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INTERIM REPORT II - PRODUCTION TEST IP-634
HALF-PLANT LOW pH TEST WATER TREATMENT AT
D REACTOR - PROCESS TUBE EXAMINATION RESULTS

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INTERIM REPORT II - PRODUCTION TEST IP-634
HAIF-PLANT LOW pH TEST WATER TREATMENT AT D REACTOR
PROCESS TUBE EXAMINATION RESULTS

By

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January 1, 1965

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INTERIM REPORT II - PRODUCTION TEST IP-634
HALF-PLANT LOW pH TEST WATER TREATMENT AT D REACTOR
PROCESS TUBE EXAMINATION RESULTS

INTRODUCTION

Two half-plant tests^{1,2} were conducted at D reactor to evaluate the effect of process water pH on corrosion of aluminum process tubes and fuel elements. The first test demonstrated that a reduction in coolant pH from 7.0 to 6.6 reduced aluminum corrosion by a factor of between 2 and 3^{3,4}. Fuel weight loss data from the second test indicated that reducing coolant pH from 6.6 to 6.2 would result in an additional factor of 2 to 3 reduction in aluminum corrosion⁵. This report presents the results of ex-reactor process tube measurements and thermocouple probe annular temperature measurements made on six new process tubes irradiated during the second test. These results were combined with similar data obtained from six new process tubes irradiated during the first test. The combined results were used to estimate the effect of 7.0, 6.6, and 6.2 pH on corrosion of aluminum process tubes.

SUMMARY

The ex-reactor top and bottom wall tube corrosion measurements and in-reactor thermocouple probe annular temperature measurements obtained from twelve aluminum process tubes were used to estimate the relationship between a relative tube corrosion correction factor (F) and process water acidity (pH). The prediction models that best explain the variation in the data are:

$$F = e^{2.74(\text{pH}-7)}, \text{ and}$$

$$F = e^{1.96(\text{pH}-7) - 1.20(\text{pH}-7)^2}$$

where: F = relative corrosion factor (F = 1.0 at pH = 7.0, by definition), and
pH = coolant water pH.

Both curves are shown in Figures 1 and 2. Statistically, there is no difference between the two models, i.e., both models explain the variation in the data equally well. Slightly different absolute values of F are obtained depending on which model is used.

This is shown in the following table where relative improvement factors and corresponding 0.95 confidence intervals for both prediction models are compared. An example of interpretation of the table is as follows:

Using the linear exponential form, our best estimate is that aluminum tube corrosion is less, by a factor of 3.0, at pH = 6.6 than at pH = 7.0. While the 3.0 is only an estimate and may not be the actual factor, we are 95 per cent confident that the actual value lies between 1.9 and 4.7.

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<u>Ratio</u>	<u>0.95 Confidence Interval Relative Improvement Factor</u>		
	<u>Lower Limit</u>	<u>Predicted Value</u>	<u>Upper Limit</u>
<u>$F = e^{2.74(pH-7)}$</u>			
$F_{7.0}/F_{6.6}$	1.9	3.0	4.7
$F_{6.6}/F_{6.2}$	1.9	3.0	4.7
$F_{7.0}/F_{6.2}$	5.3	8.9	15.1
<u>$F = e^{1.96(pH-7) - 1.20(pH-7)^2}$</u>			
$F_{7.0}/F_{6.6}$	1.7	2.65	4.2
$F_{6.6}/F_{6.2}$	2.5	3.9	6.1
$F_{7.0}/F_{6.2}$	6.1	10.3	17.5

The quadratic model agrees with simple arithmetic averages of data points in Figures 1 and 2 a little better than does the linear model; however, the linear model will extrapolate better and is easier to apply. Additional and better data would be required before further refinements could be made.

The large variation between process tubes operated at the same pH level could be reduced in future tests of this type by obtaining better annulus temperature measurements, increasing the number of monitor process tubes and by increasing the length of the test period, particularly at the lower levels of pH.

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FIGURE 1

EFFECT OF PROCESS WATER pH ON
ALUMINUM PROCESS TUBE CORROSION
(F vs pH)

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FIGURE 2

EFFECT OF PROCESS WATER pH ON
ALUMINUM PROCESS TUBE CORROSION
(LOG F vs pH)

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DISCUSSIONTest Details and Results

Two half-plant pH tests were made at D reactor to evaluate the effect of process water pH on corrosion of aluminum process tubes. During Test 1 (March-October, 1963)¹ half the reactor was operated at pH 7.0 and half at pH 6.6. During Test 2 (January-June, 1964)², half the reactor was operated at pH 6.6 and half at pH 6.2. Three new aluminum process tubes (total of six) were installed in each half of the reactor prior to starting a test. During the tests, annulus water temperatures in each process tube were monitored by means of downstream thermocouple probes. Six thermocouples were located in the annulus and one was located in the hole of the first downstream aluminum piece at the rear of each charge. Temperatures and process water pH were recorded daily. On completion of a test, the six process tubes were removed from the reactor and measured by Component Testing⁶⁻⁹. Table 1 is a summary of operating conditions and ex-reactor measurements obtained from Test 1 and previously reported in Reference 4.

TABLE 1SUMMARY OF PT-442 TEST RESULTS

Tube No.	Operating Days	Average pH	Average Top Annulus Temp., °C	Observed Top-Wall Corrosion (Mils)	Average Bottom Annulus Temp., °C	Observed Bottom Wall Corrosion (Mils)
2261	36	6.99	134.9	9	90.8	0
2161	138	6.92	123.0	21	95.4	4
2262	148	6.92	121.2	23	92.3	5
2278	36	6.59	132.5	3	90.2	0
2282	138	6.60	123.4	9	96.1	2
2283	148	6.60	124.0	13	94.3	1

Table 2 is a summary of operating conditions and ex-reactor measurements obtained from Test 2.

TABLE 2SUMMARY OF PT-634 TEST RESULTS

2879	128	6.61	128.7	10	101.8	2
1585	128	6.61	140.4	16	105.9	2
1786	128	6.61	129.2	19	95.5	2
2760	128	6.22	137.7	4	104.4	C
1360	128	6.22	130.7	9	111.2	2
2069	128	6.22	134.6	2	97.7	0

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Analysis of Test Data

The analysis of pH and tube corrosion data obtained from PT-442² and PT-634¹ was based on the following aluminum corrosion prediction model:

$$C_j = aF_j \sum_{i=1}^n e^{kT_{ij}}, \quad (1)$$

where: $i = 1, 2, 3, \dots, n$ operating days,

$j = 1, 2, 3, \dots, m$ process tubes,

T_{ij} = the annulus temperature ($^{\circ}\text{C}$) observed on the i^{th} day in the j^{th} process tube

$k = 0.057$ = a corrosion constant which weights the effect of temperature according to the hypothesis that aluminum corrosion approximately doubles for a 12°C increase in annulus water temperature,

F_j = pH correction factor for the j^{th} process tube,

$a = 1.3 \times 10^{-4}$, a constant selected to force the average value of F at 7.0 pH to be equal to 1.0,

C_j = the measured corrosion in the j^{th} process tube.

A pH correction factor (F_j) was calculated for each process tube by substituting observed data for temperature (T_{ij}) and measured corrosion (C_j) into the above equation and solving for F_j .

The daily temperature data were processed with the RCALC Fortran Code¹⁰. The code calculates R values* for each thermocouple position in the annulus and the hole. A harmonic curve was fitted to the six annular R values** to determine whether the thermocouple probes were properly oriented in the process tubes. Also, the curves were used to determine whether the individual thermocouples were correctly sensing annulus temperatures consistent with the respective tube and fuel geometries. The temperature data from Test 1 were very consistent. This was not the case in Test 2. Several of the thermocouple probes were improperly oriented in the process tube. Where this occurred, reasonable top and bottom annulus temperatures were obtained by interpolation of the harmonic curves. Also, mixer fuel elements were charged in the seven-from-rear position during the second metal cycle of Test 2, contrary to the test design. This may have resulted in downstream temperature measurements that were not representative of temperatures occurring at the point of maximum corrosion near the mixer location.

* R is the ratio of the temperature increase (or decrease) in the annulus (or hole) relative to the bulk temperature increase across the active section of the fuel charge.

** A plot of R value versus thermocouple position (in angular degrees) was made.

Examination of the ex-reactor corrosion measurements showed that the maximum top and bottom wall corrosion occurred downstream of the seven-from-rear mixer position. Therefore, it is possible that the thermocouple probe temperatures obtained during the second metal cycle were directly proportional to the annulus temperatures at the point of maximum top and bottom wall corrosion; and this was assumed to be the case. Another minor disadvantage of using mixer fuel elements is that less corrosion occurs in the top annulus, thereby reducing the test sensitivity, particularly at 6.2 pH.

The daily temperature data and pH data were then processed with a PANACEA Fortran code^{11,12}. The code calculates average top and bottom annulus temperature, average pH, and a top and bottom "temperature index" for each process tube. Temperature index is defined as follows:

$$I = \sum_{i=1}^n e^{kT_i}, \quad (2)$$

where: $i = 1, 2, \dots, n$ operating days

T_i = the annulus temperature on the i^{th} day,

$k = 0.057$, a corrosion constant which weights the effect of temperature according to the hypothesis that aluminum corrosion approximately doubles for a 12°C increase in annulus temperature.

The temperature index (I) is a measure of the cumulative or total temperature effect experienced by each process tube and provides a basis for adjusting the observed corrosion for the fact that the twelve tubes operated at different daily temperatures for different lengths of time. Temperature index is summarized in Table 3.

TABLE 3

SUMMARY OF TEMPERATURE INDEX (I) DATA

Tube No.	pH	Measured Top-Wall Corrosion (Mils)	Measured Bottom-Wall Corrosion (Mils)	Top-Wall Temperature Index ($I \times 10^{-5}$)	Bottom-Wall Temperature Index ($I \times 10^{-4}$)
2261	6.99	9	0	0.828	0.649
2161	6.92	21	4	1.937	3.258
2262	6.92	23	5	1.853	2.922
2278	6.59	3	0	0.707	0.622
2282	6.60	9	2	1.861	3.590
2283	6.60	13	1	2.003	3.302
2379	6.61	10	2	2.216	4.729
1585	6.61	16	2	4.358	5.910
1786	6.61	19	2	2.805	3.285
2760	6.22	4	0	4.225	5.432
1360	6.22	9	2	2.897	5.964
2069	6.22	2	0	3.863	3.632

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The effect of pH for each process tube was calculated as follows:

$$C = aFI, \quad (3)$$

where: C = the measured top or bottom wall corrosion in mils,

a = a constant,

F = pH correction factor, and

I = temperature index for top or bottom annulus.

The pH correction factor (F) is obtained by substituting in equation (3) the appropriate values of measured corrosion (C) and temperature index (I) from Table 3, and solving for F. The constant (a) is arbitrary and can be assigned any value. In this case, (a) was assigned the value 1.3×10^{-4} because it forces F into the range between zero and one and makes F approximately equal to 1.0 at pH 7.0. Table 4 is a summary of the F values obtained using equation 3 and Table 3.

TABLE 4
SUMMARY OF pH CORRECTION FACTORS (F)

<u>Tube No.</u>	<u>pH</u>	<u>Top-Wall F</u>	<u>Bottom-Wall F</u>
2261	6.99	0.836	0.592
2161	6.92	0.834	0.944
2262	6.92	0.955	1.316
2278	6.59	0.326	0.618
2282	6.60	0.372	0.429
2283	6.60	0.499	0.232
2879	6.61	0.347	0.325
1585	6.61	0.283	0.260
1786	6.61	0.521	0.468
2760	6.22	0.0727	0.0708
1360	6.22	0.238	0.258
2069	6.22	0.0399	0.106

Where the ex-reactor corrosion measurement indicated zero corrosion in the bottom annulus of some tubes, 0.5 mils was used to calculate the F value. Otherwise, the F value would have been zero, which is an impossibility. Also, the accuracy of the measurements is probably no better than 2 mils.

A statistical test was made to see if the F values for the tubes (2278, 2282, 2283) operated at 6.6 during Test 1 could be combined with the tubes (2879, 1585, 1786) operated at 6.6 pH during Test 2. There was no significant difference, indicating the data from both tests could be combined.

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The bottom wall F values in Table 4 are in very good agreement with the top wall F values, even though very little corrosion occurred in the bottom annulus. Using the combined top and bottom wall F values observed at each of the three levels of pH, it was possible to obtain a good estimate of the variance of the data at (within) each level of pH. This was not the case when the bottom wall data were excluded. When the bottom wall data were included, the "within pH variance" was not constant for all levels of pH, i.e., the within pH variance decreased as the pH level decreased (Figure 1). By making a logarithmic transformation of the F values, the within pH variance was observed to be essentially constant over the range of pH (Figure 2). This indicates models of the form

$$F = e^A + BpH = e^{B(pH-K)}, \text{ where } A = -BK; \text{ or } F = e^{B(pH-K)} + C(pH-K)^2$$

best represent the relationship between pH correction factor (F) and pH. Accordingly, the GEORGE Fortran linear regression code was used to estimate the parameters A, B, and C in the above models.

The two models obtained were:

$$F = e^{2.74(pH-7)} \text{ and} \quad (4)$$

$$F = e^{1.96(pH-7)} - 1.20(pH-7)^2. \quad (5)$$

Statistically, there is no difference between the two models. Both models explain the variability in the data equally well. Slightly different absolute values of F are obtained using the two models. Table 5 is a summary of the improvement factors obtained with the linear model for each level of pH and their respective 0.95 confidence limits. Table 6 gives the same information for the quadratic model.

TABLE 5

ESTIMATES OF pH CORRECTION FACTORS
AND APPROXIMATE 0.95 CONFIDENCE
INTERVALS FOR THE PREDICTION MODEL

$$F = e^{2.74(pH-7)}$$

<u>pH Correction Factor (F)</u>	<u>0.95 Confidence Interval</u>		
	<u>Lower Limit</u>	<u>Predicted Value</u>	<u>Upper Limit</u>
F _{7.0}	0.69	1.0	1.45
F _{6.6}	0.257	0.335	0.435
F _{6.2}	0.077	0.112	0.163

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TABLE 5 (Continued)

Relative Improvement Factor

$F_{7.0}/F_{6.6}$	1.9	3.0	4.7
$F_{6.6}/F_{6.2}$	1.9	3.0	4.7
$F_{7.0}/F_{6.2}$	5.3	8.9	15.1

TABLE 6

ESTIMATES OF pH CORRECTION FACTORS
AND APPROXIMATE 0.95 CONFIDENCE
INTERVALS FOR THE PREDICTION MODEL
 $F = e^{1.96(pH-7) - 1.20(pH-7)^2}$

<u>pH Correction Factor (F)</u>	<u>0.95 Confidence Interval</u>		
	<u>Lower Limit</u>	<u>Predicted Value</u>	<u>Upper Limit</u>
$F_{7.0}$	0.69	1.0	1.45
$F_{6.6}$	0.290	0.377	0.490
$F_{6.2}$	0.067	0.097	0.140
<u>Relative Improvement Factor</u>			
$F_{7.0}/F_{6.6}$	1.7	2.65	4.2
$F_{6.6}/F_{6.2}$	2.5	3.9	6.1
$F_{7.0}/F_{6.2}$	6.1	10.3	17.5

CONCLUSIONS AND RECOMMENDATIONS

It was determined that the effect of coolant water acidity on aluminum process tube corrosion is described equally well by the following two models:

$$F = e^{2.74(pH-7)},$$

$$F = e^{1.96(pH-7) - 1.20(pH-7)^2}$$

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where: F = corrosion factor ($F = 1.0$ at $pH = 7$, by definition)

pH = coolant water pH .

Statistically, there is no difference between the two models. The quadratic model agrees with the simple arithmetic averages of the data points at each pH level a little better than does the linear model. The linear model will extrapolate better and is easier to apply. Also, the linear model is probably more satisfying from a theoretical standpoint.

Future tests of this type could be improved by concentrating on efforts that would reduce the variability of the data within pH levels. This might be done by:

1. Improving annulus temperature measurements by ensuring proper orientation of thermocouple probes;
2. Avoiding use of mixer fuel elements;
3. Conducting laboratory tests to better define relationship between annulus temperatures measured at the end of the fuel charge and those occurring at the point of maximum corrosion;
4. Increasing the number of process tubes used to monitor the effect; and
5. Increasing the length of the test period.

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