MARTIN NUCLEAR DIVISION

BALTIMORE 3, MARYLAND

QUARTERLY PROGRESS REPORT

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C. Eicheldinger
Project Manager

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FOREWORD

This quarterly report is submitted by the Nuclear Division of The Martin Company to the Nuclear Power Field Office, Engineer Research and Development Laboratories, U.S. Army Corps of Engineers, in compliance with Contract DA-44-009-ENG-3581. The report describes progress from April through June 1961 in the ANPP Corrosion Program.

CONTENTS

SUMMARY

The 2000-hour autoclave tests on nickel and inconel were completed. These were the last scheduled tests in the autoclave program.

The nickel coupons exposed in the autoclave containing 1000 ppm chloride and 15 ppm oxygen suffered pitting corrosion. Maximum pit depth was 1.5 mils.

An IBM-7090 program was written for the statistical analysis of the autoclave data. The data from the 200-hour tests has been analyzed.

Operating Time Efficiency (OTE) for the corrosion loop during the report period was 72%.

Miniature heat exchangers, MIN 10, 11, 15 and 16 were re-ensured from the loop after 3000 hours of test time. They were replaced by MIN 13 and 14 (Monel) and 18 and 19 (nickel). Testing of the monel vessels, MOD SX-4 (bimetal) and SX-7 (inconel) continued.

Post-test evaluation of MIN 10, 11, 15 and 16 was initiated. The inconel (10 and 11) vessels performed quite well with relatively little corrosion of the tubes. The bimetal (15 and 16) vessels suffered extensive corrosion of the carbon steel secondary surface of the tubing.

1. HEAT EXCHANGER CORROSION PROGRAM*

The objectives of the heat exchanger corrosion program are:

(1) The determination of the effect of secondary water conditions on heat exchanger life, using various exchanger materials. The most severe water conditions are limited to 1000-ppm chloride with air cover-gas and air-saturated water.

(2) The examination of the technique of test heat exchanger fabrication.

(3) The recommendation of materials and service conditions for operating units.

Model and miniature heat exchangers currently under test, fabrication or design are shown in Table 1.

A. AUTOCLAVE TESTS

1. Testing

The second series of 2000-hour autoclave tests was completed. The conditions of the three tests performed are summarized below:

Test No.	Material	Time (hr)	Oxygen (ppm)	Chloride (ppm)
1	Inconel	2000	1	10
2	Nickel	2000	15	1000
3	Inconel	2000	15	1000

pH adjusted to 10 with Na_3PO_4 in all tests.

Termination of the above tests completes the testing phase of the autoclave program on nickel and Monel. (For purposes of comparison, 2000-hour tests were performed on Inconel.) Weight-loss determination and statistical analysis of the test results are proceeding. See below.

Prior to autoclave tests on the Inconel specimens, tensile test data and stress versus deflection curves for the beam specimens were obtained. Tensile test data are shown in Table 2.

*J. McGrew, J. Mueller, M. Norin, E. Jules, T. Page

TABLE 1

Test Heat Exchanger Status

Heat Exchanger Type	Number	Tube Material	Tube Sheet Material	Overlay	Shell Material	Water Conditions	Status*
Steam Generator	MOD SG-1	304 SS	304 SS	308 SS	304 SS	1	Service--1927 hr (sectioned)
Superheater	MOD SH-1	304 SS	304 SS	308 SS	304 SS	1	Failed--1927 hr (sectioned)
Steam Generator	MOD SG-2	Bimetal[a]	Carbon Steel	308 SS	Carbon Steel	1 (3)	Service--5041 hr
Superheater	MOD SH-2	Bimetal[a]	Carbon Steel	308 SS	Carbon Steel	1 (3)	Service--5041 hr
Steam Generator	MOD SG-3	304 SS	304 SS	308 SS	304 SS	2	Fabrication complete
Superheater	MOD SH-3	304 SS	304 SS	308 SS	304 SS	2	Fabrication complete
Steam Generator	MOD SG-4	Bimetal[a]	Carbon Steel	308 SS	Carbon Steel	13	Under test--4159 hr
Superheater	MOD SH-4	Bimetal[a]	Carbon Steel	308 SS	Carbon Steel	13	Under test--4159 hr
Steam Generator	MOD SG-5	Croloy 16-1	A350LF-1	308 SS	Carbon Steel	3	Service--4253 hr
Superheater	MOD SH-5	Croloy 16-1	A350LF-1	308 SS	Carbon Steel	3	Service--4253 hr
Steam Generator	MOD SG-6	Inconel	AISI-1020 CS	Inco "A"	Carbon Steel	3	Service--3819 hr
Superheater	MOD SH-6	Inconel	AISI-1020 CS	Inco "A"	Carbon Steel	3	Service--3819 hr
Steam Generator	MOD SG-7	Inconel	AISI-1030 CS	Inco "A"	AISI-1030 CS	12	Under test--4016 hr
Superheater	MOD SH-7	Inconel	AISI-1030 CS	Inco "A"	AISI-1030 CS	12	Under test-- 4016 hr
Steam Generator	MOD SG-8	Monel or Nickel	SA-105-II	Monel or Nickel	SA-212-B		In design
Superheater	MOD SH-8	Monel or Nickel	SA-105-II	Monel or Nickel	SA-212-B		In design
Miniature	MIN-1	304 SS	304 SS	308 SS	304 SS	4	Failed--42 hr
Miniature	MIN-2	304 SS	304 SS	308 SS	304 SS	5	Service--1929 hr
Miniature	MIN-3	304 SS	304 SS	308 SS	304 SS	6	Failed--1085 hr
Miniature	MIN-4	430-M (B W)	15 Cr, 0.17 Ni	430-M (B W)	16 Cr, 0.26 Ni	7	Failed--2578 hr
Miniature	MIN-5	430-M (B W)	15 Cr, 0.17 Ni	430-M (B W)	16 Cr, 0.26 Ni	8	Failed--941 hr
Miniature	MIN-6	430-M (B W)	15 Cr, 0.17 Ni	430-M (B W)	16 Cr, 0.26 Ni	9	Service--2767 hr
Miniature	MIN-7	Bimetal[a]	Carbon Steel	308 SS	Carbon Steel	4	Service--3256 hr
Miniature	MIN-8	Inconel	Inconel	Inco "A"	Inconel	10	Service--5296 hr
Miniature	MIN-9	Inconel	Inconel	Inco "A"	Inconel	11	Service--2631 hr
Miniature	MIN-10	Inconel	Inconel	Inco "A"	AISI-1030 CS	14	Service--3044 hr
Miniature	MIN-11	Inconel	Inconel	Inco "A"	AISI-1030 CS	15	Service--3023 hr

Miniature							
Miniature	MIN-3	304 SS	304 SS	308 SS	304 SS	6	Failed--1085 hr
Miniature	MIN-4	430-M (B W)	15 Cr, 0.17 Ni	430-M (B W)	16 Cr, 0.26 Ni	7	Failed--2578 hr
Miniature	MIN-5	430-M (B W)	15 Cr, 0.17 Ni	430-M (B W)	16 Cr, 0.26 Ni	8	Failed--941 hr
Miniature	MIN-6	430-M (B W)	15 Cr, 0.17 Ni	430-M (B W)	16 Cr, 0.26 Ni	9	Service--2767 hr
Miniature	MIN-7	Bimetal[(a)]	Carbon Steel	308 SS	Carbon Steel	4	Service--3256 hr
Miniature	MIN-8	Inconel	Inconel	Inco "A"	Inconel	10	Service--5296 hr
Miniature	MIN-9	Inconel	Inconel	Inco "A"	Inconel	11	Service--2631 hr
Miniature	MIN-10	Inconel	Inconel	Inco "A"	AISI-1030 CS	14	Service--3044 hr
Miniature	MIN-11	Inconel	Inconel	Inco "A"	AISI-1030 CS	15	Service--3023 hr
Miniature	MIN-12	Monel	AISI-1020 CS	Nickel	AISI-1030 CS	2	Under test--668 hr
Miniature	MIN-13	Monel	AISI-1020 CS	Nickel	AISI-1030 CS	15	Under test--707 hr
Miniature	MIN-14	Monel	AISI-1020 CS	Nickel	AISI-1030 CS	12	Service--3018 hr
Miniature	MIN-15	Bimetal	AISI-1020 CS	---	AISI-1030 CS	16	Service--3035 hr
Miniature	MIN-16	Bimetal	AISI-1020 CS	---	AISI-1030 CS	16	Under test--661 hr
Miniature	MIN-17	Nickel	AISI-1020 CS	Nickel	AISI-1030 CS	2	Under test--651 hr
Miniature	MIN-18	Nickel	AISI-1020 CS	Nickel	AISI-1030 CS	15	
Miniature	MIN-19	Nickel	AISI-1020 CS	Nickel	AISI-1030 CS	12	

NOTES:

(a) Primary Side--Type 304 SS

Secondary Side--Carbon Steel

1. Chloride--40-50 ppm
   Phosphate--40-80 ppm
   Sulfite--6-10 ppm
   pH--10.5-10.8
2. To be determined
3. Cl--800-1000 ppm
   O_2--none (controlled with sodium sulfite)
   pH--8.3-9.5 with PO_4
4. Chloride--991.6 ppm
   Phosphate--61 ppm
   O_2--40 ppm
   pH--10.5
5. Chloride--995.3 ppm
   Phosphate--58 ppm
   O_2--7.89 ppm
   pH--10.5
6. Chloride--108.5 ppm
   Phosphate--none
   O_2--7.85 ppm
   pH--11.5
7. Chloride--800 ppm
   Phosphate--50 ppm
   O_2--21% by volume
   pH--10.5
8. Chloride--800 ppm
   Phosphate--none
   O_2--21% by volume
   pH--10.5
9. Chloride--400 ppm
   Phosphate--none
   O_2--10% by volume
   pH--6.5
10. Chloride--1000 ppm
    O_2--Air sat. water, air as cover gas
    pH--8.3-9.5 with PO_4 water treat
11. Chloride--1000 ppm
    O_2--Air sat. water, air as cover gas
    pH--8.3-9.5 with NaOH
    PO_4--none
12. Chloride--0.5 ppm max
    Phosphate--150 ppm
    Sulfite--10 ppm
    pH--10 to 10.5
13. Chloride--0.5 ppm max
    Sulfite--10 ppm
    Total Solids--200 ppm max
    pH--8.5 (PO_4)
14. Chloride--1000 ppm
    pH--10 (NaOH)
    O_2--no treatment**
15. Chloride--1000 ppm
    pH--10 (33% Na_3PO_4 and 67% Na_2HPO_4)
    O_2--no treatment**
16. Chloride--800 ppm
    pH--10 (33% Na_3PO_4 and 67% Na_2HPO_4)
    O_2--no treatment**

*Operating times are cumulative to June 30, 1961.

**The secondary makeup tank will be maintained at 180° F, open to the atmosphere, which will maintain the oxygen concentration at somewhat less than 0.5 ppm.

Table 2

Summary of Tensile Test Results on Inconel Specimens
(average of 3 values)

Inconel	Yield Strength (psi)	90% of Yield Strength (psi)	Proportional Limit (psi)	Ultimate Tensile Strength (psi)	Modulus of Elasticity	Percent Elongation (2 in.)
Stress Relieved	166,600	149,940	131,600	169,600	29.6×10^6	29.6
Fully Annealed	32,860	29,574	23,600	93,200	26.7×10^6	26.7

2. Weight Loss Determination--2000-Hour Nickel Tests

The coupons from the 2000-hour nickel tests were descaled, using the sulfamic acid process, and weight losses were determined. Table 3 is a reproduction of Table 2 from the previous quarterly report with the 2000-hour nickel data added. Figures 1 and 2 are reproductions of Fig. 4 and 5 from the previous report; the high chloride-high oxygen curves for nickel have been added. These curves show weight loss as a function of time for the two metals under various conditions. As noted previously for similar conditions, nickel exhibits a lower weight loss from corrosion than Monel. However, the general behavior is similar, that is, at the low oxygen-low chloride condition, corrosion n proceeds almost linearly with time with no indication of a saturation level after 2000 hours of test time. For the high chloride-high oxygen environment, a protective film appears to form rapidly, and further corrosion progresses at a very slow rate. The result is a significantly greater weight loss due to corrosion after 2000 hours in the milder environment.

No cracking occured in any of the nickel coupons. However, varying degrees of pitting did occur on coupons exposed in the autoclaves containing 15 ppm oxygen and 1000 ppm chloride. All twelve vapor phase coupons and nine of twelve liquid phase coupons suffered pitting. A photograph of a typical pit is shown in Fig. 3. The pits appeared quite different from the Monel coupons which were exposed for 2000 hours to the same environment. The pits in the nickel have a diameter-to-depth ratio of about two; the area adjacent to the pits is uncorroded. The pits in the Monel coupons have about the same diameter-to-depth ratio, but a rather large corroded area, approximately 100 mils in diameter, surrounds each pit. Thus, the pits in the Monel coupons were readily apparent to the naked eye while magnification was required to detect pits in the nickel coupons. Also the nickel sustained a much larger number of pits. The maximum pit depth found on any nickel coupons was 1.5 mils.

3. Analysis of Autoclave Test Data*

The general method chosen for analyzing the autoclave coupon weight-loss data was an analysis of variance. This method is useful in separating and evaluating the effects of the independent, or controlled variables on the observed levels of corrosion. Since the method requires the summing of a large number of data in many different ways, a program for performing these operations on the IBM-7090, was written. The use of the machine made it possible to avoid human mistakes while significantly shortening the required span time.

* S. Frank

Table 3

Summary of Weight Loss Data From Autoclave Tests

			Monel						Nickel					
			10 ppm Chloride 1 ppm Oxygen			1000 ppm Chloride 15 ppm Oxygen			10 ppm Chloride 1 ppm Oxygen			1000 ppm Chloride 15 ppm Oxygen		
Hours			50	200	2000	50	200	2000	50	200	2000	50	200	2000
Annealed	S	V	0.0	1.4	8.8	1.1	6.0	7.1	1.1	0.4	6.5	2.5	2.3	4.8
		L	0.3	1.2	11.3	0.2	2.2	6.3	0.6	0.7	5.8	0.5	2.6	3.8
	U	V	0.1	1.1	10.8	1.8	6.0	8.5	0.9	0.5	4.9	2.5	1.5	5.8
		L	0.3	1.4	10.2	0.0	2.4	6.7	0.4	0.6	7.4	0.4	1.9	3.5
Stress-relieved	S	V	0.6	2.5	7.9	1.6	4.9	9.2	1.2	0.8	8.8	1.9	2.5	4.1
		L	0.3	1.6	8.2	0.8	2.1	5.0	0.5	0.9	6.8	0.6	2.7	7.6
	U	V	0.7	2.4	8.4	2.1	4.8	9.0	1.2	0.4	7.2	2.2	2.5	5.2
		L	0.3	1.5	6.7	0.7	1.9	7.6	0.6	0.8	6.0	1.2	1.3	4.3
Total			2.6	13.1	72.3	8.3	30.3	60.4	6.5	5.1	53.4	11.8	17.3	39.1

NOTE: Values are weight loss in milligrams--average of three specimens in all tests, pH adjusted to 10 with Na_3PO_4

S--Stressed to 90% at yield strength 0.02% offset

U--Unstressed

V--Vapor phase

L--Liquid phase

Curve No.	Symbol	Conditions	Curve No.	Symbol	Conditions
1	○	1 ppm oxygen 10 ppm chloride Vapor phase pH 10 (Na_3PO_4)	3	△	15 ppm oxygen 1000 ppm chloride Vapor phase pH 10 (Na_3PO_4)
2	□	1 ppm oxygen 10 ppm chloride Liquid phase pH 10 (Na_3PO_4)	4	◇	15 ppm oxygen 1000 ppm chloride Liquid phase pH 10 (Na_3PO_4)

Experimental points plotted are average values for twelve coupons exposed to conditions indicated in the legend.

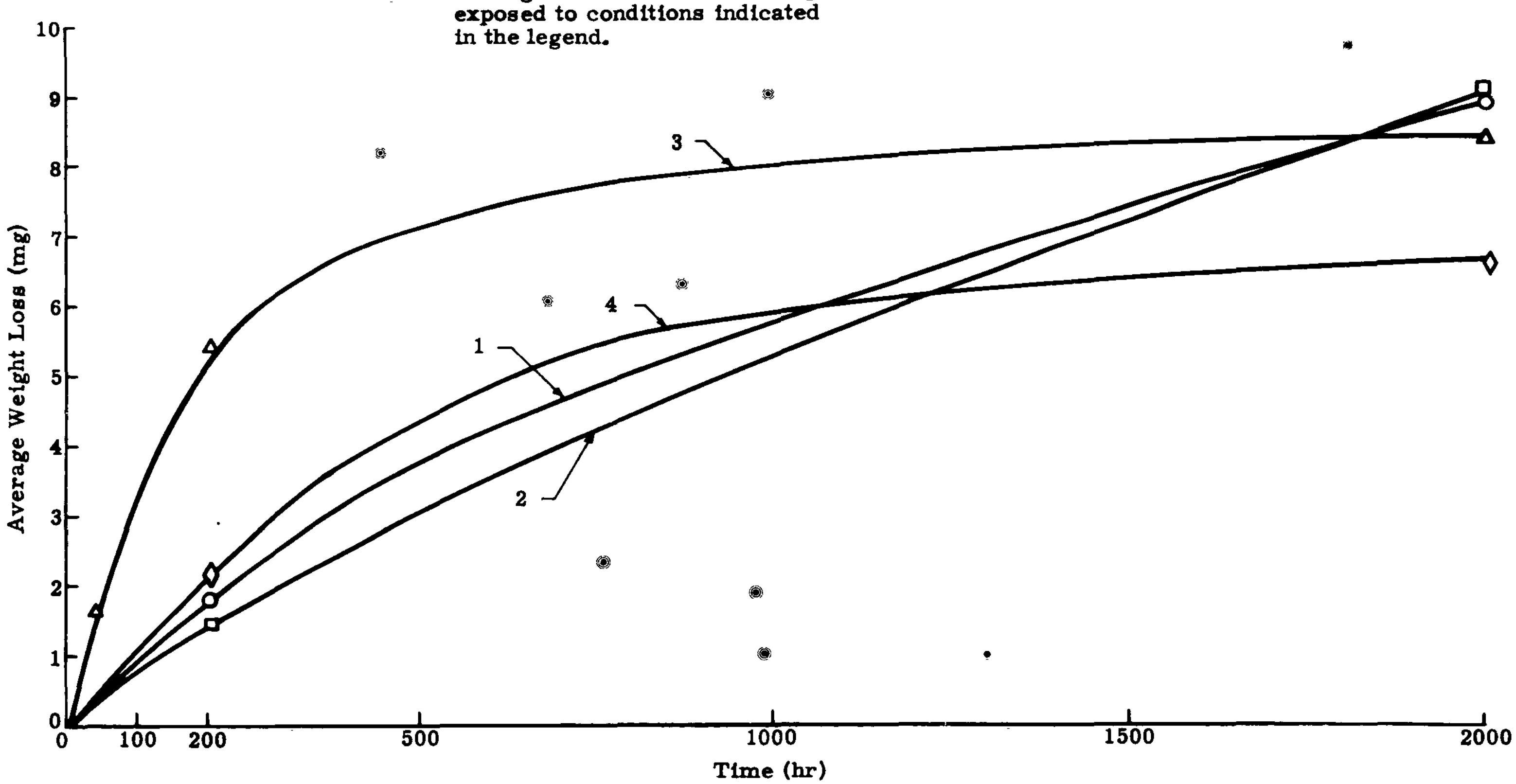


Fig. 1. Weight Loss as a Function of Time for Monel Coupons

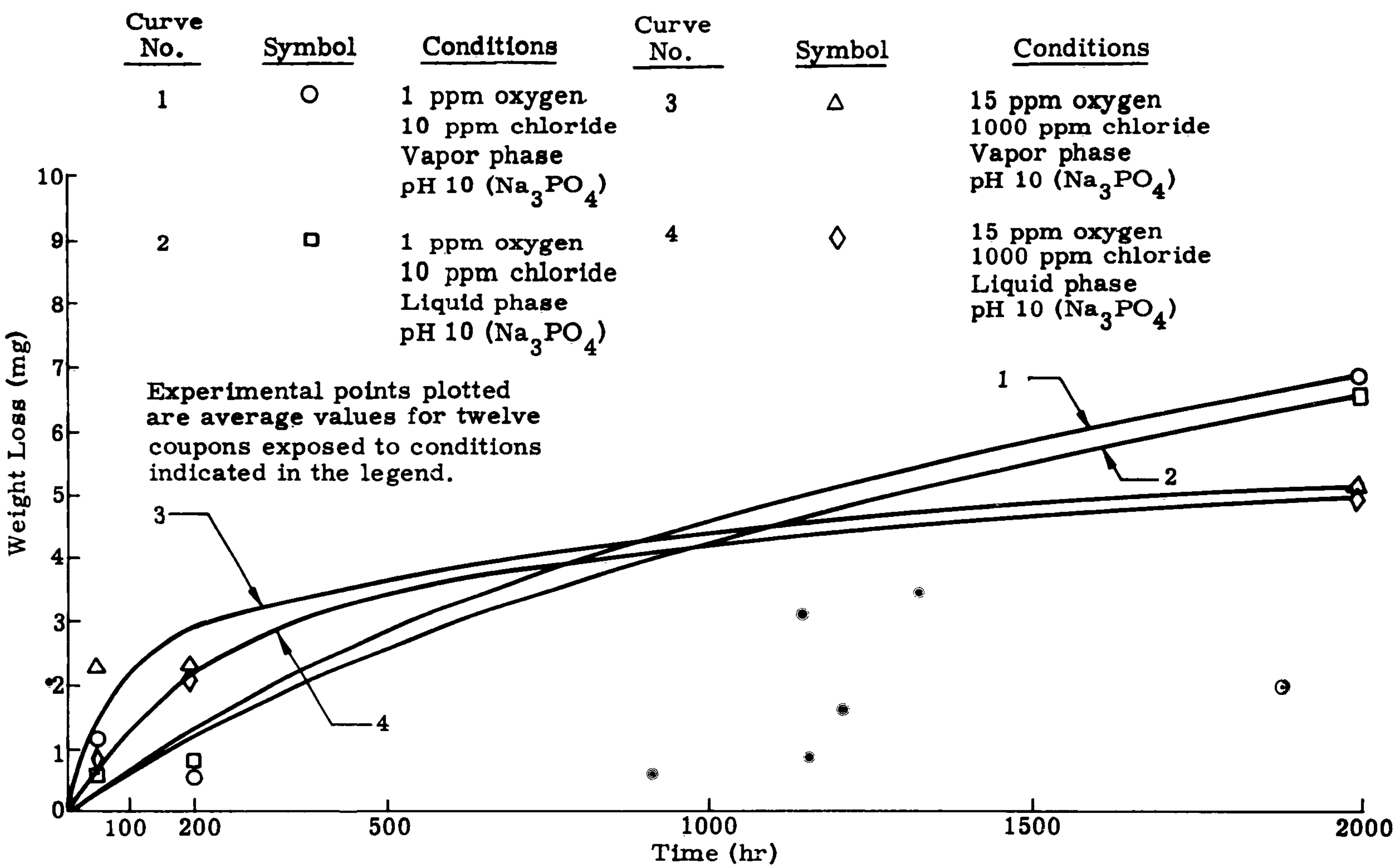
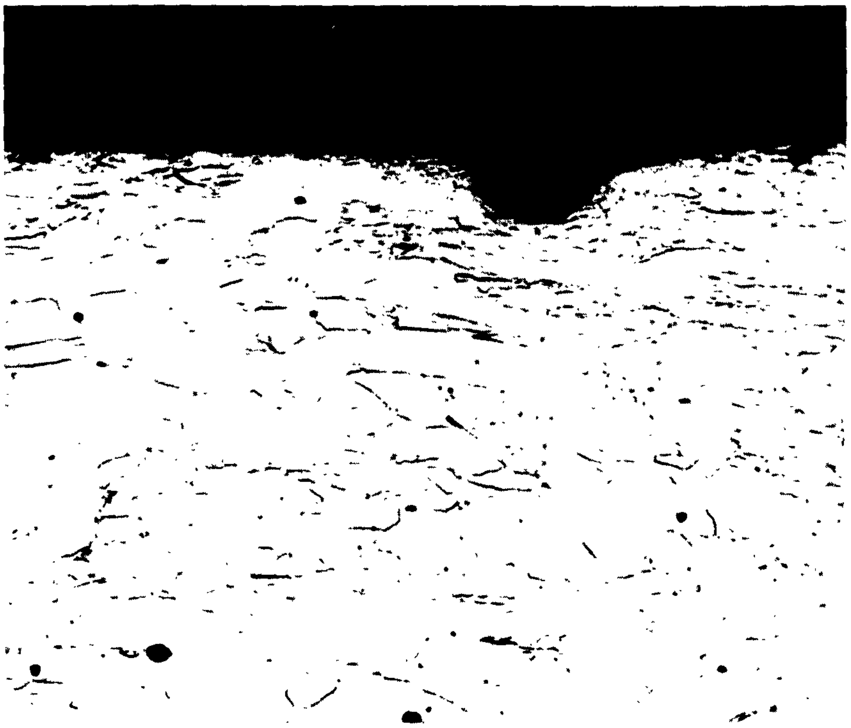


Fig. 2. Weight Loss as a Function of Time for Nickel Coupons



NL-7584 M-7156

Etchant: Acetic-nitric

Magn 750X 30 June 1961

Maximum penetration 0.00035"

Fig. 3. Photomicrograph Showing Pit in Nickel Coupon Exposed in a Water Phase of Autoclave Containing 15 ppm Oxygen and 1000 ppm Chloride for 2000 Hours

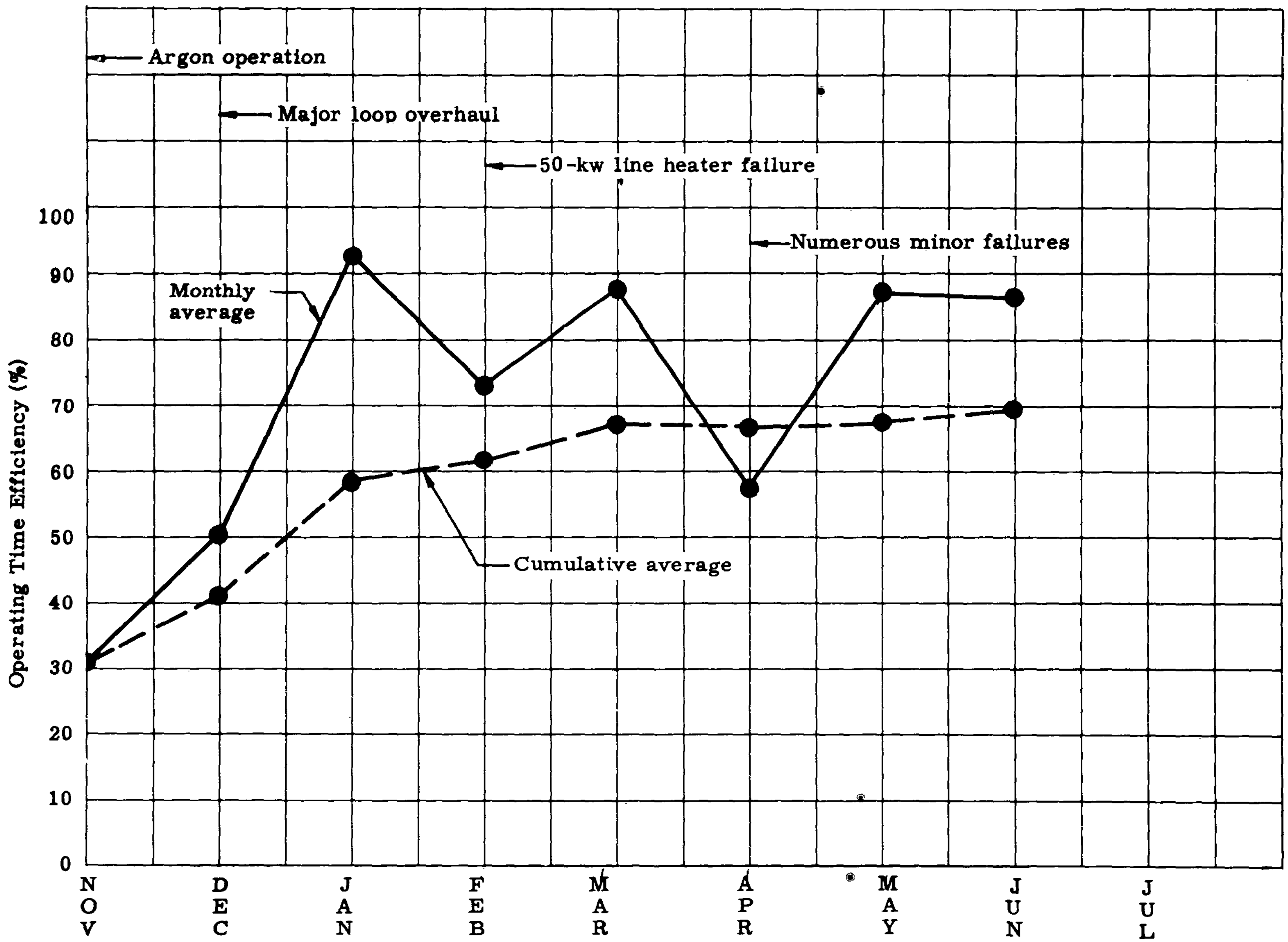
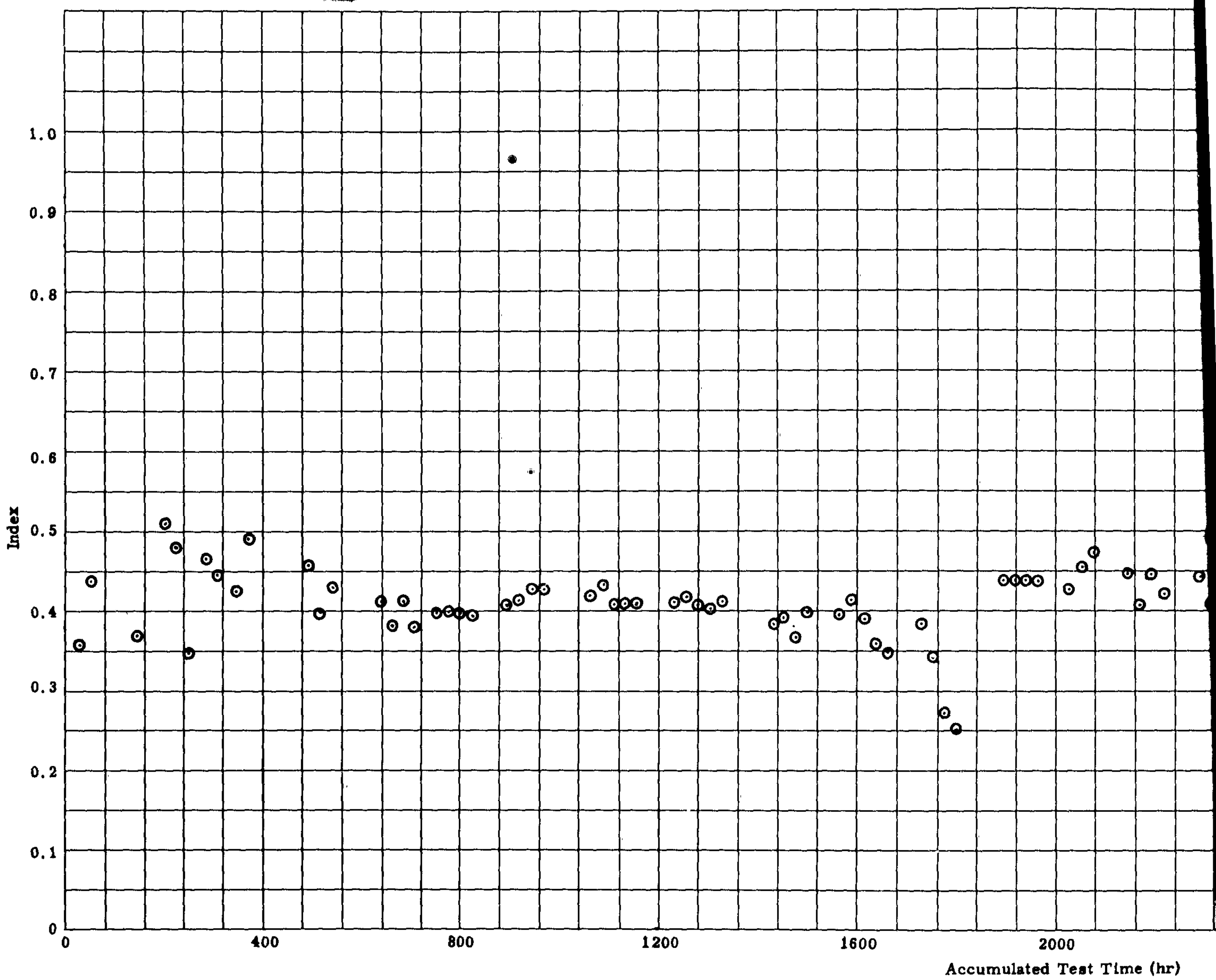


Fig. 4. Summary of Loop Operating Efficiency

1



1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
Index
0
400
800
1200
1600
2000
Accumulated Test Time (hr)

Fig. 5. Heat

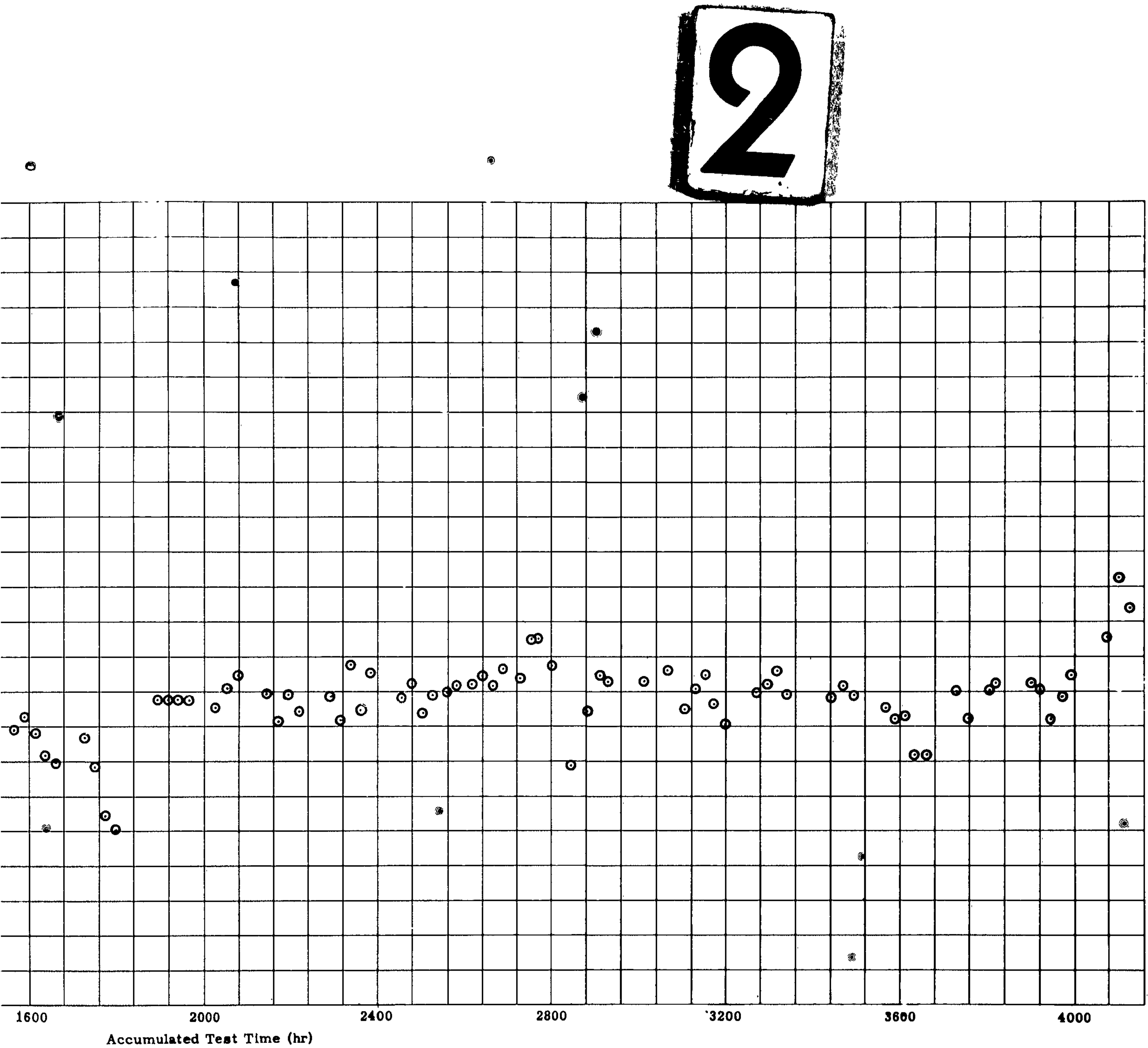


Fig. 5. Heat Transfer Index Versus Test Time; Bimetal Steam Generator (MOD SG-4)

A code was written and checked out for handling the 200-hour test results. The printout from this code gives the experimental error variance as well as the mean squares of the main effects and all two-factor interactions of the seven independent variables. The mean squares of the three-factor interactions of the oxygen and chloride concentrations with each of the other five independent variables are also presented. The code determines the significance of the effects and interactions by means of F-tests, and the results appear on the printout beside each mean square.

The test results are presented as average values of weight loss in milligrams in two-way tables between the independent variables. The test results are also given as six average values for all combinations of the four significant qualitative independent variables (the stress level was found to be insignificant). These six values at each of the 16 (2^4) combinations correspond to the results at the two levels of oxygen concentration and three levels of chloride concentration and can be plotted on graphs of corrosion level (weight loss) versus chloride concentration with oxygen as parameter. In fact, the code performed least squares fits for making such plots and tested the significance of the regression coefficients; this information also appears in the printout.

Analysis of the 200-hour autoclave test results was completed. The 50- and 2000-hour data will be analyzed early in the next quarter.

B. CORROSION LOOP OPERATION

Operation of the loop during the report period was virtually continuous; approximately 1750 hours of test time were logged. The test time accumulated for each vessel for each day during the report period is indicated in Tables 4, 5 and 6.

Operating Time Efficiency (OTE) for the loop during the report period was approximately 72%. OTE is defined as follows:

$$\text{OTE} = \frac{\text{Total hours of test time (all vessels)}}{\text{Total possible hours of test time (all vessels)}} \times 100\%$$

A plot of OTE as a function of time is shown in Fig. 4.

1. Installation of New Miniature Vessels

On May 8, 1961, the accumulated test time was slightly greater than the specified 3000 hours for miniature vessels, MIN 10, 11, 15 and 16. Consequently, these vessels were removed from the loop for post test evaluation. The miniature secondary system was cleaned with sulfamic acid to remove all traces of the previous environment. The acid was circulated through the system overnight at 120° F. The system was then thoroughly flushed with demineralized water and dried. The new test vessels, MIN 13 and 14 (Monel) and 18 and 19 (nickel) were subsequently installed in the loop and thermally insulated. Testing was started on May 26, 1961 except for MIN 13 which was started on May 29, 1961.

TABLE 4

Distribution of Test Time for Each Vessel
During the Month of April

Loop Vessel Designation	April						
							1
No. 1 Model							24.0
No. 2 Model							24.0
No. 1 Miniature							24.0
No. 2 Miniature							24.0
No. 3 Miniature							24.0
No. 4 Miniature							24.0
	2	3	4	5	6	7	8
No. 1 Model	0.0	22.0	24.0	24.0	24.0	20.5	0.0
No. 2 Model	0.0	22.0	24.0	24.0	24.0	20.5	0.0
No. 1 Miniature	0.0	22.0	24.0	24.0	24.0	20.5	0.0
No. 2 Miniature	0.0	22.0	24.0	24.0	24.0	20.5	0.0
No. 3 Miniature	0.0	22.0	24.0	24.0	24.0	20.5	0.0
No. 4 Miniature	0.0	22.0	24.0	24.0	24.0	20.5	0.0
	9	10	11	12	13	14	15
No. 1 Model	0.0	1.2	0.0	9.6	24.0	14.2	0.0
No. 2 Model	0.0	1.2	0.0	9.6	24.0	11.2	0.0
No. 1 Miniature	0.0	1.2	0.0	9.5	24.0	14.7	0.0
No. 2 Miniature	0.0	1.2	0.0	9.5	24.0	13.6	0.0
No. 3 Miniature	0.0	1.2	0.0	9.5	24.0	14.7	0.0
No. 4 Miniature	0.0	1.2	0.0	9.5	24.0	13.7	0.0
	16	17	18	19	20	21	22
No. 1 Model	0.0	21.8	24.0	24.0	24.0	19.5	1.5
No. 2 Model	0.0	21.8	24.0	24.0	24.0	3.0	0.0
No. 1 Miniature	0.0	23.0	24.0	24.0	24.0	21.2	4.5
No. 2 Miniature	0.0	23.0	24.0	24.0	24.0	21.5	4.0
No. 3 Miniature	0.0	23.0	24.0	24.0	24.0	21.2	4.3
No. 4 Miniature	0.0	23.0	24.0	24.0	24.0	21.5	4.2
	23	24	25	26	27	28	29
No. 1 Model	0.0	20.0	24.0	24.0	24.0	24.0	14.5
No. 2 Model	0.0	20.0	24.0	24.0	24.0	24.0	14.2
No. 1 Miniature	0.0	20.0	24.0	24.0	24.0	24.0	14.6
No. 2 Miniature	0.0	20.0	24.0	24.0	24.0	24.0	14.5
No. 3 Miniature	0.0	20.0	24.0	24.0	24.0	24.0	14.5
No. 4 Miniature	0.0	20.0	24.0	24.0	24.0	24.0	14.5
	30						
No. 1 Model	0.0	No. 1 Model - MOD SG-4 and SH-4 - 2845.8*					
No. 2 Model	0.0	No. 2 Model - MOD SG-7 and SH-7 - 2714.1*					
No. 1 Miniature	0.0	No. 1 Miniature - MIN 10 - 2872.2*					
No. 2 Miniature	0.0	No. 2 Miniature - MIN 15 - 2854.4*					
No. 3 Miniature	0.0	No. 3 Miniature - MIN 11 - 2850.9*					
No. 4 Miniature	0.0	No. 4 Miniature - MIN 16 - 2861.4*					

Times shown are number of hours of test time on vessel in 24-hour period starting at 8:30 on the morning of that day in which the time is shown.

*Total hours test time to April 30, 1961

TABLE 5

Distribution of Test Time for Each Vessel
During the Month of May

Loop Vessel Designation	May						
		1	2	3	4	5	6
No. 1 Model		21.2	24.0	24.0	24.0	24.0	24.0
No. 2 Model		21.5	24.0	24.0	24.0	24.0	24.0
No. 1 Miniature		20.5	24.0	24.0	24.0	24.0	24.0
No. 2 Miniature		20.7	24.0	24.0	24.0	20.5	24.0
No. 3 Miniature		20.5	24.0	24.0	24.0	24.0	24.0
No. 4 Miniature		20.7	24.0	24.0	24.0	24.0	24.0
	7	8	9	10	11	12	13
No. 1 Model	24.0	7.2	Primary Circulating Pump Overall		23.5	24.0	24.0
No. 2 Model	24.0	7.2			23.7	24.0	24.0
No. 1 Miniature	24.0	8.0					
No. 2 Miniature	24.0	8.2					
No. 3 Miniature	24.0	8.2					
No. 4 Miniature	24.0	8.2					
	14	15	16	17	18	19	20
No. 1 Model	24.0	24.0	24.0	24.0	24.0	24.0	23.1
No. 2 Model	24.0	24.0	24.0	24.0	24.0	24.0	23.2
No. 1 Miniature							
No. 2 Miniature							
No. 3 Miniature							
No. 4 Miniature							
	21	22	23	24	25	26	27
No. 1 Model	0.0	22.0	24.0	24.0	24.0	24.0	24.0
No. 2 Model	0.0	20.5	24.0	24.0	24.0	24.0	24.0
No. 1 Miniature						17.4	24.0
No. 2 Miniature							
No. 3 Miniature						17.4	24.0
No. 4 Miniature						17.4	24.0
	28	29	30	31			
No. 1 Model	11.3	19.5	24.0	24.0			
No. 2 Model	3.4	18.5	24.0	24.0			
No. 1 Miniature	5.8	19.0	24.0	24.0			
No. 2 Miniature		17.0	24.0	17.0			
No. 3 Miniature	5.8	19.0	24.0	24.0			
No. 4 Miniature	5.8	19.0	24.0	24.0			

No. 1 Model - MOD SG-4 and SH-4--3478.1 hr*
No. 2 Model - MOD SG-7 and SH-7--3336.1 hr*
No. 1 Miniature - MIN 10--3044.7 hr**--MIN 18--114.2 hr***
No. 2 Miniature - MIN 15--3018.8 hr**--MIN 13-- 58.0 hr***
No. 3 Miniature - MIN 11--3023.6 hr**--MIN 19--114.2 hr****
No. 4 Miniature - MIN 16--3035.3 hr**--MIN 14--114.2 hr***

Times shown are number of hours of test time on vessel in 24-hour period starting at 8:30 on the morning of that day in which the time is shown.

*Total hours test time to May 31, 1961.
**Total hours service time on vessel. Test terminated May 8, 1961.
***MIN 18, 19 and 14 in service May 26, 1961.
****MIN 13 in service May 29, 1961.

TABLE 6

Distribution of Test Time for Each Vessel
During the Month of June

Loop Vessel Designation	June						
					1	2	3
No. 1 Model					24.0	24.0	24.0
No. 2 Model					24.0	24.0	24.0
No. 1 Miniature					24.0	24.0	24.0
No. 2 Miniature					24.0	24.0	3.6
No. 3 Miniature					24.0	18.7	0.0
No. 4 Miniature					24.0	24.0	6.6
	4	5	6	7	8	9	10
No. 1 Model	24.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 2 Model	24.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 1 Miniature	0.0	14.0	15.1	20.5	24.0	24.0	0.0
No. 2 Miniature	0.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 3 Miniature	0.0	24.0	24.0	22.5	24.0	24.0	2.5
No. 4 Miniature	0.0	24.0	24.0	24.0	24.0	24.0	14.7
	11	12	13	14	15	16	17
No. 1 Model	24.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 2 Model	24.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 1 Miniature	0.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 2 Miniature	0.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 3 Miniature	0.0	24.0	24.0	21.8	24.0	24.0	24.0
No. 4 Miniature	0.0	24.0	24.0	24.0	24.0	24.0	24.0
	18	19	20	21	22	23	24
No. 1 Model	24.0	24.0	24.0	24.0	24.0	24.0	24.0
No. 2 Model	24.0	24.0	24.0	24.0	22.9	24.0	24.0
No. 1 Miniature	24.0	24.0	21.2	20.0	24.0	24.0	4.0
No. 2 Miniature	24.0	24.0	22.2	24.0	24.0	24.0	24.0
No. 3 Miniature	24.0	24.0	14.4	24.0	24.0	24.0	5.7
No. 4 Miniature	24.0	24.0	21.9	24.0	24.0	24.0	16.4
	25	26	27	28	29	30	
No. 1 Model	13.6	Primary	20.0	24.0	24.0	24.0	
No. 2 Model	13.6	Circulating	20.0	24.0	24.0	24.0	
No. 1 Miniature	0.0	Pump	20.0	24.0	24.0	24.0	
No. 2 Miniature	14.0	Overhaul	20.0	24.0	24.0	24.0	
No. 3 Miniature	0.0		20.0	24.0	24.0	24.0	
No. 4 Miniature	10.0		20.0	24.0	24.0	24.0	

No. 1 Model - MOD SG-4 and SH-4 -- 4159.7*
No. 2 Model - MOD SG-7 and SH-7 -- 4016.6*
No. 1 Miniature - MIN 18 -- 661.0*
No. 2 Miniature - MIN 13 -- 669.8*
No. 3 Miniature - MIN 19 -- 651.8*
No. 4 Miniature - MIN 14 -- 707.8*

Times shown are number of hours of test time on vessel in 24-hour period starting at 8:30 on the morning of that day in which the time is shown.

*Total hours test time in June 30, 1961.

The new miniature vessels are grouped into two sets of two vessels each; radically different secondary environments have been specified for each set (see Section C, Paragraph 2). In order to prevent cross contamination due to chemical carry-over to the storage tank, two steps were taken. First, new steam separators were installed in the miniature system. These are rated to remove at least 99% of all entrained particles 10 microns in size or larger for a steam flow of 8 to 32.4 lb/hr at the system operating pressure. In addition, a demineralizer resin bed was added to the miniature secondary system between the circulating pump and the circulating heater. This is intended to remove any contaminants carried over to the storage tank before they can be fed into other vessels. A check valve was installed downstream of the demineralizer so that the resin can be changed with a minimum of disturbance to the system.

2. Loop Downtime

There were no periods of prolonged shutdown during the report period and, in general, loop operation was satisfactory. However, a number of minor difficulties arose which did result in periodic shutdown of a portion or all of the loop.

The primary system circulating pump was overhauled twice during this report period. Each time the bearings were found to be worn and the thrust face of the rotor roughened. In the first instance, the thrust face was machined smooth and reinstalled with the original 6 in. impeller. However, in the second instance, the rotor shaft was worn and the original rotor which had been repaired in the mean time was installed with the 4-in. impeller from the standby pump. The rotor with the damaged shaft (from the standby pump) has been returned to the vendor for rework. The 6-in. impeller has also been returned for any necessary rework.

The primary system was shutdown for several days during the report period because of several leaks through various flanges. The leaks started during a weekend and the make-up tank was pumped dry in an effort to maintain the water level in the system.

The entire loop was shutdown by the automatic control system on two weekends during the report period due to loss of water level in one of the model steam generators. The first shutdown was the result of failure of the circulating pump bypass relief valve. The valve was repaired and has subsequently operated satisfactorily. The second incident was the result of rupture of the copper inner liner of one of the model secondary system condensate coil. A new coil, with an Inconel inner liner, was obtained and installed in place of the ruptured unit. As preventive maintenance, new coils were obtained for the other model system also.

3. Maintenance of Secondary Environments

Maintenance of the specified environments in the model system secondaries has not been a serious problem. Gradual buildup of the chloride concentration requires occasional blowdown or flushing of the system.

However, the miniature secondary systems have presented some difficulties. The measures mentioned above, new steam separators and a pump effluent demineralizer, to prevent cross contamination have been only partially successful. The steaming rate in the miniature vessels tends to oscillate with the result that the steam passes through the separator in bursts. The separation process is inefficient under these conditions and considerable carryover results. The demineralizer has been reasonably effective in removing contaminants carried over to the storage tank. However, some buildup has been experienced in MIN 14 and 19 for which low chloride, "reactor grade" environments have been specified. When this occurs, the vessels are blown down until the satisfactory conditions are established.

Chemicals are added to MIN 13 and 18 each morning to maintain the specified high chloride level. In addition, attempts are being made to establish a more uniform steaming rate in the vessels and to reduce the amount of carryover.

C. CORROSION VESSEL TESTING

1. Model Heat Exchangers

Testing of the bimetal (MOD SX-4) model vessels and the Inconel (MOD SX-7) model vessels continued during the quarter. The secondary environment in the Inconel vessels is as follows (PM-1 conditions):

pH	10 to 10.5
PO_4	150 ppm
SO_3	10 ppm
Cl	0.5 ppm (max)
Total solids	175 to 200 ppm

The secondary environment for the bimetal vessels is as follows (SM-1 water conditions):

pH	Approximately 8.5 with Na_3PO_4

Cl	0.5 ppm (max)
O_2	0.5 ppm (max)
Total solids	200 ppm (max)

Accumulated test time, as of June 30, is as follows:

MOD SX-4	4159 hr
MOD SX-7	4016 hr

The regular schedule of water sampling and analysis was maintained. Boiler water and condensate are sampled and analyzed Monday, Wednesday and Friday of each week. Makeup chemicals are added as required.

2. Miniature Heat Exchangers

Testing of the Inconel (MIN 10 and 11) and bimetal (MIN 15 and 16) miniature heat exchangers was completed during the quarter. Accumulated test times as of May 8, 1961, when the tests were terminated, are as follows:

MIN 10	3044 hr
MIN 11	3023 hr
MIN 15	3018 hr
MIN 16	3035 hr

The secondary environments for these vessels are summarized as follows:

MIN 10	
Cl	1000 ppm
pH	10 (with NaOH)
O_2	No treatment (see note at end of tabulation)
MIN 11	
Cl	1000 ppm

pH	10 (with mixture of 33% Na_3PO_4 and 67% Na_2HPO_4)
O_2	No treatment (see note at end of tabulation)
<u>MIN 15 and 16</u>	
Cl	800 ppm
pH	10 (with mixture of 33% Na_3PO_4 and 67% Na_2HPO_4)

Note:

The secondary makeup tank is makeup tank is maintained at 180° F, open to the atmosphere, which will maintain the oxygen concentration at somewhat less than 0.5 ppm.

In MIN 15 the bimetallic tubing was defected, exposing the stainless steel sublayer to the secondary environment. Defects are in the vapor phase, the liquid phase and at the vapor-liquid interface.

Preliminary results of the post test analysis of the above vessels are discussed in Section D.

After a thorough cleaning of the miniature secondary system, the following vessels were installed in the loop: MIN 13 and 14 (Monel) and MIN 18 and 19 (nickel). Testing was initiated on 26 May with the following secondary environments:

<u>MIN 13 and 18</u>	
Cl	1000 ppm
pH	10 (33% Na_3PO_4 and 67% Na_2HPO_4)
O_2	No treatment (see note)
<u>MIN 14 and 19</u>	
Cl	0.5 ppm (max)
Phosphate	150 ppm
Sulfite	10 ppm
pH	10 to 10.5

Note:

The secondary makeup tank maintained at 180° F, open to the atmosphere, which will maintain the oxygen concentration at somewhat less than 0.5 ppm.

Accumulated test times for the above vessels, as of 30 June 1961 are as follows:

MIN 13	669 hr
MIN 14	707 hr
MIN 18	661 hr
MIN 19	651 hr

The regular schedule of water sampling and analysis was maintained. Boiler water and condensate are sampled and analyzed Monday, Wednesday and Friday of each week. Makeup chemicals are added as required.

3. Reduction of Heat Transfer Data

Reduction of heat transfer data continued. The heat transfer index for each model steam generator was determined as a function of time during the report period. The index is a qualitative measure of the overall coefficient of heat transfer of the vessel. Because scale or corrosion products affect the quantity of heat transferred, the index should give an indication of scale buildup and a measure of its effect on the heat transfer rate in the generator.

The definition of the index is as follows:

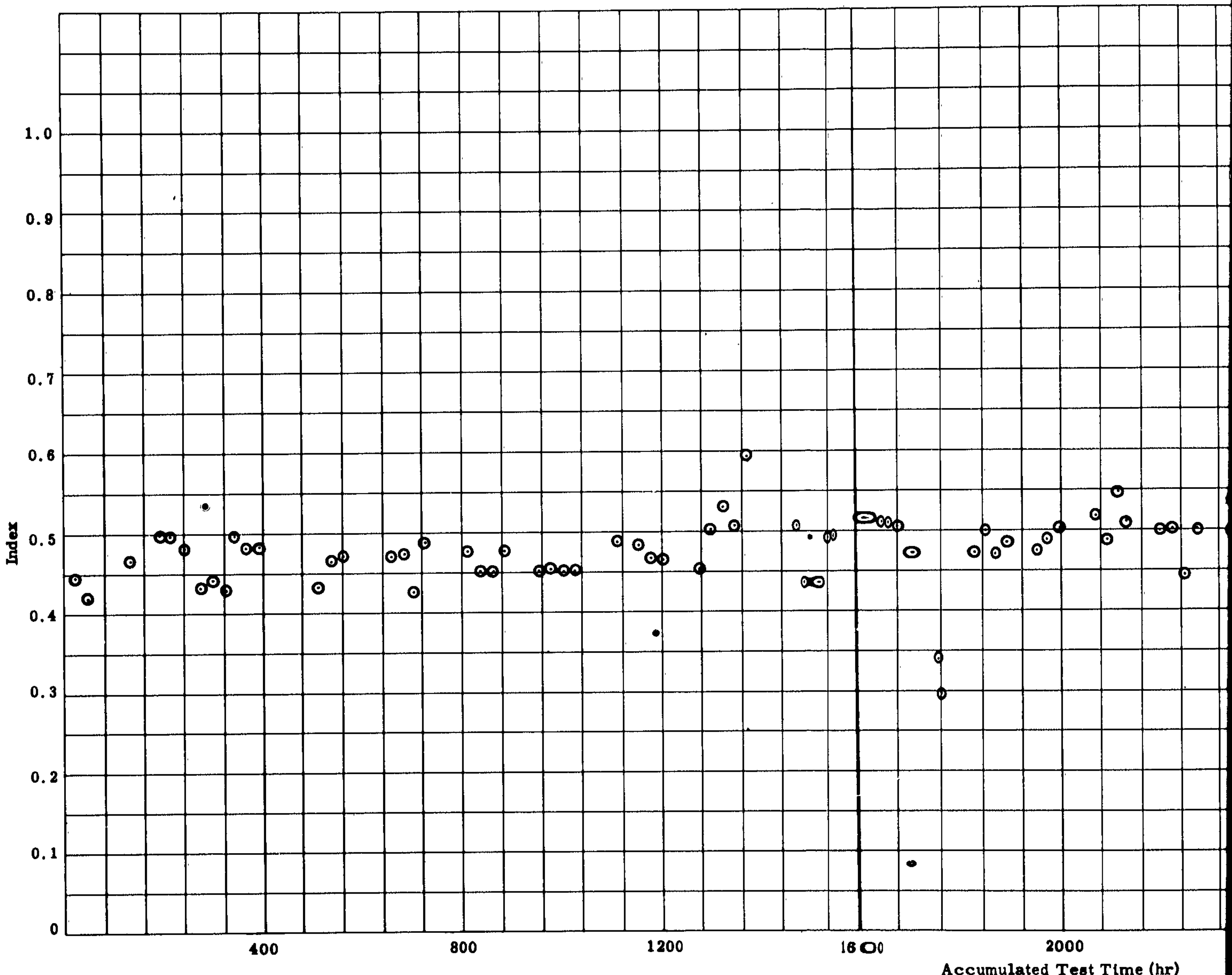
$$\text{Heat transfer index} = \frac{\text{Heat transferred to secondary fluid}}{\text{Primary fluid heat available for transfer}}$$

Derivation of the actual equation used for its computation was given in the previous quarterly report. Figures 5 and 6 are plots of the index as a function of time since the initiation of testing. The curves appear to indicate that large effects on heat transfer due to scaling have not occurred.

D. POST TEST EVALUATION OF MINIATURE VESSELS

Post test evaluation of miniature vessels, MIN 10, 11, 15 and 16, was initiated and partially completed. These vessels had been loop tested for approximately 3000 hours; the secondary environments were reviewed in the previous section of this report.

1



1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
Index
400
800
1200
1600
2000
Accumulated Test Time (hr)

Fig. 6. Heat

2

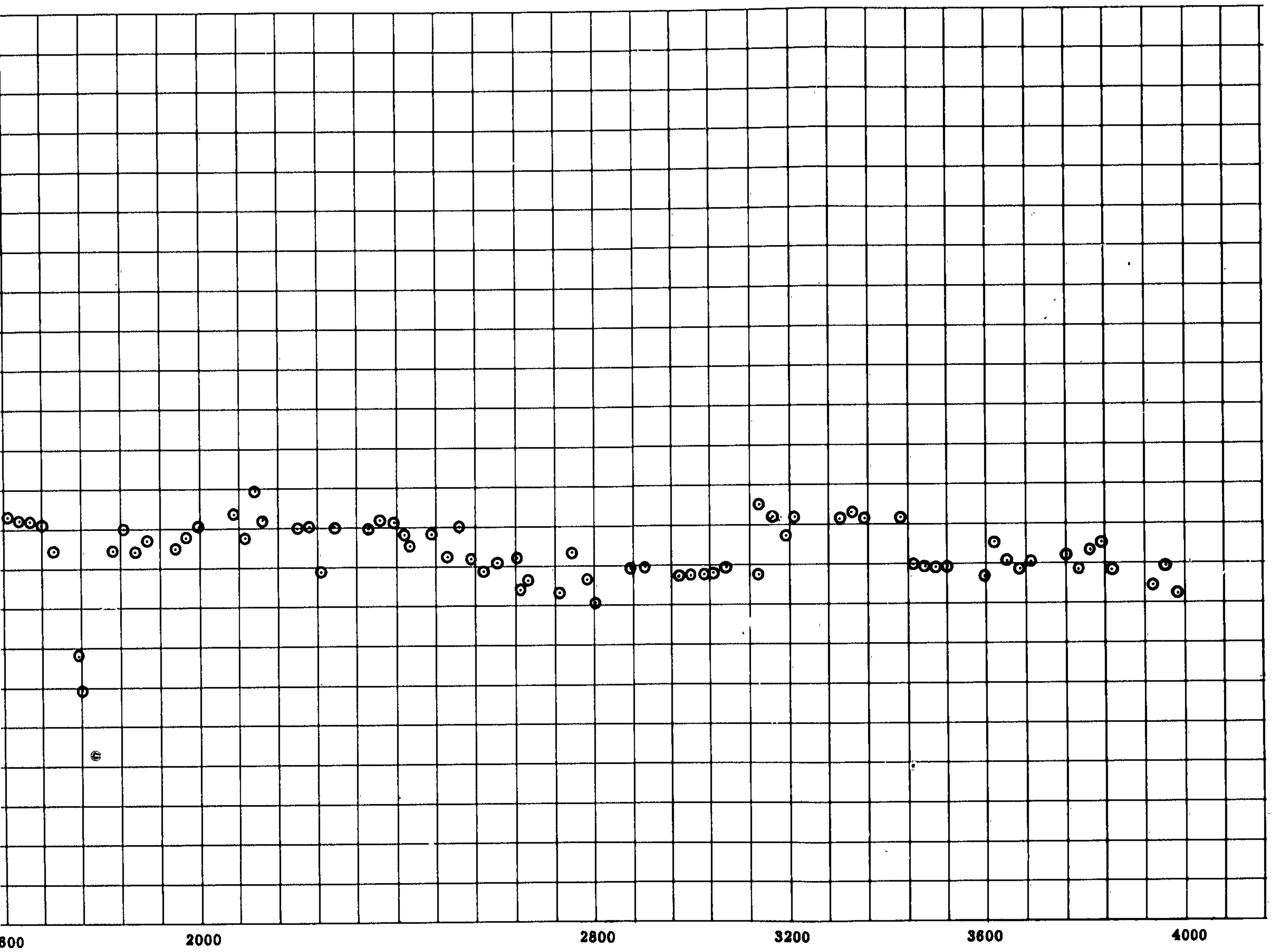


Fig. 6. Heat Transfer Index Versus Test Time; Inconel Steam Generator (MOD SG-7)

1. MIN 10 and 11

The overall appearance of the Inconel miniature vessels, MIN 10 and 11, after removal of the carbon steel secondary shells is shown in Figs. 7 and 8 respectively. A considerable buildup of boiler scale is evident on all tubes in both the liquid and vapor phases; this was anticipated and is the result of the use of tap water for makeup. X-ray diffraction analysis indicated that $CaSO_4$ was the major constituent of the scale with $CaCO_3$ as a minor constituent. Scale on the tubes exposed in the liquid phase also contained Fe_2O_3; a heavy deposit of the typical red iron rust appeared as a ring at the vapor-water interface on the tube bends. The origin of this corrosion product was undoubtedly the carbon steel secondary shell.

The Inconel tubes exhibited little evidence of corrosion. In areas where the boiler scale had flaked off, bright metal was apparent.

In fabricating these vessels, one tube in each was rolled into the tube sheet and seal welded on the primary side. The rolling operation was omitted for the second tube to determine its effectiveness in preventing environmental penetration. The results are shown in Fig. 9 which shows sections through the tube sheet at the tube penetrations as well as corresponding portions of the tubes from MIN 10. The rolled tube (2 and 3) was very effective in preventing penetration. However, corrosion did occur in the crevice between the tube sheet and the unrolled tube; this was particularly evident in the vapor phase where penetration extended the entire depth of the crevice.

2. MIN 15 and 16

The overall appearance of the bimetal miniature vessels, MIN 15 and 16, after removal of the carbon steel secondary shell is shown in Figs. 10 and 11 respectively. The carbon steel surface of the tubing, which was exposed to the secondary environment was severely attacked, particularly in the vapor phase. The major constituent of the film was Fe_2O_3; $CaCO_3$ and $CaSO_4$ were also present.

The tubing of MIN 16 was defected to expose the stainless steel sublayer to the secondary environment. The objective of this phase of the test was to obtain maximum exposure of stainless steel to the high chloride environment and, thus, to amplify any tendency toward stress corrosion cracking. It has been proposed that, in this situation, the carbon steel may provide cathodic protection and effectively prevent cracking of the stainless steel. Figure 12 shows photographs of the bimetal tubing after the corrosion film was stripped off. Visual inspection of the defected areas revealed no evidence of cracking. Numerous metallographic specimens will be prepared from these areas to firmly establish the condition of the material.

Figures 12 and 13 which shows the stripped tubes from MIN 15, indicate that the bimetallic tubing was subject to severe pitting corrosion, particularly in the vapor phase. Many of these pits progressed to the point where the stainless steel sublayer was exposed.

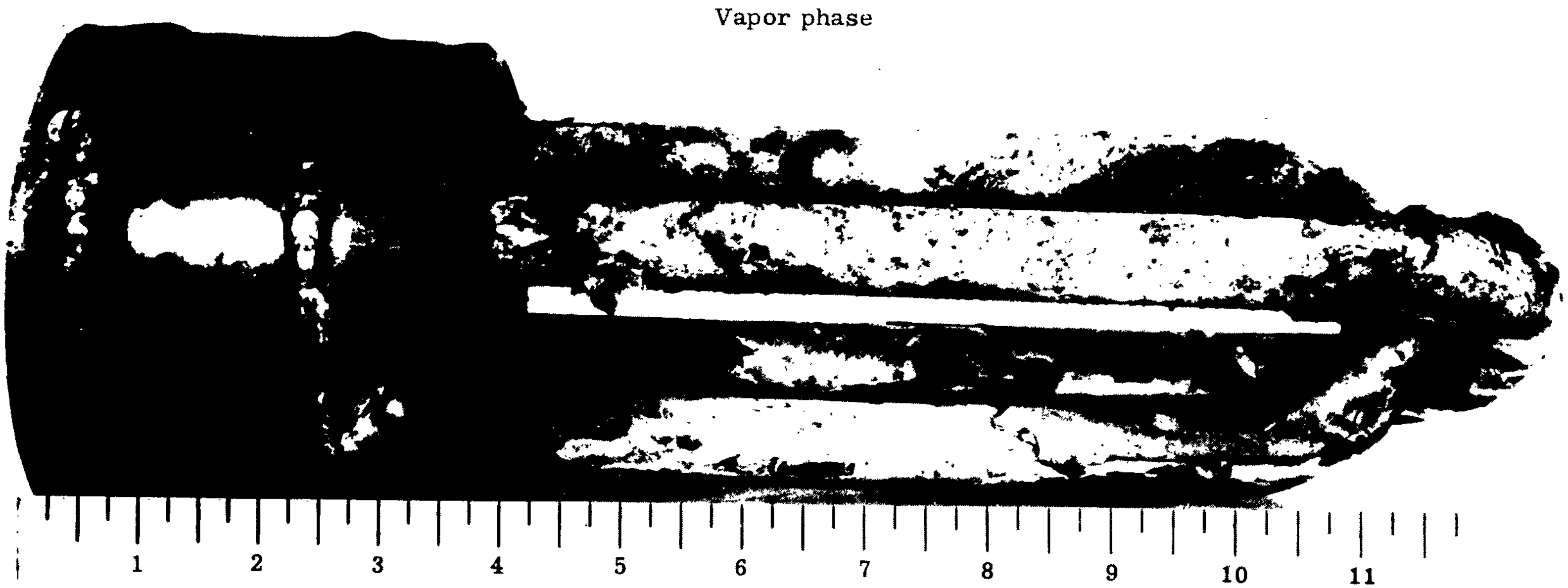


Fig. 7. MIN 10--Overall View of Secondary Shell Removed

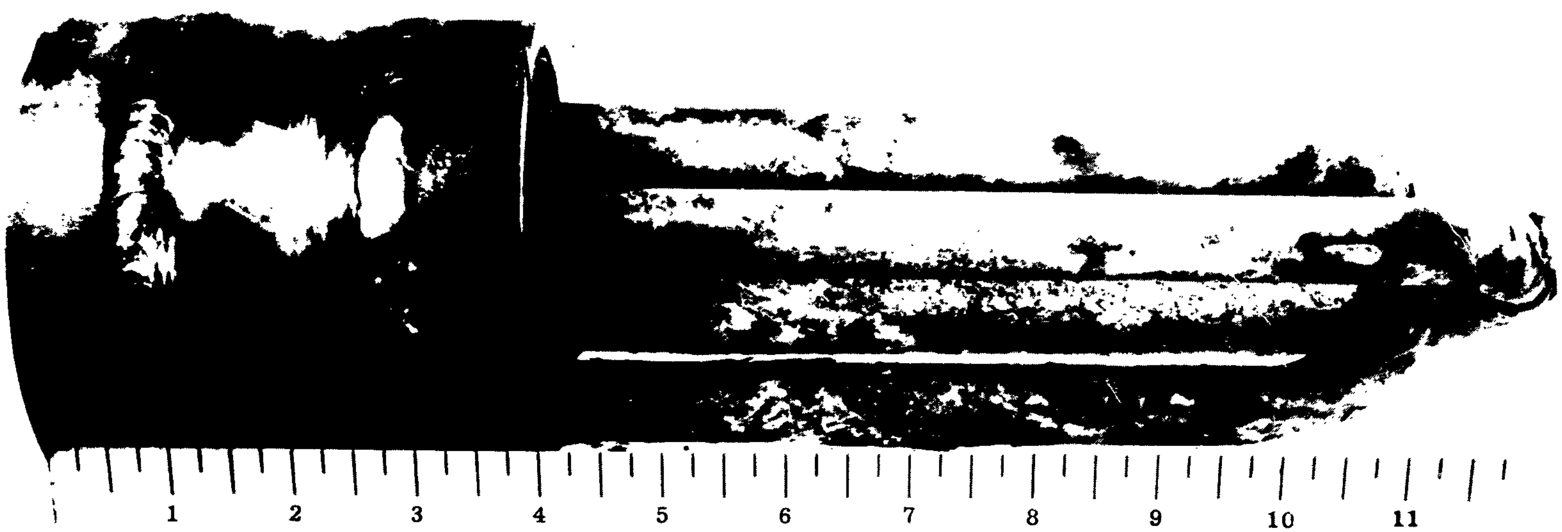


Fig. 8. MIN 11--Overall View of Secondary Shell Removed

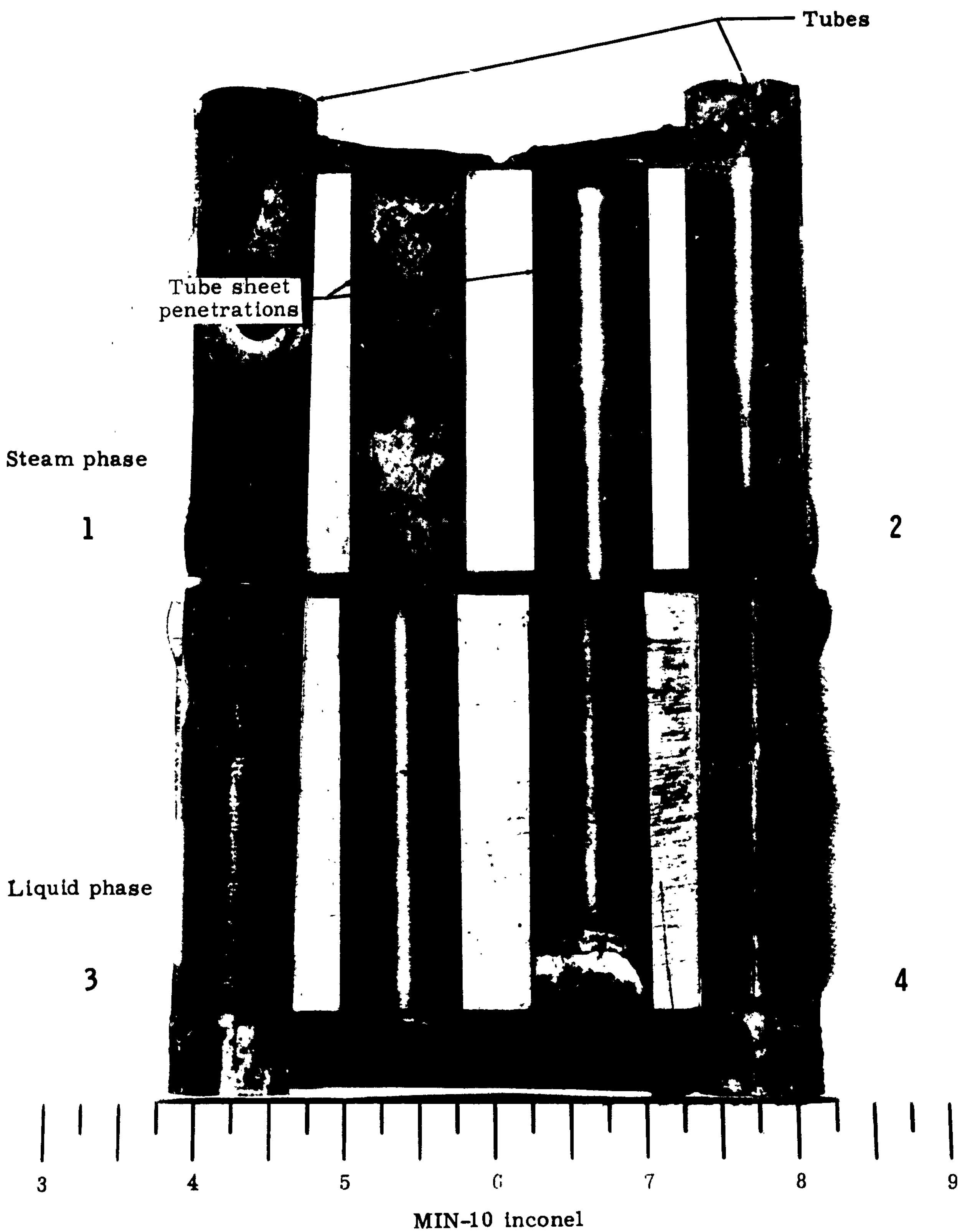


Fig. 9. Tubes and Tube Sheet Penetrations Showing Effect of Rolling Tubes

Vapor phase

Fig. 10. MIN 15--Overall View of Secondary Shell Removed

Vapor phase

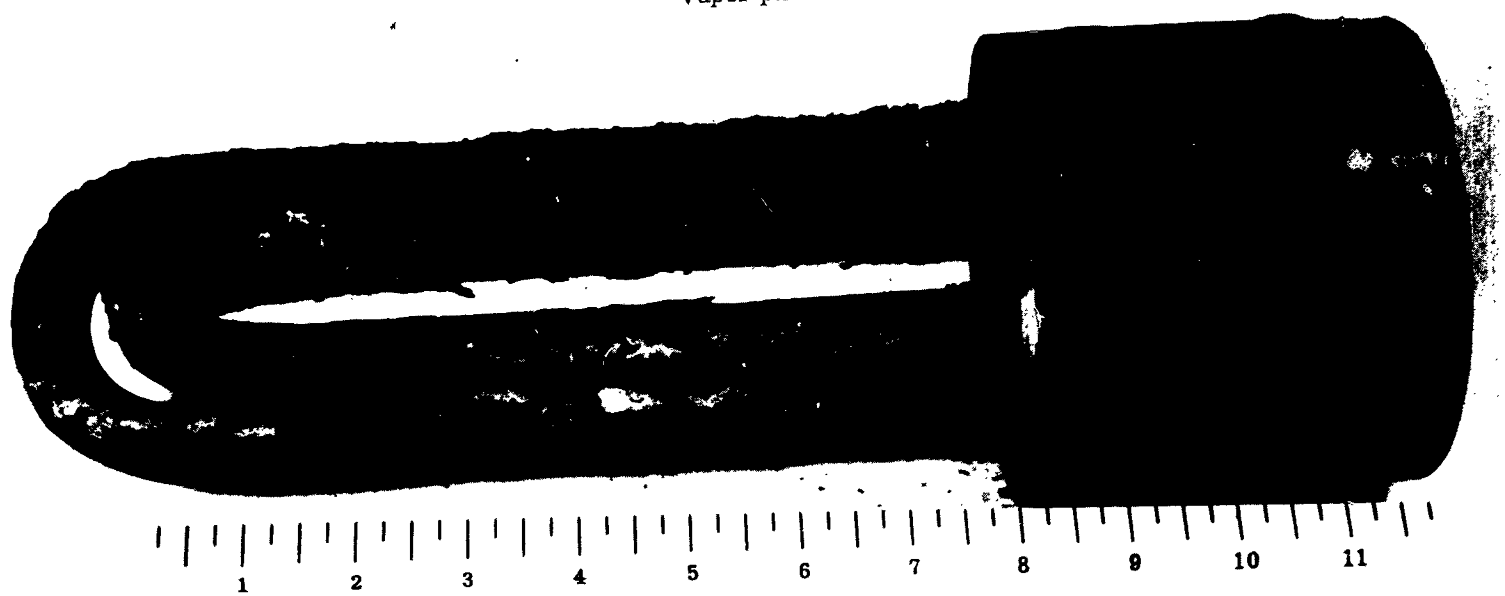


Fig. 11. MIN 16--Overall View of Secondary Shell Removed

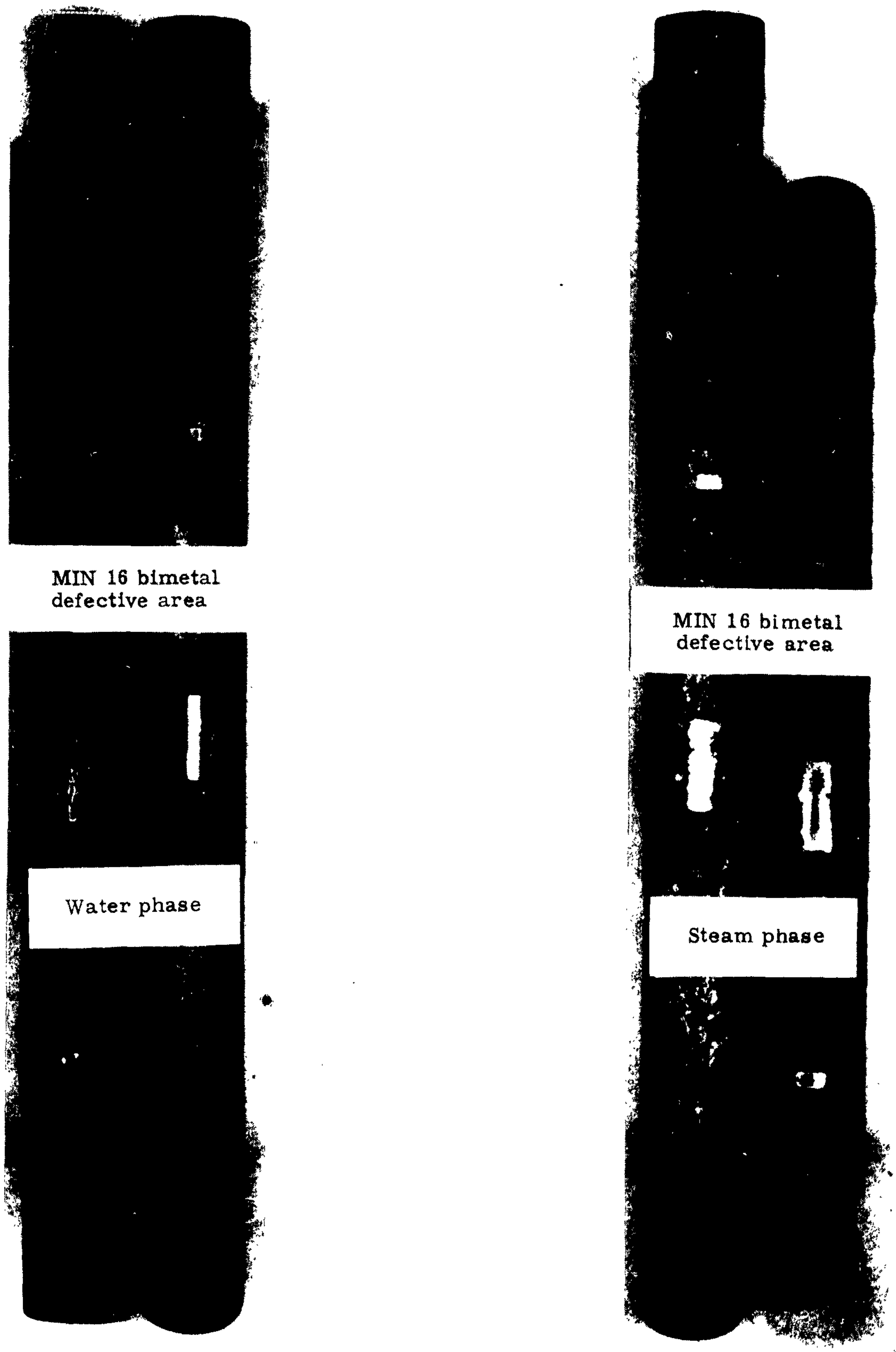


Fig. 12. Bimetal Tubing After Removal of Corrosion Film Showing Defected Areas

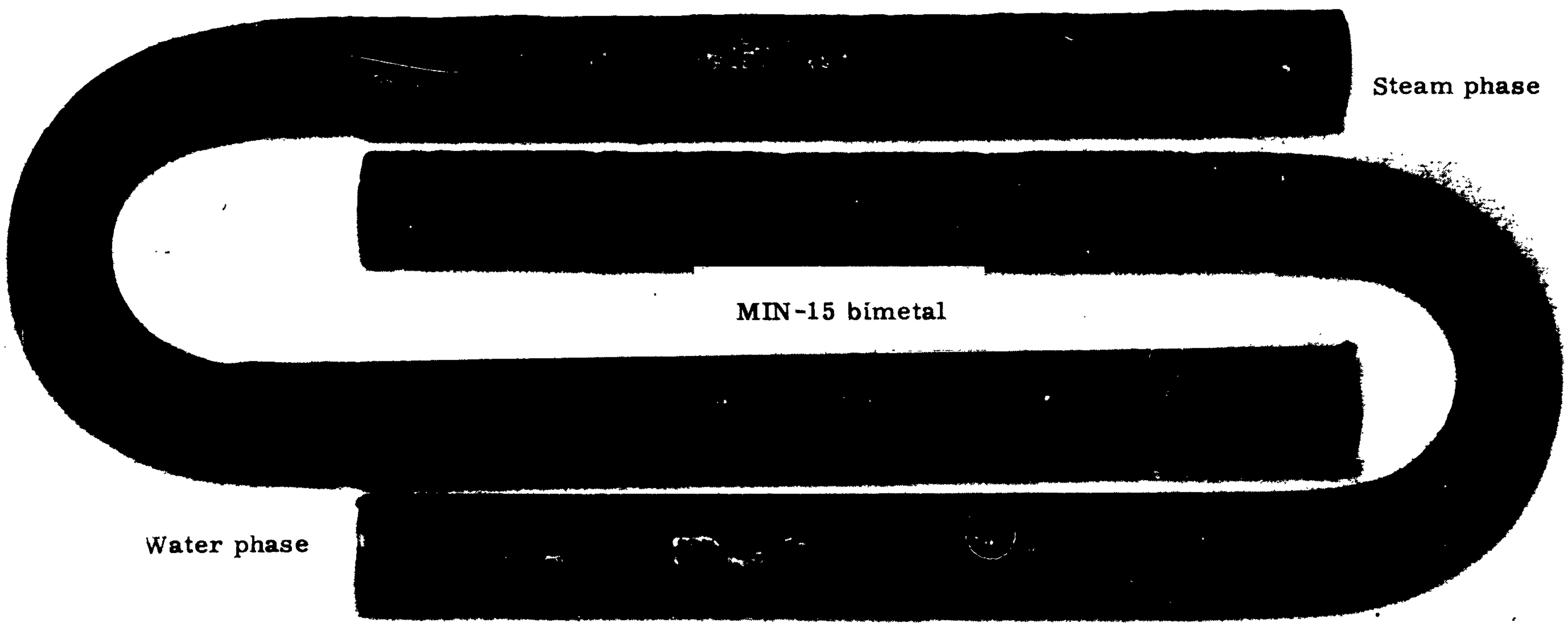


Fig. 13. Bimetal Tubing From MIN 15 After Removal of the Corrosion Film. Note Severe Pitting in the Vapor Phase

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