.0000				
345		0.0000						
DD 279		0.0002	0.0001		0.0001		0.0001	
384								0.0001
385						0.0001		
422				0.0001				
425		0.0003						
DD 377		0.0002	0.0001		0.0001		0.0001	
383								0.0001
387						0.0001		
410				0.0002				
419		0.0005						
E 206		0.0000	0.0000		0.0000		0.0000	
212								0.0000
235						0.0000		
249				0.0000				
264		0.0000						
E 220		0.0000	0.0000		0.0000		0.0000	
246								0.0000
256						0.0000		
272				0.0000				
274		0.0000						
EE 288		0.0001	0.0000		0.0000		0.0000	
290								0.0000
293						0.0000		
305				0.0001				
347		0.0001						

(Continued)

APPENDIX 3 (Continued)

Specimen No.	6 1/2 Hr Period Rate	24 Hr Period Rate	48 Hr Period Rate	72 Hr Period Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 281		0.0001	0.0001		0.0001		0.0000	
311								0.0000
312						0.0000		
322				0.0000				
334		0.0001						
I 77	0.3466							
84								
94								
99								
108	0.3285							
I 63	0.3966							
81								
87								
105								
106	0.5458							
II 125	0.3878							
128								
137								
144								
149	0.1526							
II 116	0.4423							
117								
127								
129								
130	0.4170							

Ta: Welded and unwelded Titanium 55A and unwelded Tantalum completely

Ti:

TTi: resist 10% nitric acid at all temperatures up to boiling.

Notes: D: Unwelded stainless steel type 347.
 DD: Welded stainless steel type 347.
 E: Unwelded stainless steel type Carpenter-20.
 EE: Welded stainless steel type Carpenter-20.
 I: Unwelded Monel.
 II: Welded Monel.
 Ta: Unwelded Tantalum.
 Ti: Unwelded Titanium 55A.
 TTi: Welded Titanium 55A.

APPENDIX 4

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS METALS AND ALLOYS IN BOILING 10% CITRIC ACID

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 301			0.0000				
326							0.0000
338	0.0000						
346					0.0000		
363	0.0000	0.0000		0.0000		0.0000	
D 327							0.0000
342					0.0000		
347			0.0000				
352	0.0000	0.0000		0.0000		0.0000	
362	0.0000						
DD 292	0.0001						
293							0.0000
294	0.0001	0.0000		0.0000		0.0000	
296					0.0000		
297			0.0000				
DD 276					0.0000		
277							0.0000
278	0.0000						
288	0.0001	0.0000		0.0000		0.0000	
291			0.0000				
E 208							0.0003
226	0.0000	0.0002		0.0000		0.0000	
232			0.0001				
238	0.0001						
247					0.0000		
E 207							0.0000
209	0.0000	0.0000		0.0000		0.0000	
248			0.0000				
252					0.0000		
253	0.0000						
EE 282			0.0000				
296	0.0002						
300	0.0001	0.0002		0.0001		0.0000	
341							0.0000
342					0.0002		

(Continued)

APPENDIX 4 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 283					0.0000		
304							0.0000
307	0.0001						
331			0.0000				
332	0.0000	0.0001		0.0001		0.0000	
I 69					0.0026		
73	0.0023	0.0023		0.0029		0.0058	
79	0.0022						
80							0.0042
113			0.0023				
I 81	0.0012						
87							0.0024
98					0.0011		
103	0.0014	0.0021		0.0020		0.0036	
112			0.0018				
II 29	0.0008	0.0001		0.0002		0.0002	
34	0.0006						
37					0.0001		
53							0.0002
60			0.0001				
II 35							0.0002
42					0.0002		
50	0.0007	0.0001		0.0002		0.0001	
51			0.0001				
54	0.0007						
TI 154	0.0000	0.0000		0.0000		0.0000	
157							0.0000
160					0.0000		
164			0.0000				
166	0.0000						
TI 172	0.0000	0.0000		0.0000		0.0000	
178							0.0000
183					0.0000		
187			0.0000				
190	0.0000						
TTI 220	0.0000	0.0000		0.0000		0.0000	
221							0.0000
224					0.0000		
232			0.0001				
240	0.0001						

(Continued)

APPENDIX 4 (Continued).

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TTI 247	0.0001	0.0000		0.0000		0.0000	
250							0.0000
258					0.0000		
259			0.0001				
261	0.0000						
Ta 27	0.0000	0.0000		0.0000		0.0000	
34							0.0000
36					0.0000		
38			0.0000				
53	0.0000						
Ta 59	0.0000	0.0000		0.0000		0.0000	
62							0.0000
66					0.0000		
69			0.0000				
72	0.0000						

D: Unwelded stainless steel type 347.
DD: Welded stainless steel type 347.
E: Unwelded stainless steel type Carpenter-20.
EE: Welded stainless steel type Carpenter-20.
I: Unwelded Monel.
II: Welded Monel.
Ta: Unwelded Tantalum.
Ti: Unwelded Titanium 55A.
TTi: Welded Titanium 55A.

APPENDIX 5

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS METALS AND ALLOYS IN BOILING 2.5 TARTARIC AND 10% SODIUM HYDROXIDE

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 312					0.0000		
308							0.0000
300	0.0000	0.0000		0.0000		0.0000	
329			0.0000				
330	0.0000						
D 328	0.0001	0.0000		0.0000		0.0000	
335							0.0000
348					0.0000		
349			0.0000				
370	0.0001						
DD 376	0.0002	0.0000		0.0000		0.0000	
394							0.0000
402					0.0000		
414			0.0000				
418	0.0002						
DD 373	0.0003	0.0000		0.0000		0.0000	
374							0.0000
389					0.0000		
403			0.0000				
433	0.0002						
E 203	0.0000	0.0000		0.0000		0.0000	
213							0.0000
255					0.0000		
267			0.0000				
269	0.0000						
E 219	0.0000	0.0000		0.0000		0.0001	
222							0.0000
236					0.0000		
239			0.0000				
250	0.0000						
EE 302	0.0000	0.0000		0.0000		0.0000	
314							0.0001
336					0.0000		
339			0.0000				
340	0.0000						

(Continued)

APPENDIX 5 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 299	0.0000	0.0000		0.0000		0.0000	
328							0.0000
330					0.0000		
338			0.0000				
345	0.0000						
I 73	0.0033	0.0003		0.0001		0.0002	
89							0.0004
92					0.0006		
110			0.0014				
114	0.0024						
I 78	0.0000	0.0000		0.0000		0.0001	
102							0.0001
107					0.0000		
108			0.0000				
109	0.0000						
II 135	0.0000	0.0000		0.0000		0.0004	
141							0.0002
150					0.0000		
156	0.0000						
153			0.0000				
II 133	0.0001	0.0000		0.0000		0.0001	
152							0.0000
154					0.0000		
157			0.0000				
159	0.0000						
Ti 464	0.0024	0.0023		0.0015		0.0012	
465							0.0001
466					0.0008		
467			0.0013				
468	0.0030						
Ti 469	0.0024	0.0027		0.0013		0.0012	
470							0.0001
471					0.0009		
472			0.0011				
473	0.0022						
TTi 474	0.0023	0.0034		0.0020		0.0016	
475							0.0000
476					0.0006		
477			0.0013				
478	0.0028						

(Continued)

APPENDIX 5 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TTi 479	0.0022	0.0037		0.0021		0.0019	
480							0.0000
481					0.0006		
482			0.0014				
483	0.0028						
Ta 26	0.0000	0.0000		0.0001		0.0000	
40							0.0000
41					0.0000		
43			0.0000				
47	0.0000						
Ta 50	0.0001	0.0000		0.0000		0.0000	
51							0.0000
54					0.0000		
56			0.0000				
64	0.0001						

D: Unwelded stainless steel type 347.
 DD: Welded stainless steel type 347.
 E: Unwelded stainless steel type Carpenter-20.
 EE: Welded stainless steel type Carpenter-20.
 I: Unwelded Monel.
 II: Welded Monel.
 Ta: Unwelded Tantalum.
 Ti: Unwelded Titanium 55A.
 TTi: Welded Titanium 55A.

APPENDIX 6

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS METALS AND ALLOYS IN BOILING 10% OXALIC ACID

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 303	0.0047	0.0028		0.0009			
310							0.0014
318					0.0015		
321			0.0026				
332	0.0052						
D 339	0.0061	0.0036		0.0016		0.0016	
353							0.0021
355					0.0025		
359			0.0029				
368	0.0040						
DD 420	0.0098	0.0001		0.0014		0.0010	
426							0.0018
427					0.0039		
429			0.0037				
430	0.0082						
DD 437	0.0091	0.0022		0.0016		0.0006	
438							0.0024
441					0.0029		
442			0.0038				
443	0.0097						
E 204	0.0015	0.0012		0.0010		0.0001	
215							0.0002
224					0.0004		
230			0.0010				
231	0.0011						
E 244	0.0012	0.0010		0.0011		0.0001	
245							0.0002
259					0.0004		
270			0.0010				
273	0.0012						
EE 278	0.0081	0.0013		0.0018		0.0002	
284							0.0003
294					0.0010		
295			0.0010				
297	0.0018						

(Continued)

APPENDIX 6 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 301	0.0015	0.0018		0.0012		0.0001	
310							0.0003
313					0.0010		
330			0.0001				
327	0.0016						
I 118	0.0198	0.0418		0.0210		0.0376	
121							0.0111
125					0.0526		
126			0.0230				
129	0.0181						
I 131	0.0242	0.0239		0.0388		0.0310	
133							0.0151
134					0.0359		
135			0.0395				
138	0.0153						
II 143	0.0098	0.0285		0.0252		0.0473	
145							0.0236
146					0.0221		
148			0.0251				
149	0.0128						
II 151	0.0064	0.0436		0.0139		0.0318	
152							0.0217
154					0.0267		
155			0.0189				
156	0.0099						
TI 129	0.1789	0.0314		0.0148		0.0049	
147							0.0160
150					0.0253		
152			0.0336				
159	0.1409						
TI 161	0.1437	0.0307		0.0190		0.0082	
169							0.0186
180					0.0303		
185			0.0438				
189	0.1223						

(Continued)

APPENDIX 6 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TTi 193	0.1002	0.0350		0.0319		0.0110	
196							0.0213
200							
202			0.0442				
205	0.0982						
TTi 208	0.0954	0.0359		0.0370		0.0171	
214							0.0278
217					0.0451		
218			0.0456				
230	0.1303						
TA 58	0.0000	0.0000		0.0000		0.0001	
63							0.0000
65					0.0000		
67			0.0000				
68	0.0000						
TA 24	0.0000	0.0000		0.0000		0.0001	
29							0.0000
30					0.0000		
42			0.0000				
73	0.0000						

D: Unwelded stainless steel type 347.
 DD: Welded stainless steel type 347.
 E: Unwelded stainless steel type Carpenter-20.
 EE: Welded stainless steel type Carpenter-20.
 I: Unwelded Monel.
 II: Welded Monel.
 Ta: Unwelded Tantalum.
 Ti: Unwelded Titanium 55A.
 TTi: Welded Titanium 55A.

APPENDIX 7

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS METALS AND ALLOYS IN BOILING 0.003M PERIODIC ACID IN 0.05M NITRIC ACID

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 314	0.0001	0.0001		0.0000		0.0000	
336							0.0000
344					0.0001		
367			0.0000				
372	0.0000						
D 302	0.0000	0.0001		0.0000		0.0000	
324							0.0000
337					0.0001		
354			0.0004				
357	0.0000						
DD 386	0.0001	0.0002		0.0000		0.0000	
395							0.0000
400					0.0001		
411			0.0001				
416	0.0001						
DD 392	0.0001	0.0002		0.0000		0.0000	
393							0.0000
405					0.0001		
408			0.0001				
421	0.0001						
E 216	0.0000	0.0001		0.0000		0.0000	
243							0.0000
260					0.0000		
268			0.0001				
276	0.0000						
E 225	0.0000	0.0001		0.0000		0.0000	
229							0.0000
242					0.0000		
257			0.0001				
262	0.0000						
EE 277	0.0000	0.0001		0.0000		0.0000	
279							0.0000
291					0.0001		
321			0.0001				
329	0.0000						

(Continued)

APPENDIX 7 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 287	0.0000	0.0001		0.0000		0.0000	
289							0.0000
333					0.0000		
335			0.0001				
346	0.0000						
I 74	0.0082	0.0047		0.0032		0.0033	
83							0.0031
96					0.0039		
97			0.0047				
110	0.0106						
I 70	0.0064	0.0037		0.0031		0.0045	
90							0.0039
101					0.0041		
104			0.0049				
107	0.0099						
II 118	0.0086	0.0046		0.0073		0.0038	
119							0.0035
120					0.0051		
121			0.0041				
122	0.0042						
II 115	0.0026	0.0045		0.0041		0.0036	
132							0.0035
136					0.0042		
138			0.0045				
146	0.0091						
TA 31	0.0000	0.0000		0.0000		0.0000	
33							0.0000
45					0.0000		
46			0.0000				
49	0.0000						
TA 19	0.0000	0.0000		0.0000		0.0000	
20							0.0000
32					0.0000		
44			0.0000				
48	0.0000						
TI 126	0.0000	0.0000		0.0000		0.0000	
127							0.0000
128					0.0000		
139			0.0000				
143	0.0000						

(Continued)

APPENDIX 7 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TI 124	0.0000	0.0000		0.0000		0.0000	
135							0.0000
141					0.0000		
149			0.0000				
170	0.0000						
TTI 222	0.0000	0.0000		0.0000		0.0000	
231							0.0000
245					0.0000		
255			0.0000				
256	0.0001						
TTI 236	0.0000	0.0000		0.0000		0.0000	
242							0.0000
246					0.0000		
262			0.0000				
265	0.0000						

D: Unwelded stainless steel type 347.
 DD: Welded stainless steel type 347.
 E: Unwelded stainless steel type Carpenter-20.
 EE: Welded stainless steel type Carpenter-20.
 I: Unwelded Monel.
 II: Welded Monel.
 Ta: Unwelded Tantalum.
 Ti: Unwelded Titanium 55A.
 TTI: Welded Titanium 55A.

APPENDIX 8

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS METALS AND ALLOYS IN 3% SODIUM FLUORIDE-20% NITRIC ACID AT 144°F ± 10°F

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 307	0.0489	0.0234		0.0141		0.0070	
333							0.0106
350					0.0183		
351			0.0203				
365	0.0441						
D 304	0.0403	0.0284		0.0186		0.0070	
309							0.0131
356					0.0185		
361			0.0235				
364	0.0508						
DD 396	0.0893	0.0116		0.0076		0.0038	
409							0.0073
412					0.0102		
431			0.0126				
432	0.0287						
DD 375	0.0246	0.0104		0.0074		0.0043	
398							0.0071
399					0.0103		
401			0.0112				
436	0.0264						
E 217	0.0151	0.0055		0.0041		0.0021	
221							0.0030
228					0.0045		
234			0.0055				
251	0.0130						
E 218	0.0112	0.0031		0.0024		0.0017	
227							0.0025
233					0.0030		
258			0.0042				
275	0.0118						

(Continued)

APPENDIX 8 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 285	0.0108	0.0044		0.0029		0.0015	
306							0.0042
308					0.0042		
309			0.0048				
325	0.0100						
EE 303	0.0100	0.0034		0.0029		0.0014	
315							0.0025
317					0.0036		
319			0.0045				
326	0.0102						
I 75	0.3773	0.1715		0.0251			
82							
93					0.1010	Rate 300 m/m dissolved	
100			0.1565				
105	0.3289						
I 116	0.3482	0.1922		0.0888			
117							
119					0.1240	Rate 300 m/m dissolved	
120			0.1618				
139	0.3492						
II 124	0.3451	0.1571		0.0974			
126							
139					0.1309	Rate 300 m/m dissolved	
147			0.1638				
160	0.3281						
II 142	0.3517	0.1631		0.0865			
144							
147					0.1295	Rate 300 m/m dissolved	
150			0.1675				
153	0.3307						
TA 23	0.0107	0.0123		0.0037		0.0012	
37							0.0032
57					0.0063		
70			0.0070				
71	0.0101						

(Continued)

APPENDIX 8 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TA 21	0.0169	0.0116		0.0047		0.0010	
22							0.0029
52					0.0059		
55			0.0054				
60	0.0162						
TI 118	0.2173	0.0801		0.0432		0.0257	
125							0.0477
145					0.0530		
146			0.1012				
151	0.2251						
TI 156	0.2586	0.0785		0.0377		0.0306	
162							0.0450
173					0.0788		
176			0.0902				
181	0.2652						
TTI 209	0.1796	0.1168		0.0365		0.0226	
212							0.0528
223					0.0685		
226			0.1069				
251	0.1896						
TTI 203	0.1784	0.0973		0.0521		0.0364	
211							0.0530
213					0.0699		
225			0.0805				
253	0.1652						

D: Unwelded stainless steel type 347.
 DD: Welded stainless steel type 347.
 E: Unwelded stainless steel type Carpenter-20.
 EE: Welded stainless steel type Carpenter-20.
 I: Unwelded Monel.
 II: Welded Monel.
 Ta: Unwelded Tantalum.
 Ti: Unwelded Titanium.
 TTi: Welded Titanium 55A.

(Continued)

APPENDIX 9

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS METALS AND ALLOYS IN BOILING 0.25M PHOSPHORIC ACID

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 305	0.0001	0.0000		0.0000		0.0000	
322							0.0000
460					0.0000		
461			0.0000				
462	0.0001						
D 463	0.0001	0.0000		0.0000		0.0000	
464							0.0000
465					0.0000		
466			0.0000				
467	0.0001						
DD 279	0.0002	0.0000		0.0000		0.0000	
280							0.0000
281					0.0000		
283			0.0000				
284	0.0002						
DD 285	0.0002	0.0002		0.0000		0.0000	
286							0.0000
287					0.0000		
289			0.0000				
280	0.0001						
E 363	0.0001	0.0000		0.0002		0.0000	
364							0.0000
365					0.0006		
366			0.0000				
367	0.0001						
E 368	0.0001	0.0000		0.0001		0.0000	
369							0.0000
370					0.0003		
371			0.0000				
372	0.0001						
EE 353	0.0001	0.0006		0.0000		0.0000	
354							0.0000
355					0.0000		
356			0.0000				
357	0.0001						

(Continued)

APPENDIX 9 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 358	0.0001	0.0000		0.0000		0.0000	
359							0.0000
360					0.0000		
361			0.0000				
362	0.0001						
I 122	0.0102	0.0210		0.0129		0.0226	
123							0.0300
127					0.0131		
128			0.0200				
130	0.0100						
I 132	0.0200	0.0220		0.0124		0.0200	
136							0.0300
137					0.0138		
140			0.0200				
141	0.0200						
II 166	0.0020	0.0010		0.0012		0.100	
167							0.0200
168					0.0012		
169			0.0014				
170	0.0030						
II 171	0.0020	0.0011		0.0012		0.0100	
172							0.0160
173					0.0014		
174			0.0012				
176	0.0020						
Ta 76	0.0000	0.0000		0.0000		0.0000	
77							0.0000
78					0.0000		
79			0.0000				
80	0.0000						
Ta 81	0.0000	0.0000		0.0000		0.0000	
82							0.0000
83					0.0000		
84			0.0000				
85	0.0000						

(Continued)

APPENDIX 9 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TI 158	0.0031	0.0154		0.0043		0.0050	
168							0.0047
186					0.0040		
278			0.0040				
279	0.0039						
TI 280	0.0074	0.0100		0.0050		0.0054	
281							0.0054
282					0.0050		
283			0.0044				
284	0.0074						
TTI 273	0.0000	0.0216		0.0067		0.0057	
274							0.0079
275					0.0076		
276			0.0077				
277	0.0000						
TTI 191	0.0000	0.0000		0.0000		0.0000	
195							0.0000
197					0.0000		
206			0.0000				
215	0.0000						

D: Unwelded stainless steel type 347.

DD: Welded stainless steel type 347.

E: Unwelded stainless steel type Carpenter-20.

EE: Welded stainless steel type Carpenter-20.

I: Unwelded Monel.

II: Welded Monel.

Ta: Unwelded Tantalum.

Ti: Unwelded Titanium 55A.

TTi: Welded Titanium 55A.

APPENDIX 10

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS METALS AND ALLOYS IN BOILING TURCO 4501

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 517	0.0001	0.0002		0.0001		0.0002	
518							0.0002
519					0.0001		
520			0.0001				
521	0.0001						
D 522	0.0001	0.0001		0.0001		0.0002	
523							0.0001
524					0.0000		
525			0.0001				
526	0.0001						
DD 537	0.0000	0.0000		0.0000		0.0001	
538							0.0000
539					0.0000		
540			0.0000				
541	0.0000						
DD 542	0.0000	0.0000		0.0000		0.0001	
543							0.0000
544					0.0000		
545			0.0001				
546	0.0000						
E 431	0.0000	0.0000		0.0000		0.0000	
432							0.0000
433					0.0000		
434			0.0000				
435	0.0000						
E 436	0.0000	0.0001		0.0000		0.0000	
437							0.0000
438					0.0000		
439			0.0000				
440	0.0000						
EE 451	0.0000	0.0000		0.0000		0.0000	
452							0.0000
453					0.0000		
454			0.0000				
455	0.0000						

(Continued)

APPENDIX 10 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 456	0.0000	0.0000		0.0000		0.0000	
457							0.0000
458					0.0000		
459			0.0000				
460	0.0000						
I 209	0.0000	0.0001		0.0001		0.0002	
210							0.0001
211					0.0000		
212			0.0002				
213	0.0001						
I 214	0.0001	0.0003		0.0001		0.0002	
215							0.0002
216					0.0001		
217			0.0001				
218	0.0001						
II 189	0.0002	0.0001		0.0001		0.0002	
190							0.0002
191					0.0001		
192			0.0001				
193	0.0002						
II 194	0.0002	0.0001		0.0001		0.0002	
195							0.0001
196					0.0001		
197			0.0001				
198	0.0002						
TI 320	0.0409	0.0654		0.0248		0.0156	
321							0.0161
322					0.0147		
323			0.0304				
324	0.0375						
TI 325	0.0415	0.0472		0.0291		0.0100	
326							0.0116
327					0.0162		
328			0.0269				
329	0.0398						

(Continued)

APPENDIX 10 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TTI 342	0.0273	0.0280		0.0227		0.0250	
343							0.0263
344					0.0238		
345			0.0288				
346	0.0381						
TTI 347	0.0303	0.0447		0.0227		0.0378	
348							0.0257
349					0.0187		
350			0.0201				
352	0.0390						

Specimen No.	2 Hr Period Rate
TA 118	0.1907
TA 119 Boiling	0.2065
TA 120	0.1159
TA 121 90°C	0.1065

D: Unwelded stainless steel type 347.
 DD: Welded stainless steel type 347.
 E: Unwelded stainless steel type Carpenter-20.
 EE: Welded stainless steel type Carpenter-20.
 I: Unwelded Monel.
 II: Welded Monel.
 Ta: Unwelded Tantalum.
 Ti: Unwelded Titanium 55A.
 TTi: Welded Titanium 55A.

APPENDIX 11

INCHES PENETRATION PER MONTH CORROSION RATE OF VARIOUS
METALS AND ALLOYS IN REFLUXED BOILING TURCO 4502

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
D 527	0.0000	0.0000		0.0000		0.0000	
528							0.0000
529					0.0000		
530			0.0000				
531	0.0000						
D 532	0.0000	0.0000		0.0000		0.0000	
533							0.0000
534					0.0000		
535			0.0000				
536	0.0000						
DD 547	0.0001	0.0000		0.0000		0.0000	
548							0.0000
549					0.0000		
550			0.0000				
551	0.0001						
DD 552	0.0001	0.0000		0.0000		0.0000	
553							0.0000
554					0.0000		
555			0.0000				
556	0.0001						
E 441	0.0000	0.0000		0.0000		0.0000	
442							0.0000
443					0.0000		
444			0.0000				
445	0.0000						
E 446	0.0000	0.0000		0.0000		0.0000	
447							0.0000
448					0.0000		
449			0.0000				
450	0.0000						
EE 461	0.0001	0.0000		0.0000		0.0000	
462							0.0000
463					0.0000		
464			0.0000				
465	0.0000						

(Continued)

APPENDIX 11 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
EE 466	0.0001	0.0000		0.0000		0.0000	
467							0.0000
468					0.0000		
469			0.0000				
470	0.0000						
I 220	0.0001	0.0000		0.0004		0.0001	
221							0.0004
222					0.0003		
225			0.0001				
226	0.0000						
I 227	0.0001	0.0000		0.0009		0.0002	
235							0.0001
236					0.0004		
237			0.0000				
238	0.0000						
II 200	0.0002	0.0000		0.0006		0.0001	
201							0.0002
202					0.0006		
205			0.0001				
206	0.0001						
II 207	0.0001	0.0000		0.0011		0.0002	
231							0.0001
232					0.0005		
233			0.0001				
234	0.0001						
TI 363	0.0003	0.0005		0.0004		0.0006	
364							0.0003
365					0.0001		
366			0.0001				
367	0.0003						
TI 368	0.0003	0.0004		0.0003		0.0006	
369							0.0001
370					0.0001		
371			0.0002				
372	0.0003						

(Continued)

APPENDIX 11 (Continued)

Specimen No.	24 Hr Period Rate	48 Hr Period Rate	72 Hr Term Rate	96 Hr Period Rate	168 Hr Term Rate	192 Hr Period Rate	360 Hr Term Rate
TTI 353	0.0004	0.0005		0.0004		0.0005	
354							0.0001
355					0.0001		
356			0.0003				
357	0.0005						
TTI 358	0.0005	0.0003		0.0003		0.0005	
359							0.0002
360					0.0002		
361			0.0002				
362	0.0007						

D: Unwelded stainless steel type 347.
 DD: Welded stainless steel type 347.
 E: Unwelded stainless steel type Carpenter-20.
 EE: Welded stainless steel type Carpenter-20.
 I: Unwelded Monel.
 II: Welded Monel.
 Ta: Unwelded Tantalum.
 Ti: Unwelded Titanium 55A.
 TTi: Welded Titanium 55A.

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION