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SODIUM MASS TRANSFER: II SCREENING TEST DATA AND ANALYSIS - MASS TRANSFER RESULTS

Compiled by
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U.S. ATOMIC ENERGY COMMISSION
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ADVANCED PRODUCTS OPERATION
GENERAL ELECTRIC
SAN JOSE, CALIFORNIA

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SODIUM MASS TRANSFER: II
SCREENING TEST DATA AND ANALYSIS
— MASS TRANSFER RESULTS

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ADVANCED PRODUCTS OPERATION

GENERAL ELECTRIC
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I. ABSTRACT

Preliminary results from 21 test runs are presented, covering about 40,000 hours of exposure of corrosion samples in flowing sodium at temperatures between 700 and 1200 F. Samples of Type 316 stainless steel, $2\frac{1}{4}$ Cr and 5 Cr alloy steel were exposed in three temperature steps in both the hot leg and cold leg of each loop. After exposure the samples were removed from the loop, cleaned, weighed and examined for structural or compositional changes. When activated foil samples were used there were counted with a multichannel pulse height analyzer. It is apparent from the resulting information that in respect to uniform corrosion, any of the materials tested could be used in sodium systems up to 1200 F. The specific physical properties desired would determine the choice of materials.

Selective transport of alloying elements has been indicated by the formation of a transformed (ferritic) layer on the surface of the stainless samples exposed above 1000 F and by radio tracer evidence. In the bimetallic Type 316 stainless steel - $2\frac{1}{4}$ Cr loops, carbon is transferred from the ferritic to the austenitic material. The weight change data has indicated nearly the same corrosion rate (~ 1 mil/yr at 1200 F) for each of the materials exposed under similar conditions. The local corrosion rate is dependent on the temperature, geometry, and location of the sample in the test loop. Sufficient material has been transferred (under some test conditions) to foul or partially plug small channels in the cold leg. The program is being continued to provide extended exposures of samples according to a five-run fractional replicate test plan.

EDITOR'S NOTE

The overall results of this work and the correlations derived here have been published and were presented at the first three meetings of the AEC Ad Hoc review Group of the Sodium Components Development Program, and elsewhere. This volume represents an archival record of the weight change data for the first 21 test runs through 1962. This report has been issued primarily to provide a convenient record of the early runs on a pattern consistent with that later adopted for the balance of the Phase I program. It should be considered in context with subsequent reports in the same series, especially GEAP-4182 and GEAP-4438.

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II. INTRODUCTION

In 1959 the U. S. Atomic Energy Commission contracted with the General Electric Company to carry out a Sodium Mass Transfer investigation with the objective of evaluating three prospective steel construction materials for utilization in liquid sodium cooled reactor systems operated in the temperature range of 600 to 1300 F. Six test loops were designed and built to implement the study of the three selected materials: AISI Type 316 stainless steel (316 SS); $2\frac{1}{4}$ percent Chromium - 1 percent Molybdenum steel ($2\frac{1}{4}$ Cr), and 5 percent Chromium - $\frac{1}{2}$ percent Molybdenum - $\frac{1}{2}$ percent Titanium steel (5Cr). The latter two were chosen because of their extensive service in central station power plant applications. A complete description of the design and fabrication of the test loops is included in GEAP-3725. The test plans for the loop operating conditions are intended to examine the following variables: types of steel, temperature, temperature differential, velocity, oxygen content, other contaminants and time. The initial test runs were intentionally made with the most severe set of test conditions within the scope of this program.

To date, 21 test runs have been completed with the six test loops operating over 40,000 test hours. Many of the runs provided 2800 hour (4 month) sample exposures for some of the 570 corrosion samples produced to date. Twice during the test runs (at 700 hours and 1400 hours of test time) the loops were shut down and drained so that select samples could be removed from the sample holders for examination. Due to the volume of information available, this report is presented in three volumes. It is believed that separate publication of the Metallurgy report, "Volume 2", and the 1961 Corrosion Sample Data, "Volume 3", was desirable due to the specific nature of these sections. The cutoff date for samples removed from the test loops was December 31, 1961 for all volumes of this report. After that date, many samples were analyzed for weight change, hardness, carbon content, and metallurgical and surface composition changes.

The basic objective of the present study as defined in the AEC contract is to investigate materials suitable for use in a sodium cooled nuclear system which can deliver 1050 F steam to a turbine. Taking temperature drops into account, this requirement has been interpreted to mean that sodium-metal compatibilities be investigated for temperatures up to 1300 F.

In order to carry out this commitment, the G. E. -APED test loops have been designed to carry out studies of corrosion and mass transfer. The present investigation differs from previous work with pumped non-isothermal loops in the following characteristics:

1. The principal emphasis is on higher temperatures ($1100\text{ F} \leq T_{\text{Max}} \leq 1300\text{ F}$) and higher ΔT 's, (500 F or more).
2. A wide range of flow velocities, up to 25 ft/sec, and oxygen levels up to 50 ppm, are available.
3. Corrosion samples are located in three temperature zones of both the hot leg and the cold leg of each loop.
4. The six test loops fabricated from different combinations of the steel alloys makes it possible to examine corrosion, mass transfer, and similar phenomena in both the familiar homo-metallic, and in the hetero-metallic systems which are not nearly so well known or understood.

5. The equipment includes components for both the measurement and close control of the important variables such as the oxygen content, flow rate, temperature, and temperature distribution.

From other work on the steel-sodium systems, the transfer of matter through and with the liquid metal coolant, and deposition of solids in various parts of the loop, are believed to be the factors tending to limit the extension of sodium technology to higher temperatures. A satisfactory solution of the problem then will require (1) the selection of materials with suitable mechanical and corrosion properties, and (2) a model for the corrosion-mass transfer processes taking place which will make quantitative prediction of the extent of these phenomena possible. This model also can be used to guide engineering design and to determine the effective life of the components employed.

So far as the nature of the corrosion processes in liquid sodium, and the associated mass transfer phenomena, are concerned, there is good reason to believe that in the absence of impurities (oxygen, for example), sodium is a relatively inert material and the corrosion mechanisms involved in this medium are relatively simple in concept. These interpretations of the nature and characteristics of the corrosion and mass transfer phenomena in liquid metals in the past have been based largely on observations made at lower temperatures ($T < 1000$ F) and small ΔT values. These data have led to a theoretical picture, and the conclusions that the processes observed are dependent on:

1. A basic chemical reaction with the oxygen in the sodium, forming adherent oxides of some of the metallic constituents of the system and consequently resulting in weight gains, and
2. A process of solution of the loop materials in the hot zone, and deposition in the colder regions of the system. The rate for this process is determined by either the rate of solution phenomena, the diffusion of dissolved metal through the liquid sodium solvent or by diffusion of constituents in solid solution in the metal to the liquid-solid interface. All three classes of effects have previously been observed.

From these concepts it is possible to predict the form of the dependence of the corrosion and mass transfer rates on such variables as time, temperature, ΔT , flow velocity, oxygen content, etc., once the basic nature of the corrosion process for any given material has been established. In the present experiments, the results to be expected at higher temperatures, ΔT 's, and flow velocities have, to a first approximation, been estimated by extrapolation of the older information, and a major task in this study is to compare the results of the new work with these predictions from theories derived from studies made under somewhat milder conditions.

Sections III and IV of this volume provide information on details and modifications of the experimental equipment and on the test plan and operating conditions. Sections V and VI provide the results of the activated foil radio-tracer work and the statistical analysis of the corrosion sample weight change data.

References to the Theory of Liquid Metal Corrosion

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6. Keyes, J. J., Jr., "Some Calculations of Diffusion Controlled Thermal Gradient Mass Transfer," ORNL Report 57-7-115 (July 22, 1957).
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III. EXPERIMENTAL EQUIPMENT AND TEST CONDITIONS

- III A Test Loops and Components
- III B Test Loop Geometry, Temperature and Flow Characteristics
- III C Status of Test Runs and List of Test Conditions

A. Test Loops and Components

Six forced circulation test loops, described in detail in report GEAP-3725, were designed, fabricated and operated to provide the experimental data in this report. The steels used for construction and test are: Type 316 Stainless Steel, $2\frac{1}{4}$ Cr-1 Mo alloy steel and 5 Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti alloy steel. Three loops are monometallic - representing each of these materials - and three are bimetallic. Each bimetallic loop has type 316 SS in the heated leg; two loops have $2\frac{1}{4}$ Cr steel and one loop has 5 Cr steel in the cooled leg.

Figure III-1 is an isometric drawing of the loop arrangement. Primary components include seven flanged sample holders, three heaters, three air coolers, an electromagnetic pump, a surge tank and a dump tank. A bypass line around the pump, in parallel with the main loop, contains a plugging meter to measure and a cold trap to control sodium oxide impurity level.

System modifications incorporated after the initiation of the first test runs included:

1. Installation of a sodium valve at the cold trap inlet to obtain trap isolation during plugging meter determinations.
2. Installation of an immersion type thermocouple. Installation of a smaller flow area plugging plate and mounting the cooler in a horizontal flow position in the plugging indicator. These changes were made to facilitate more reproducible plugging determinations, particularly at the lower oxygen level of 305 F (12 ppm oxygen) planned for the next series of runs.
3. Replacement of all ferritic steel flowmeter cells in the bypass purification lines with type 316 SS flowmeter pipes to obtain a stronger and more reliable flow signal.
4. Installation of an orifice, in the inlet of the bypass line, to compensate for the high flow resistance of the main loop and to facilitate obtaining proper bypass flow (~ 0.2 gpm) required for cold trap and plugging indicator operation.
5. Replacement of the pump cell in all the loops by a modified cell with a thinner sodium depth (from 0.197 inch to 0.142 inch) to provide additional developed head. Figure III-2 shows performance curves of shutoff head versus pump voltage for two different materials and the modified cell. The $2\frac{1}{4}$ Cr curve is representative of the original ferritic steel cells. All the original cells, due to heating effects in the flexible nickel electrodes, were limited to a maximum pump voltage of about 210 volts. Under these conditions the maximum developed head was 38 psi for the $2\frac{1}{4}$ Cr pump cell and 66 psi for the 316 SS cell. By reducing the sodium depth as indicated above, the shutoff at 210 volts was almost doubled (to 121 psi for the 316 SS cell). The modified cells also had solid copper

FIGURE III-1 LOOP ARRANGEMENT

FIGURE III-2 E M PUMP PERFORMANCE

electrodes substituted for the flexible nickel electrodes, which permitted operation up to 265 volts without evidence of excessive heating. This increase from 210 to 265 volts for the 316 SS cell further increased the available head by another 61 psi (to 182 psi).

Although data are not shown for the modified ferritic cells, similar improvement has been experienced with them.

6. Fitting the top of the surge tank vapor trap with a 1-inch o.d. tubing connection and installed a side port gas connection to facilitate sodium sample removal.
7. Relocation of the "in-pipe" corrosion samples to provide more convenient access during removal. Hot leg pipe samples for Loop 1-run 1 were located between the H3 heater and the H3 sample holder; during all other runs and on the other loops these samples were located between H3 and H3R. Similarly, cold leg pipe samples for run 1 tests on all loops were located between the C3 cooler and the C3 sample holder for later runs and on the other loops these samples were located immediately downstream of C3.
8. Modification of the sample loading pattern for a special series of tests. In this case one sample was loaded in each of the seven sample holders as shown in Figure III-3. The special series of runs included runs 2-2, 2-3, 2-4, 3-3, 3-4, 3-5, and 3-6. The normal sample loading pattern was used for all other runs with 26 corrosion samples in the loop sample holders, and was described in more detail in GEAP-3725. The pattern is shown in Figure III-4.

B. Test Loop Geometry, Temperature and Flow Characteristics

A summary of the test loop geometry, temperature and flow characteristics for the six loops are described by curves and tables in Appendix A.

C. Test Conditions

Twenty-one test runs were completed prior to December 31, 1961. Table III-1 provides a summary of test conditions for the completed, present and near-future test runs. The first three runs in loop 1 (runs 1-P, 1-1, 1-1F) plus the initial runs in loops 3, 4, 5 and 6 were conducted at maximum planned conditions of temperature, system ΔT and oxygen level using "commercial" grade sodium. The initial run in loop 2 was operated at the same conditions except with low oxygen level. All subsequent runs have been with reduced oxygen level (~10 ppm) and "reactor" grade sodium. Current runs include one operating at reduced temperature (run 5-3) and one at reduced ΔT (run 1-3). The test conditions are provided by the statistical, two level, fractional replicate test program discussed in Section VI.

In the "sample arrangement" designated line in Table III-1, the following notations are useful:

1. "26" refers to the standard 26-sample loading as shown in Figure III-4.
2. "7" refers to the 7-sample loading shown in Figure III-3.
3. "2 + 2F" is based upon two standard samples in H3 plus two filter-screen specimens (one in C1 and one in C3) for collection of fouling material.
4. "3 + 3F" is based upon 3 corrosion samples (two in H3 and one in H3R) plus filter-screen specimens in each of the three cold leg sample holders.

SAMPLES: (26) REGULAR · (1) 6 MIL FOIL

FIGURE III-3 SEVEN SAMPLE LOADING PATTERN

SAMPLES: (25) REGULAR : 6 MIL FOIL

FIGURE III-4 NORMAL SAMPLE LOADING PATTERN

TABLE III-1
LIST OF TEST CONDITIONS - DECEMBER 1961

Completed				Test Conditions	Present	Next		
				<u>Loop No. 1 (316 SS)</u>				
<u>1-P</u>	<u>1-1</u>	<u>1-1F</u>	<u>1-2</u>	Run number	<u>1-3</u>	<u>1-4</u>		
1300	1300	1300	1200	T _{max} , °F	1200	1100		
500	500	500	500	ΔT, °F	250	250		
50	50	50	10	Oxygen, ppm	10	50		
26	26	2+ 2F	26	Sample Arrangement	26	26		
18&6	12&4	18	18&6	Velocity, ft/sec	18&6	18&6		
No	Yes	No	Yes	Radioactive Tab	No	Yes		
4	7, 14, 28	4-8	7, 14, 21	Samples removed, 100's hr	7, 14, 28	7, 14, 28		
6/60	10/60	5/61	8/61	Start, mo/yr	2/62	7/62		
11/60	5/61	7/61	12/61	Complete, mo/yr	-	-		
-	-	"F"	-	Special Condition*	-	-		
				<u>Loop No. 2 (316 SS - 2¼ Cr)</u>				
<u>2-1</u>	<u>2-2</u>	<u>2-3</u>	<u>2-4</u>	Run number	<u>2-5</u>	<u>2-6</u>		
1200	1200	1200	1200	T _{max} , °F	1200	1100		
500	500	500	500	ΔT, °F	500	500		
10	10	10	10	Oxygen, ppm	10	10		
26	7	7	7	Sample Arrangement	26	26		
20&7	20	7	7	Velocity, ft/sec	21&7	21&7		
No	Yes	Yes	Yes	Radioactive Tab	No	No		
7, 14, 28	7	7	28	Samples removed, 100's hr	14, 28, 56	7, 14, 28		
10/60	5/61	7/61	9/61	Start, mo/yr	2/62	1/63		
2/61	7/61	8/61	12/61	Complete, mo/yr	-	-		
-	-	-	-	Special Condition*	"C"	-		
				<u>Loop No. 3 (316 SS - 2¼ Cr)</u>				
<u>3-1</u>	<u>3-2</u>	<u>3-3</u>	<u>3-4</u>	<u>3-5</u>	<u>3-6</u>	Run number	<u>3-7</u>	<u>3-8</u>
1200	1200	1200	1200	1200	1200	T _{max} , °F	1200	1200
500	500	500	500	500	500	ΔT, °F	500	250
50	10	10	10	10	10	Oxygen, ppm	10	10
26	26	7	7	7	7	Sample Arrangement	26	26
17&6	17&6	9	22	20	23	Velocity, ft/sec	21&7	21&7
No	No	No	No	No	No	Radioactive Tab	No	No
7, 14, 28	7	7	7	7	15	Samples removed, 100's hr	7, 14, 28	7, 14, 28
8/1/60	3/61	4/61	6/61	8/61	9/61	Start, mo/yr	2/62	8/62
12/60	4/61	5/61	7/61	9/61	11/61	Complete, mo/yr	-	-
-	"A"	-	-	-	-	Special Condition*	"D"	-

*Special conditions or remarks:

"A" - 4S8 and 4S4 steel alloy samples in the cold leg sample holders.

"B" - All hot leg sample holders at 1200 F; all cold leg sample holders at 700 F (if possible without modifying sodium system).

"C" - Re-run of 2-1 after ageing; double time

"D" - Same as 3-1 except 10 ppm O₂; Same as 2-1

"F" - Run to obtain fouling material.

TABLE III-1 (Continued)

Completed			Test Conditions	Present	Next
<u>Loop No. 4 (316 SS-5 Cr)</u>					
<u>4-1</u>	<u>4-2</u>	<u>4-3</u>	<u>Run number</u>	<u>4-4</u>	<u>4-5</u>
1200	1200	1200	T _{max} , °F	1200	1200
500	500	500	ΔT, °F	500	250
50	10	10	Oxygen, ppm	10	10
26	26	26	Sample Arrangement	26	26
12&4	11&4	12&4	Velocity, ft/sec	12&4	12&4
No	Yes	No	Radioactive Tab	No	No
7, 14, 28	7, 14, 28	7	Samples removed, 100's hr	7, 14, 28	7, 14, 28
8/60	5/61	10/61	Start, Mo/yr	11/61	5/62
1/61	9/61	10/61	Complete, mo/yr	-	-
-	-	"B"	Special Condition*	"B"	-
<u>Loop No. 5 (2½ Cr)</u>					
<u>5-1</u>	<u>5-2</u>		<u>Run number</u>	<u>5-3</u>	<u>5-4</u>
1200	1200		T _{max} , °F	1100	1200
500	500		ΔT, °F	500	250
50	10		Oxygen, ppm	10	10
26	26		Sample Arrangement	26	26
12&4	18&6		Velocity, ft/sec	15&5	15&5
No	No		Radioactive Tab	No	No
7, 14, 28	7, 14, 30		Samples removed, 100's hr	7, 14, 28	7, 14, 28
9/60	7/61		Starting Date	1/62	7/62
3/61	12/61		Completion Date	-	-
-	-		Special Condition*	-	-
<u>Loop No. 6 (5 Cr)</u>					
<u>6-1</u>	<u>6-2</u>		<u>Run number</u>	<u>6-2F</u>	<u>6-3</u>
1200	1200		T _{max} , °F	1200	1200
500	500		ΔT, °F	500	500
50	10		Oxygen, ppm	10	10
26	26		Sample Arrangement	3 + 3F	26
15&5	13&4		Velocity, ft/sec	13&4	13&4
No	No		Radioactive Tab	No	No
7, 14, 28	7, 14, 28		Samples removed, 100's hr	-	7, 14, 28
9/60	5/61		Starting Date	10/61	3/62
1/61	10/61		Completion Date	-	-
-	-		Special Condition*	"F"	"B"

IV. OPERATING CONDITIONS (G. Billuris)

- IV A Introduction
- IV B Plugging Indicator Data (Oxygen Control)
- IV C Temperature of Sample Holders
- IV D Flow Rate and Deposition
- IV E Sodium Impurity Analysis
- IV F Sodium Leaks

A. Introduction

The experimental procedures used for each of the six loops, during initial runs, were essentially those described in the test loop design report ("Sodium Mass Transfer: I, Test Loop Design," GEAP-3725.) Changes in procedures and operating conditions for subsequent tests are described in the following paragraphs:

1. Sodium Replacement

Prior to Run 3 of loop 3 and Run 2 in all other loops, the original commercial-grade sodium was replaced with DuPont Reactor Grade Sodium. To facilitate more complete removal of the original charge, the loop sodium was circulated for 4 to 5 days at 1 gpm with flow through the cold trap at about 600 F, then dumped while hot and cooled to 300 F in the dump tank. This cycle was repeated three times until finally, the sodium was transferred from the dump tank, through a temporary dip line, into waste containers.

Approximately 800 pounds of Reactor Grade Sodium, in two 55 gallon drums was supplied by E. I. DuPont de Nemours Co. from Lot 52. (The sodium supplied to MSAR for a related experimental program on physical property investigations and 19,000 lb. of the EBR-II sodium also was from Lot 52). About 40 pounds of this sodium, at 250 F, was transferred to each loop dump tank through a 65 μ pore size, stainless steel filter. The flushing and draining procedure was similar to that employed with the original sodium. Then, this initial Reactor Grade sodium was drained from the loop dump tank and a new charge of Reactor Grade sodium was added as before. The final charge was circulated for several days, with cold trapping as necessary to establish a 290 F to a 300 F plugging temperature. All subsequent tests have used this sodium, although limited makeup (5 to 10 lb), due to loss by leakage and sodium sampling, has been necessary for loops 2 and 4.

2. Establishing Test Conditions

To establish test conditions for a new run or for a continuing run after the sodium has been dumped, the loop was filled with 300 F sodium from the dump tank. A subsequent period of 4 to 6 hours was usually required to attain desired flow, temperature and oxide conditions. In this transient period during startup, the logging of test time was initiated when the desired flow, C3 temperature and cold trap conditions were reached, and the system ΔT was 300 F (for normal 500 F ΔT operation). The sequence of steps for attaining test conditions after filling the loop is as follows:

- a. Establish desired main loop flow.
- b. Heat C3 to 700 F (or to the normal test temperature if less than 700 F) and heat H3 to 800 F using the main heaters.
- c. Establish cold trapping at 300 F corresponding to 10 ppm oxygen conditions.
- d. Continue loop heating so that H3 approaches test conditions at a nominal rate of 100 F/hr.
- e. Discontinue cold trap operation after 4 to 24 hours and monitor the plugging temperature two to 2 to 4 hours later.
- f. Proceed as for normal run operation.

3. Sodium Samples

During initial testing, sodium samples for chemical analyses were removed from the dump tank at 500 F. In order to provide samples more representative of actual test environment, the sodium samples subsequent to run 1 were taken from the surge tank during normal flow and temperature (operating) conditions.

Recent sampling procedure modifications to minimize system contamination utilize a pre-evacuated pyrex tube which is immersed and broken below the sodium surface while contained in a close-fitting, perforated metal tube. Sampling at temperatures up to 700 F has resulted in less than 2 ppm oxygen addition to the loop based upon plugging data.

4. Continuous Bypass Flow

Flow in the bypass purification lines was maintained only for cold trap operation or plugging determinations during the initial test run. When these lines were not used, temperatures in the finned coolers dropped below the oxide saturation temperature. In order to overcome potential oxide "hide-out" or non-representative plugging data due to re-dissolving the oxide, continuous flow has since been maintained through the plugging indicator during test runs. The cold trap is normally maintained at the desired saturation temperature and isolated from the circulating sodium except during a cold trap run.

5. Oxide Additions

During the initial runs when the plugging temperature decreased below prescribed limits (435 F plugging temperature lower limit for nominal 50 ppm oxygen runs) operation of the cold trap at 450 F or higher was initially used to increase the oxygen level. In the latter part of this run Na_2O was added to the dump tank, the loop sodium dumped, then refilled at 450 F or higher temperature, to increase the concentration of dissolved oxide.

During the first, low oxygen runs some of the loops showed evidence of increasing oxygen concentration during test. Atmospheric contamination from sampling, diagnosed as one

of the potential factors, was minimized by sampling procedure modifications previously described. Another contributing factor was found to be gradual leakage of dump tank sodium back through the dump valve into the circulating loop. The dump tank was operated at 500 F to 600 F and it probably contained a relatively high oxygen level. This source of difficulty was overcome by (a) maintaining the dump tank at 300 F so that any sodium leakage into the main loop would result in negligible change in oxide concentration and (b) adjusting the surge tank and dump tank pressures (~ 2 psi ΔP) to hold constant sodium level.

For later runs, when the oxygen concentration dropped below ~ 9 ppm (plugging temperature of 285 F) a weighed quantity of Na_2O (usually 50 mg to 100 mg) was loaded into a porous stainless steel capsule and immersed in the surge tank sodium. Increase in plugging temperatures indicate close agreement with calculated oxygen rise from the added oxide. Data obtained in verification of this procedure is illustrated in Figure IV-2. In loop 6, following run 6-1, duplicate plugging determinations showed an oxide saturation temperature of 340 F, corresponding to 18 ppm oxygen. Plugging values, obtained 22 and 38 hours after the addition of 0.388 gm of Na_2O , showed a saturation temperature of 375 F (26 ppm oxygen) corresponding to a net oxygen addition of 8 ppm. This compares with a calculated increase of 10 ppm oxygen. Calculations based upon a sodium volume of 758 in.³ (Appendix A data) or 12,300 cc, at a mean sodium temperature of 950 F (where $\rho = 0.83$ gm/cc) indicate $\sim 10^4$ grams of sodium in the circulating loop. To raise the oxygen concentration in the sodium by 1 ppm for these conditions requires:

$$\frac{62}{16} \frac{\text{gm Na}_2\text{O}}{\text{gm O}_2} \times 10^4 \text{ gm Na} \times \frac{1}{10^6} \frac{\text{gm O}_2}{\text{gm Na}} = 0.0387 \text{ gm Na}_2\text{O}$$

The addition of 0.388 gm Na_2O therefore should have increased the saturation value of 0.388/0.0387 or 10 ppm oxygen.

B. Plugging Indicator Data

Most of the sodium corrosion work which has been reported by previous investigators did not measure oxygen level during their tests. Instead, the oxide concentration was estimated from initial charging conditions, samples analyzed at the end of run, or diffusion cold trap temperatures. In the current Mass Transfer investigations sodium oxide was considered one of the significant variables which should be carefully controlled. On the basis of available technology, the plugging indicator was selected as the most convenient and reliable device for measuring the oxygen concentration during test. The excellent agreement between oxygen determinations from chemical analysis and plugging determinations over the region of 10 ppm to 200 ppm is reported in literature⁽¹⁾. The solubility curve for sodium oxide in sodium, shown in Table IV-1 and plotted in Figure IV-1 was based upon reported KAPL data⁽²⁾ and extrapolated to lower levels by L. F. Epstein and G. Billuris, was used in converting plugging temperature to oxygen concentration.

(1) Gray, I. L., Neal, R. W., Voorhees, B. G., *Nucleonics* 14, 10, 34, (1956).

(2) Salmon, O. N., and Cashman, T. J., Report KAPL-1653, (1956).

TABLE IV-1

SOLUBILITY OF SODIUM MONOXIDE IN SODIUM

<u>Temperature °F</u>	<u>Solubility ppm Oxygen</u>	<u>Temperature °F</u>	<u>Solubility ppm Oxygen</u>
290	10	480	65
300	11	490	71
310	12	500	77
320	14	510	84
330	16	520	92
340	18	530	100
350	20	540	108
360	22	550	116
370	24	560	125
380	27	570	134
390	30	580	144
400	33	590	154
410	36	600	164
420	39	610	175
430	42	620	187
440	46	630	200
450	50	640	213
460	55	650	227
470	60		

The time averaged values for sodium oxide determined from plugging temperature measurements are tabulated in Volume 3 of this report (1961 Corrosion Sample Data) for each run. In addition, operating data curves showing plugging temperatures versus test time for each run are included in Appendix B of this volume. In the initial test runs planned for 50 ppm oxygen, excluding periods of known contamination, the sodium oxide concentration showed a consistent tendency to decrease with time. Most of the later runs conducted in the region of 10 ppm oxygen did not exhibit as consistent a trend with time.

Because the oxide content during the initial runs was observed to generally decrease with time even though appendage line temperatures exceeded the plugging temperature, several short duration tests were conducted prior to the second runs to obtain additional data on this behavior. The following information was sought in these runs:

1. Rate of change in oxide content with time.
2. Lower limit at which the oxide concentration did not change.
3. Effect on plugging temperature of the batchwise addition of Na_2O in porous capsule form, to the loop surge tank.

276-43

FIGURE IV-1 SOLUBILITY OF SODIUM MONOXIDE IN SODIUM (FROM KAPL DATA)

Test data obtained for this evaluation, plotted in the form of plugging temperature versus time, are shown in Figure IV-2.

In loop 3 during a seven day period, with plugging temperatures normally measured four times daily, the saturation temperature decrease was approximately linear from 455 F to 420 F (or 5 F per day). This corresponds to a change from 52 to 39 ppm oxygen, or 2 ppm/day. In a subsequent 5 day period the saturation temperature dropped from 315 F (13 ppm oxygen) to 295 F (10 ppm) in one day; it then remained essentially constant at 290 F to 300 F over the next four days.

In loop 4, during an 18 day run, the plugging temperature decreased from 390 F to 340 F at a rate equivalent to about 1 ppm oxygen/day. During the last 5 days of this run, the decrease was only 5 F indicating a leveling-off effect at the lower saturation temperature.

In loop 6 with the saturation temperature at 340 F (18 ppm oxygen) on consecutive days, a porous stainless capsule containing 0.388 gm of Na_2O was immersed in the surge tank in order to raise the oxide content. The plugging temperature 14 hours later was 360 F (22 ppm). At periods of 22 and 38 hours following the capsule addition, the saturation temperature was constant at 375 F (26 ppm) indicating 80 percent recovery of the added Na_2O by the system sodium within a one day period. Continued plugging measurements showed that with loop temperatures ranging from 1050 F to 700 F, the saturation temperature decreased within two days to 370 F and held constant over the following six days. The loop maximum temperature was then increased to 1170 F, and within four hours the plugging temperature increased to 380 F. Over the remaining 10 days of the test, the plugging temperature decreased only 5 F.

C. Temperature Control of Sample Holders

A summary of the temperatures which were measured at sample holders C3 and H3 during all completed standard test runs is shown in Table IV-2. The mean value columns are the time averaged temperatures throughout each individual run. The other columns represent the percentage of time during a run at which the logged temperature was within 20 F (or 30 F) of the nominal value. The nominal values for run 1-1 were 800 F and 1300 F; in the other runs nominal values were 700 F and 1200 F.

A tabulation of time averaged temperatures for all sample holders is included in Volume 3. Typical temperature variation for the seven sample holders during a test run is illustrated in Figure IV-3, which shows temperature versus time for run 3-1.

D. Flow Rate

A characteristic of all runs completed thus far has been a decrease in flow with time. Figure IV-4 presents the loop flow for test runs in two loops. Both runs were operated with low oxygen level. The top curve is representative of most bimetallic loop runs and shows a 10 percent decrease in flow over 2800 hours of operation. The lower curve indicates flow behavior in the 316 SS loop during run 1-2 where the flow decrease became so severe that the run was discontinued at 2165 hours instead of the planned 2800 hours. Breaks in the lower curve correspond to partial flow recovery obtained after scheduled sample replacement; of

TEST CONDITIONS: NO OXIDE CONTROL EXCEPT AS NOTED.
TESTS CONDUCTED FOLLOWING RUN 1.

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FIGURE IV-2 PLUGGING TEMPERATURE vs TIME

FIGURE IV-3 SAMPLE HOLDER TEMPERATURE vs TIME

TABLE IV-2

OPERATING TEMPERATURE SUMMARY

Run No.	C3 Sample Holder			H3 Sample Holder		
	Tm	% Run Time		Tm	% Run Time	
		T = Tm ± 20 F	T = Tm ± 30 F		T = Tm ± 20 F	T = Tm ± 30 F
1-1	793	92	99	1297	94	97
1-2	704	97	100	1199	99	100
2-1	690	92	97	1181	69	90
2-2	698	93	100	1179	90	93
2-3	704	98	100	1200	100	100
2-4	700	98	99	1196	97	99
3-1	693	80	85	1198	90	95
3-2	707	83	99	1198	81	85
3-3	706	96	98	1199	100	100
3-4	702	98	100	1202	98	98
3-5	699	86	93	1202	98	98
3-6	706	94	97	1202	97	98
4-1	703	80	91	1192	84	94
4-2	699	100	100	1184	98	100
4-3	701	94	98	1192	90	97
5-1	695	80	91	1187	71	92
5-2	701	98	100	1197	98	99
6-1	696	66	79	1179	64	79
6-2	705	94	97	1197	95	99

T = Recorded temperature, °F

Tm = Mean temperature, °F

the six samples exchanged at each sampling period, three were exposed to cold leg specimens and replaced by three new specimens at 700 and at 1400 hours. This flow decrease is attributed to deposition of corrosion products in the cold leg resulting in increased pressure drop across sample flow passages. The standard sample cleaning procedure, consisting of reacting the sodium with ethanol followed by a water wash, was found to be effective in removing most of the deposit. Initial flow conditions during runs 1-1 and 5-1 were restored by washing all the cold leg samples and reinserting them in the loop.

Special runs with in-line filters have been conducted to collect fouling material for identification. The photograph in Figure IV-5 shows some of the fouling material collected in the cold

276-42

FIGURE IV-4 LOOP FLOW vs TEST TIME

277-40

PRE-EXPOSURE

1200 F 953 HR
POST-EXPOSURE

FIGURE IV-5 316 SS LOOP - COLD LEG - FOULING DEPOSIT

leg during run 1-1F with the 316 SS loop. An NH_3 washing process⁽¹⁾ was used to dissolve the sodium without removing the deposited material. X-ray diffraction data indicates that crystals collected on the screen filter are predominantly αFe . Chemical analysis also shows the presence of Cr, Ni and Mn in proportions which approximate the composition of stainless steel.

Curves showing loop flow versus test time for all runs are included in Appendix B of this Volume. A review of the flow change data listed in Table IV-3 indicates the following:

1. All the loops exhibited definite evidence of fouling.
2. Except for run 6-2 (and deleting the questionable run 3-1 flow change data) fouling was more severe at 50 ppm oxygen than at 10 ppm oxygen.
3. The monometallic loops (1, 5, 6) generally showed higher fouling rates than the bimetallic loops.

Sodium velocity past corrosion samples as a function of loop flow rate is indicated in Figure IV-6 for the inlet and outlet positions of each of the seven sample holders. Velocities were calculated on the basis of reference design dimensions for the samples and sample holder passages, the standard 26 sample loading (Figure III-4) and the temperature distribution where $C3 = 700\text{ F}$, $C2$ and $H1 = 1000\text{ F}$, $C1$ and $H2 = 1100\text{ F}$, and $H3$ and $H3R = 1200\text{ F}$. Deposition of mass transfer products on the cold leg sample holders and samples will increase the actual flow rate past these samples.

E. Sodium Impurity Analyses

Procedures for analyzing the various impurities in the sodium is reported in GEAP-4178.⁽²⁾ The analyses were performed by wet-chemical analytical techniques for better sensitivity at low impurity levels.

Table IV-4 presents the impurity levels in the "as-received" sodium, both commercial and reactor grade, and in three of the test loops after the initial runs. Note that the loop sodium and analyses, of sodium that was initially "commercial" sodium, compare favorably with the "Reactor Grade" impurity levels. The analyses of each sample taken during the test runs are reported in Appendix B. Table IV-5 presents a list of typical test loop sodium impurities. Figure IV-7 shows a plot of the loop 3 analyses which are typical of all the loops.

Most of the initial run chemical analysis data reported in Volume 3 for lithium and potassium in sodium were found to be in error. Initial procedures for these elements used a Didymium filter to absorb the sodium emission peaks but did not correct for the relatively high background of other wave lengths from the sodium emission. Consequently, earlier reported values for Li and, to a lesser extent, for K were higher than actual. To correct for this error the procedures were revised so that the emission was scanned with a recorder over the peak regions

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- (1) Alter, H. W. and McManus, P. A., "Sodium Mass Transfer: III The Application of Liquid Ammonia as a Leaching Agent," GEAP-4006, April 1962.
 - (2) Sabol, W. W., and Lockhart, R. W., "Sodium Mass Transfer: VII Methods for Determining Impurities in Sodium Samples," GEAP-4178, September 1963.

TABLE IV-3

SUMMARY OF FLOW CHANGE DURING TEST RUNS

Run No.	Average Flow gpm	Average Flow Change	
		gpm/wk	% Change/Wk*
1-1	0.40	-0.135	-33.7
1-2	0.56	-0.0371	- 6.62
2-1	0.70	-0.0042	- 0.60
2-2	0.60	-0.0360	- 6.00
2-3	0.26	-0.0043	- 1.65
2-4	0.24	-0.0041	- 1.71
3-1	0.53	+0.0003**	+ 0.06
3-2	0.52	-0.0191	- 3.67
3-3	0.29	-0.0114	- 3.93
3-5	0.90	-0.0550	- 6.11
3-6	0.72	-0.0184	- 2.55
4-1	0.41a.	+0.0000	+ 0.00
	b.	-0.0252	- 6.15
	c.	-0.0055	- 1.34
4-2	0.35	-0.0042	- 1.20
4-3	0.39	-0.0048	- 1.23
5-1	0.35	-0.109	-31.2
5-2	0.55	-0.0270	- 4.92
6-1	0.49a.	-0.0116	- 2.37
	b.	-0.117	-23.9
	c.	-0.0478	- 9.75
6-2	0.40	-0.0861	-21.5

NOTES: *Based upon average gpm for run.

**Change rate values are questionable due to incomplete flow data for first 900 hours of test run.

- a. Change during first 1600 to 2200 hrs.
- b. Change during last 600-1200 hrs.
- c. Overall average during run.

FIGURE IV-6 SAMPLE VELOCITY vs. LOOP FLOW RATE

FIGURE IV-7 SODIUM IMPURITY CONCENTRATION IN LOOP #3

to provide a measure of the background interference. Available spare samples were re-analyzed for Li and K. Representative analytical values before and after use of these modified procedures are shown in Table IV-6.

TABLE IV-4

SODIUM IMPURITIES

<u>Source of Sodium</u>	<u>Impurities, ppm</u>								
	<u>Ca</u>	<u>C</u>	<u>Cr</u>	<u>Fe</u>	<u>K</u>	<u>Li</u>	<u>Mn</u>	<u>Mo</u>	<u>Ni</u>
Commercial Grade	400	15	0.2	1.3	200	<13	2.8	0.6	1.7
DuPont Reactor Grade	<10	7	0.5	1.7	185	<8	1.7	0.3	0.4
Loop 3 (End of Run 1)	<12	-	0.2	0.6	-	-	1.4	0.4	1.2
Loop 4 (End of Run 1)	<16	-	0.3	1.4	-	-	1.1	0.6	1.7
Loop 1 (Run 2, 700 hr)	<10	15	0.8	3.5	280	<11	1.5	0.4	1.9

TABLE IV-5

TYPICAL TEST LOOP SODIUM IMPURITIES (ppm)

<u>Element</u>	<u>Sample 1-2-2</u>	<u>Typical Range</u>
C	15	4 - 29
Ca	<10	<20
Cr	0.8	0.1 - 3.0
Fe	3.5	1.0 - 10
K	280	129 - 366
Li	<11	<15
Mn	1.5	<3.0
Mo	0.4	0.2 - 1.5
Ni	1.9	0.5 - 2.5

TABLE IV-6

IMPROVED ANALYSES FOR Li AND K

<u>Sample</u>	<u>Li, ppm</u>		<u>K, ppm</u>	
	<u>Before</u>	<u>After</u>	<u>Before</u>	<u>After</u>
5-1-10	104	<9	550	314
4-1-8	130	<8	438	344
3-2-7	176	<5	368	165
3-2-8	207	<8	365	217

Of the impurities analyzed, calcium was the only one which showed a definite change with time and the only element which appeared different in the two grades of sodium (commercial grade and reactor grade). Corresponding to commercial grade sodium specifications, the initial samples contained about 400 ppm calcium. With continued operation the measured calcium concentration decreased until by the end of the initial run, values below the lower limit of detection (~ 10 ppm) were obtained. The reactor grade sodium has consistently been low in calcium.

In most analyses the measured iron and nickel concentrations were considerably below saturation values (~ 10 ppm at 700 F) calculated from reported solubility data.^(1, 2) A few of the early iron analyses, however, were considerably above the solubility limit, but these sodium samples usually had evidence of scattered black deposits which presumably contained undissolved iron. It is not apparent at this time that the impurity levels are related to the temperature, oxygen level or corrosion rate in the loop.

F. Sodium Leaks

Sodium leaks, ranging from a few grams to several pounds, have been encountered during test. In general, the leaks have been relatively infrequent and readily controlled so that operations were not seriously impaired.

One significant result of the sodium leaks observed was that corrosion on the air side of the structural steels is considerably more severe at temperatures above approximately 1000 F than at lower temperatures.

In the higher temperature range the corrosive attack appears catastrophic. For example, from leakage at 1300 F the magnitude of the corrosive attack was sufficient to penetrate the entire thickness of a two-inch schedule 40 pipe within a 1-hour period.

The following illustrations are typical examples of the appearance of the corrosive attack:

Figure IV-8a shows a 10-inch long section of a $2\frac{1}{4}$ Cr- 1 Mo steel H3R sample holder after a small sodium leak occurred at 1200 F. The damage took place within one day of operation. The leak was detected at a routine daily inspection of the flanges. It was not detected otherwise.

Figure IV-8b shows the components in Figure IV-8a after cleaning. The crescent-shaped piece is all that remained of a stainless steel drip pan ring. Note the deep grooves eroded by the leaking sodium, and the damage to the flange at the upper end. This damage was so severe that the sample holder was replaced.

Figure IV-9 shows a similar attack of a type 316 SS flanged connection which was operating at 1300 F. As in the previous case, this leak was discovered at the time of a routine inspection. This sample holder was repaired by replacing the flange.

⁽¹⁾ Liquid Metals Handbook (Sodium-NaK Suppl.) Jackson, C. B. (Ed.), (1955).

⁽²⁾ Rodgers, S. J., and Mausteller, J. W., Report MSA-TR-27, (1954).

FIGURE IV-8A 2 1/4 Cr SAMPLE HOLDER AFTER
SMALL FLANGE LEAK AT 1200 F

FIGURE IV-8B SAME AS FIGURE IV-8A AFTER
WASHING IN ALCOHOL AND WATER.

Figure IV-10 shows the equipment from Figure IV-9 after removing the clamp and cutting the flange off the holder. The deep grooves in the pipe flange body completely penetrate the 0.154-inch thick pipe wall at one point. Although it does not show in the photograph, the inner mating surfaces of the flange were not attacked.

Similar damage has been observed with 5 Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti steel and with A-285 stainless steel bolt material. In every case, the damage has been self-propagating, since the schedule 40 pipe walls were corroded through by what was initially an extremely small flange (gasket) leak. It should be noted that this severe corrosion of steels by leaking sodium occurs at temperature levels at which sodium is compatible with the inside of the stainless steel vessel walls; therefore, this leakage corrosion problem, rather than ordinary coolant corrosion, may be the factor which limits the application of sodium coolant at high temperature to steel systems operating in an air atmosphere.

To facilitate installation and removal of corrosion samples, mechanical joints were provided at each sample holder. Marman Conoseal joints were selected for this service because available data and operating experience indicated that this type of seal was satisfactory up to 1000 F and appeared superior to other mechanical joints for service up to 1300 F. However, during the early stages of test operation frequent sodium leakage was encountered with these joints at temperatures above 1000 F. Improvements in design, material, installation techniques and quality control (size of flange seats) were necessary in order to achieve reasonable reliability against sodium leakage in the higher temperature region. Table IV-7 shows a chronological listing of all the Conoseal joint leaks encountered through 12/31/61 with pertinent descriptive information.

The failure causes can be categorized into three main groups as follows:

1. Installation difficulties due primarily to incomplete vendor instructions. Since the first few months of operation, installation no longer presents any real problem. Following initial sodium leaks, Marman prepared more complete installation instructions with somewhat modified procedures. In addition, the operators have become more adept in the art of Conoseal joint installation.
2. Under-designed couplings made of formed sheet metal were originally supplied with all Conoseal units. Since leakage problems have been encountered, the vendor has re-evaluated the design and concluded that a heavy-duty coupling should have been supplied for service at 1000 F and higher. At the present time, the recommended heavy-duty couplings are in general use except for the minimum temperature regions.
3. Critical dimensions beyond specified tolerances - the sealing surface dimensions, including male and female flange diameters, were not measured in initial installation nor during early failures. However, since this kind of data has been accumulated, it appears to afford an excellent means of control. Nearly all recent leaks can be attributed to worn sealing surfaces which are grooved, scaled or exceed specified dimensional tolerances. External oxidation, scaling and general wear, particularly with the 2 $\frac{1}{4}$ Cr alloy at 1200 F, represent obvious causes for this problem.

FIGURE IV-9 LOOP 1 H3R SAMPLE HOLDER AFTER LEAK

FIGURE IV-10 H3R FLANGE WITH CLAMP REMOVED

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TABLE IV-7

CONOSEAL JOINT LEAK SUMMARY TO 12/31/61

Date	Loop	Flange Material	Location & Temp. °F	Probable Leak Cause	Corrective Action
7-9-60	1	316 SS	H1-1100	Gasket loading not uniform	Coupling centered
7-9-60	1	316 SS	C1-1200	Gasket loading not uniform	Coupling centered
7-12-60	1	316 SS	H3-1300	Gasket loading not uniform	Coupling centered
7-13-60	1	316 SS	H3R-1300	Low temperature bolt mat'l	Changed bolt material, new 316 SS cap
8-14-60	1	316 SS	H3R-1300	Gasket loading insufficient	Replaced flanges; increased bolt torque
9-11-60	1	316 SS	H3-1300	Weak coupling	Replaced flanges & coupling (heavy duty type)
10-22-60	1	316 SS	H3R-1300	Gasket loading insufficient	New SS flanges and coupling
10-22-60	1	316 SS	C1-1200	Gasket loading insufficient	New SS flanges and coupling
11-5-60	2	316 SS	H3-1200	Weak coupling	Replaced coupling (heavy duty type)
11-5-60	5	2 $\frac{1}{4}$ Cr	H3R-1200	Weak coupling	Replaced flanges & coupling (heavy duty type)
11-26-60	5	2 $\frac{1}{4}$ Cr	H1-1000	Leak cause unknown	Rebuilt female flanges & reused
11-28-60	5	2 $\frac{1}{4}$ Cr	H3-1200	Weak coupling	Replaced coupling (heavy duty type)
					Replaced flanges with chrome plated 2 $\frac{1}{4}$ Cr.
12-13-60	1	316 SS	H3-1300	Excessive flange wear	New SS flanges
12-18-60	5	2 $\frac{1}{4}$ Cr	H3-1200	Scaling of Cr plate	Cleaned and reused
1-20-61	5	2 $\frac{1}{4}$ Cr	H3-1200	Excessive flange wear	Rebuilt female flange
2-13-61	5	2 $\frac{1}{4}$ Cr	H3-1200	Male flange undersize	Replaced flange
2-19-61	6	5 Cr	H3-1200	Excessive flange wear	Replaced-5Cr flanges coated with aluminum paint
2-21-61	5	2 $\frac{1}{4}$ Cr	C2-1000	Female flange grooved	Rebuilt flanges with 316 SS weld in gasket area
2-28-61	6	2 $\frac{1}{4}$ Cr	H3R-1200	Worn sealing surface	Rebuilt flanges with 316 SS weld in gasket area
3-1-61	1	316 SS	H3-1300	Sealing surface scored	New SS flanges, better quality control
3-2-61	5	2 $\frac{1}{4}$ Cr	C1-1100	Excessive flange wear	Replaced flanges - 316 SS with 2 $\frac{1}{4}$ Cr liner
3-5-61	4	316 SS	H2-1100	Male flange undersize	Replaced flanges
3-18-61	2	316 SS	H1-1000	Cause unknown	Replaced flanges
5-13-61	2	316 SS	H3R-1200	Excessive flange wear	Replaced flanges
5-25-61	5	2 $\frac{1}{4}$ Cr	C2-1000	Excessive flange wear	Replaced flanges - 316 SS with 2 $\frac{1}{4}$ Cr liner
6-6-61	6	5 Cr	H2-1100	Excessive flange wear	Replaced flanges - 316 SS with 5 Cr liner
6-19-61	1	316 SS	H3-1300	Crack in metal	Replaced flanges
8-9-61	3	2 $\frac{1}{4}$ Cr	C2-1000	Male flange undersize	Replaced flanges - 316 SS with 2 $\frac{1}{4}$ Cr liner
9-4-61	6	5 Cr	H3-1200	Female flange at tolerance limit	Replaced female flanges - 316 SS with 2 $\frac{1}{4}$ Cr liner
10-30-61	6	5 Cr	H1-1000	Scaling and flange wear	Replaced flanges - 316 SS with 5 Cr liner

The first two types of difficulties have been essentially eliminated, but the wear and scaling of flange surfaces will undoubtedly continue. However, leakage from this problem source has been minimized by subjecting each flange to a routine quality control inspection, including male and female dimensional checks during each opening cycle. The condition of each flange can thus be assured of satisfying vendor specifications and when sufficient wear is evident, replacement can be made during a normal down cycle to prevent possible later leakage. These dimensional controls and inspection procedures were initiated in April, 1961. At this time a decision was made to convert the ferritic steel flanges, as replacement became necessary, with stainless steel joints which were lined, on the sodium side, with the appropriate ferritic material. This latter change was based upon two factors: (1) excessive external, or air-side, oxidation and scaling at the higher temperature, and (2) stress calculations by the vendor which showed that the female flange design was questionable for 1200 F operation with the ferritic steel materials.

Figure IV-11 illustrates the results of the preceding modifications and control. The significant drop in frequency of flange leaks is principally attributed to the replacement of worn Conoseal joints before they attain the condition resulting in sodium leaks. Table IV-8 summarizes the accumulated test time and number of use cycles for each sample holder Conoseal unit currently in service.

TABLE IV-8

CURRENT STATUS OF LOOP CONOSEAL JOINTS (12/31/61)

Loop	No. 1		No. 2		No. 3		No. 4		No. 5		No. 6	
Holder	C*	T**	C*	T**	C*	T**	C*	T**	C*	T**	C*	T**
H1	15	7000	12	7700	15	8170	13	8960	9	4590	1	1570
H2	9	7000	9	7700	14	8170	10	8170	6	2510	1	3110
H3	4	2260	1	2800	15	8170	7	4940	3	3010	10	8170
H3R	7	6260	1	2800	5	2950	3	1740	6	3230	4	5260
C1	14	6330	4	4430	2	1520	3	1740	6	3230	3	2970
C2	12	7000	1	2800	3	2950	11	8960	3	3230	5	8170
C3	15	7000	13	7700	15	8170	13	8960	10	5970	10	8170

*C = Number of use cycles or number of times joint is opened and closed.

**T = Number of test hours exposure to circulating sodium.

The sources of sodium leakage and the number of leaks from each source are listed below:

Source	Number of Leaks
Conoseal Joints	29
Valve (bellows)	8
Pump Cell	2
Heater	2
Piping	6

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FIGURE IV-11 CONOSEAL JOINT LEAKS

The joint leaks have previously been described. All of the valve leaks resulted from bellows failures and occurred prior to run 2. Failures were attributed to possible operation while the sodium was frozen or application of excessive torque to the bellows in initial installation. Installation and operation procedures were accordingly modified to minimize the risk of recurrence.

Porosity in the 5 Cr steel fittings accounted for two of the piping leaks. Evidence for the causes of the remaining failures is inconclusive; however, it is likely that occlusions or similar hidden flaws may have been responsible. Such faults would escape the mass spectrometer and dye check tests used during construction.

The first pump cell leak occurred during initial startup of the 316 SS - $2\frac{1}{4}$ Cr loop (No. 2). Subsequent metallographic examination of the failure in the $2\frac{1}{4}$ Cr-1 Mo tube section is discussed in Appendix C. The actual rupture in the cell was masked by the corroding action of the burning sodium and, therefore, interfered with diagnosis of the cause. However, it was suspected that a subsurface crack in the braze joint or the original tubing contributed to the failure. In an attempt to minimize the risk of recurrent leaks the decision was made to x-ray each pump cell in the brazed joint region for detection of subsurface cracks or braze porosity.

The second pump cell leak, in the 316 SS loop (No. 1) after 587 hours operation with 800 F sodium, was the result of a pinhole failure in the 3/8-inch o.d., 0.049-inch wall tubing. The hole was patched by welding after completion of dye penetrant and x-ray tests the cell was successfully reused.

On two different occasions main heaters leaked sodium. Both leaks occurred in the 5 Cr loop (No. 6); the first in the H3 heater after 2862 operating hours, and the second in the H2 heater after 5530 operating hours. In the H3 heater during test sodium entered at 1100 F and discharged at 1200 F. In the H2 heater the inlet sodium temperature was 1000 F and the outlet 1100 F. The leaks were detected only after the heater electrical circuit breaker tripped. Inspection of the high temperature heater leads showed evidence of sodium at the heater elements. The single, faulty tube section from the second leak was subjected to a comprehensive metallurgical examination and the results are reported in Appendix C.

V. RADIOACTIVE TRACER ANALYSIS (By T. N. Lee, H. E. Dodge, R. W. Lockhart and W. W. Sabol)

- V. A. Introduction
- V. B. Procedures
- V. C. Results
- V. D. Individual Elements
- V. E. Mass Balance
- V. F. Run Analysis
- V. G. Conclusions and Recommendations.

A. Introduction

The objective for using the radioactive tracer techniques in this program was to assist in the evaluation of corrosion and/or mass transfer (should weight change measurement not provide sufficient information) and to attempt to determine whether the steels were transferred as an alloy, or by selective movement of the alloying elements. The technique used consisted of activating foils of program materials in the G. E. Test Reactor (GETR) and then exposing them to sodium in the special outlet slot of the H3R sample holder. The foils were nondestructively counted with a multichannel pulse height analyzer before and after exposure. All other corrosion samples and the sodium were analyzed after the test run was completed.

The first of the objectives became unnecessary when actual corrosion sample weight changes were shown to be large enough to be measured with considerable accuracy. The second objective has been met since the data clearly shows that some of the ingredients of stainless steel move selectively. These results, however, were limited due to the following reasons:

- a. The scatter of the data was large and an isotopic mass balance could not be made.
- b. Of the three major components of Type 316 stainless steel, only chromium was tagged well enough to trace its path throughout the loop. Unfortunately the Co^{60} and Fe^{59} activity of the foil samples could not be separated with the nondestructive multichannel pulse height counter technique. As reported in GEAP-3725, radiochemical separations were used to separate these isotopes in the foil but the sample was destroyed so further metallurgical examinations would not be carried out.
- c. The foil samples were exposed to sodium in a region of the loop (H3R sample holder) where the corrosion rate was so low that it seems likely that exchange of atoms rather than mass transfer may account for a significant portion of the activity observed in the loop.

B. Procedures

Gamma scintillation spectrometry provided the basis for all activity measurements. In cases where radiochemical separations were made, counts were obtained on a single channel analyzer, using a $1\frac{1}{2} \times 2$ -inch NaI(Tl) crystal which contained a $\frac{1}{2}$ -inch diameter well. Where non-destructive measurements were made, a 256 channel pulse height analyzer and a 2×3 -inch flat-faced NaI(Tl) crystal were used to obtain the gross gamma spectra. In most cases, the

count rates were compared to those obtained from sources containing known amounts of radioactivity. All data were reduced to absolute disintegration rate and decay corrected to a common date.

The aluminum irradiation capsules were fabricated to hold the steel foil samples during irradiation in the General Electric Test Reactor (GETR). The capsule drawing number is APED 693C283 and the capsules were made of Type 1100-0 aluminum. Figure V-1 shows a photograph of capsule MT-4. This capsule is typical of the four capsules that were irradiated; as shown, this capsule contained five Type 316 stainless steel foil samples. All foil samples were folded from 3/16 inch wide by 3 inches long by approximately 6 mils thick sheet material. Table V-1 contains the irradiation information for capsule MT-4 as well as the other capsules used in the radio-tracer work. After irradiation, the samples in MT-1 and the 5 Cr samples in MT-2 were analyzed radiochemically to determine if the foils contained tramp impurities. These results were reported in GEAP-3725, Appendix C. One of the stainless steel samples irradiated in MT-2 was exposed to sodium in run 1-1 and the other sample was stored and then counted at nearly the same time as the exposed sample. Thus the difference in count rates was caused by the sodium exposure. Unfortunately, the MT-3 capsule was lost so MT-4 was used as a replacement. All five Type 316 stainless steel foils in this capsule were scanned and then individually shipped to San Jose, Building D, for sodium exposure.

TABLE V-1
IRRADIATION CAPSULE DATA

Capsule No.	No. of Foils	Foil Material	GETR Cycle No.	Position in GETR	Inches from Core Base	Thermal Flux nv	Estimated nvt	GETR MWD
MT-1	2	316 SS	10	X-1	19	1.2×10^{14}	2.66×10^{20}	772.8
	2	2 $\frac{1}{4}$ Cr						
MT-2	2	316 SS	14	X-7	15	1.4×10^{14}	2.71×10^{20}	662.0
	2	5 Cr						
MT-3	4	316 SS	20	G-6	18	2.8×10^{14}	1.03×10^{20} (capsule lost)	128.1
				Y-9	14			
MT-4	5	316 SS	21	E-5	16	2.4×10^{14}	1.13×10^{20}	163.7

The activated foils were loaded into sample holder H3R during test runs 1-1, 1-2, 2-2, 2-3, 2-4, and 4-2. After the foil was exposed to flowing sodium in the test loop, the foil, samples of the sodium and the steel corrosion samples were analyzed for their radioactive content. The individual foil sample activation and sodium exposure information is given in Table V-2.

When the corrosion samples were removed from the loop, the sodium on the surfaces was carefully washed with alcohol and water. Both the samples and washes (some times) were analyzed for activity. Chemical separations were performed on the washes before counting, and gross spectra were obtained on the corrosion samples. The chemical separations for the specific elements were made as specified in GEAP-3725, Appendix B and/or in GEAP-4178.

TABLE V-2
ACTIVATED FOIL SUMMARY

Radiochemical Analysis

Foil No.	Material	Total Activity, mc/gram*			
		Cr-51	Mn-54	Fe-59	Co-60
MT-1 A	316 SS	1882	0.272	22.2	9.06
B	316 SS	1885	0.271	22.8	8.89
C	2 $\frac{1}{4}$ Cr	243	0.376	35.9	0.666
D	2 $\frac{1}{4}$ Cr	237	0.376	34.4	0.683
MT-2 C	5 Cr	541	0.678	26.8	0.563
D	5 Cr	536	0.851	26.4	0.326

*Corrected for in-pile and out-of-pile decay

Multichannel Pulse Height Analyzer

Foil No. ***	Material	Na Exposure During Run No.	Period Exposed to Sodium	Weight, g		Activity Before Na Exposure, $\mu\text{c}/\text{Foil}^{**}$			Activity After Na Exposure, $\mu\text{c}/\text{Foil}^{**}$			No. of Corrosion Samples In Loop
				Before	After	Cr-51	Mn-54	Co-60+ Fe-59	Cr-51	Mn-54	Co-60+ Fe-59	
MT-2 A	316 SS ↑	1-1	12/60 - 5/61	0.5168	0.5694	---	---	---	1485	359	1710	37
-2 B		None	--	0.5744	---	1718	363	1790	---	---	---	--
MT-4-1	316 SS ↓	2-2	5/61 - 7/61	0.5781	0.5641	---	---	---	1.54×10^5	2300	2440	7
-2		4-2	5/61 - 9/61	0.5505	0.5472	---	---	---	---	---	---	35
-3		2-3	7/61 - 8/61	0.5679	0.5641	2.19×10^5	710	2200	---	---	---	7
-4		1-2	8/61 - 11/61	0.5566	0.5400	2.13×10^5	2770	2650	---	---	---	41
-5		2-4	8/61 - 12/61	0.5636	0.5590	2.34×10^5	2530	3080	---	---	---	11

**All activities corrected to June 1, 1961

***Foil were identified with end notches

Note: The MT-2 foils were activated about seven months prior to the MT-4 foils so the short lived activities such as Cr-51 appear much lower in the MT-2 foils.

GEAP-3726

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~0.4X

**FIGURE V-1 MT-4 IRRADIATION CAPSULE WITH FIVE
316 SS SAMPLES PRIOR TO ASSEMBLY**

The counting method used on all corrosion samples except the foils were capable of ± 10 percent precision when activity levels greater than 100 times background were present. The foils, on the other hand, presented a problem since they were highly radioactive and had to be counted as far away as 4 to 6 feet away from the crystal in order to obtain meaningful spectra. Removal of a piece of the foil for counting close to the crystal was not desirable. Known standards of corresponding high source strength were not available, and standardizations were made from attenuation factors derived by counting the available standards at the same distance. Although long count times were used the precision of the foil count normalized from such standardization is estimated to be on the order of 20-30 percent. The initial or before-exposure activation data, therefore, is not very satisfactory.

As the count rate (or any of the samples) approached counter background levels, precision became poorer. Counting error was magnified when a large decay-time correction was applied to normalize all data to a common date. In addition separating the Fe^{59} activity from the Co^{60} with gross spectra was improved by spectrum stripping techniques when the Fe^{59} to Co^{60} ratio was high, but for these samples the ratio was relatively low after long sodium exposures.

C. Results

The results of the analysis of irradiated foils of each of the program materials used to determine the usable isotopes for radio-tracer work are contained in GEAP-3725 - Appendix C. The analysis reports for the irradiated foils and corrosion samples from test runs 1-1, 1-2, 1-3, 2-2, 2-3, 2-4 and 4-2 are contained in Volume 3 of this report (GEAP-3726).

The data showed that the activity of the corrosion samples decreased with distance around the loop from the source; however, the activity of sample wash liquid is significantly higher in the C3 position than for those samples exposed in C1 or C2 position. Apparently some of the surface deposits are not as adherent (or are more soluble) when formed at lower temperatures in the cold leg. Over 90 percent of the measured activity in all of the hot leg samples remains on the samples, while only about 50 percent of the total activity remains on the samples exposed at the minimum temperature region in the C3 holder.

For example, a special experiment was performed at the end of run 4-2 in an attempt to determine how much deposited material (activity) was "washed" off of the C3 samples during ammonia extraction and alcohol-water washing. Corrosion samples to be washed, No. 4912, 4916, and 4918, had been exposed in the inlet passage of the C3 sample holder in Loop 4 for the last 2803, 2074 and 1314 hours respectively, of the run. An activated foil, MT-4-2, was located in the H3R holder for the complete 2800 hour test run. The samples were removed from the loop and transferred to VAL for the cleaning, weighing and counting. The data in the following table were obtained by weighing the samples separately but counting them as a group with the multi-channel pulse height analyzer.

TABLE V-3
SAMPLE COUNT RATE DURING CLEANING

	Total Activity, * dpm/sample				
	<u>Cr⁵¹</u>	<u>Co⁵⁸</u>	<u>Mn⁵⁴</u>	<u>Fe⁵⁹</u>	<u>Co⁶⁰</u>
A. Before NH ₃ extraction	8.67×10^7	9.33×10^5	5.92×10^5	1.86×10^6	1.60×10^5
B. After NH ₃ extraction	5.45×10^7	7.03×10^5	4.23×10^5	1.65×10^6	1.40×10^5
Percent Removed, (A-B/A) × 100	27%	21%	27%	11%	12%
C. After Alcohol Water Wash	4.19×10^7	5.00×10^5	3.16×10^5	1.18×10^6	0.932×10^4
Percent Removed, (B-C/B) × 100	23%	29%	25%	28%	33%
Total Percent Removed, (A-C/A) × 100	52%	36%	36%	27%	32%

*All dpm values were decay-corrected to June 1, 1961.

	<u>C3 Sample Weights, gms</u>	
<u>Sample No.</u>	<u>After NH₃ Extraction</u> (appeared wet)	<u>After Alcohol-Water Wash</u> (dried)
4912	4.09356	4.07454
4916	4.12650	4.07565
4918	4.07197	3.99728

The NH₃ extraction procedures and equipment are reported in GEAP-4006, "SMT: III The Application of Liquid Ammonia as a Sodium Leaching Agent." The alcohol-water wash and drying procedures followed the projects standard method. Both of the washes apparently removed significant amounts of the transferred material. There is some indication the NH₃ extraction was more selective to the removal of Cr⁵⁴, Co⁵⁸ and Mn⁵⁴ than to Fe⁵⁹ and Co⁶⁰.

The variation in types of activity observed on the corrosion samples around the loop indicated not only selective corrosion of certain components of steel, but also selective deposition in given temperature regions. Selective corrosion of certain components of steel could result in corrosion damage completely out of proportion to the weight loss. This may very well be the significant criterion for the usefulness of certain materials for sodium systems.

The fact that there was activity buildup on samples which showed a weight loss gives some indication of the complex nature of the mass transfer mechanism. There may have been an interchange of atoms. Since the separated Co⁶⁰ and Fe⁵⁹ activities of many of the foil samples is unavailable, the use of the activity ratios has provided some useful information. On the average the Cr⁵¹ activity is about 78 times that of the iron plus cobalt activity. Correcting this for the fact that one dpm of iron does not equal one cpm based on the assumption that the Co/Fe ratio of the foil is the same as that of the other samples, we arrive at a true ratio of Cr to Fe plus Co of about 40/1. Similarly we can arrive at an average ratio of Cr to Mn of about 70/1.

D. Individual Elements

It was possible to determine where the various elements existed around the loop by comparing changes in ratios of their activities on various samples. The manganese appeared to migrate toward the hot leg where it was 10 to 250 times more abundant, relative to the other tracers, on the corrosion samples than on the irradiated foil. Enough manganese appeared in the coldest, C3, samples to indicate that the sodium may be saturated at this temperature. The chromium seemed to be deposited fairly uniformly throughout the loop. The iron appears to go primarily to the coolest samples while the cobalt appears uniformly distributed through the cold leg.

E. Mass Balance

At one time it was hoped that a material transfer mass balance around the loop might be made utilizing the radioactive tracer techniques. Similar to the weight change data, this was not the case. The accuracy of activity measurements of the samples (about 30 percent) is significantly lower than the accuracy of the sample weight change (about 1 percent). The one exception may be Cr^{51} , which shows about a 30 percent decrease from the mean ratio value of the foil samples. Since this is the only isotope showing any significant change in the cold leg, we would expect the chromium count on the test coupons to be relatively higher (compared to the other isotopes) than they are on the foil itself.

F. Individual Run Analyses

Run 2-2

Figures V-2, V-3, and V-4 show the activities measured on the corrosion samples as a function of the sample weight change. Unfortunately there were so few points (seven samples) available in this run, that the curves must of necessity be rather unreliable. Figure V-2 shows manganese weight change in both the hot and cold legs. From the points plotted on the curve, it may be seen that the hot leg data seems far less scattered than the cold leg, a phenomenon that was noted when the run 1-1 data was plotted the same way. (This holds true for all the other radio-isotopes and can be seen in the run 4-2 data as well.) Unlike run 1-1, however, the activity in the hot leg increases as the weight loss increases. This can be due in part to the short duration of the test run, but most likely is a result of the very low corrosion rates observed during this run.

Figure V-3 shows the activities of the various isotopes in the cold leg. The scatter of the data in all cases was about as great as that of the manganese, hence the slopes of these lines cannot be assumed to be too reliable. The ratio of the Cr^{51} activity to the Co^{60} and Fe^{59} ranges from 120-300 to 1. This ratio is about five times as high as it is on the foil, and bears out the assumption that the bulk of the material transferred must be chromium. Similarly, the ratio of chromium to manganese activities is also about 250 to 1, or about three times as high as on the foil. This again shows the chromium moving very rapidly, but tends to indicate more mobility for the manganese than for the iron or cobalt. While this does not confirm the order of mobility seen in run 1-1, there is reason to suspect the values of the foil activity in that run.

Figure V-4 shows activities measured in the hot leg as a function of weight change. All the isotopes seem to show the same (wrong for activity to be proportional to sample weight loss)

FIGURE V-2 MANGANESE ACTIVITY Vs. COUPON WEIGHT CHANGE - RUN 2-2

FIGURE V-3 COUNT DATA FOR COLD LEG - RUN 2-2

FIGURE V-4 COUPON ACTIVITY V's. WEIGHT CHANGE - RUN 2-2 HOT LEG

slope as the manganese. Again the chromium activity ratio is about one order of magnitude higher than on the foil. In this case, however, the manganese activity is lower than expected from the previous data. The bulk of the isotopes appears in the cold leg, indicating that there is little left in the sodium for deposition in the hot leg.

Run 4-2

Figures V-5, V-6 and V-7 are similar curves for run 4-2. Since they are based on more data points (samples), they are perhaps a little more reliable, but again cold leg activities show a wide scatter. The conclusions are similar to those in run 2-2. There are two points of difference, however:

1. Manganese activity in the hot leg is a good deal higher compared to the chromium. This might be due in part to the fact that the exposure time is four times as long (2800 hours).
2. The cobalt is the only element showing a "reversed" slope in the hot leg.

This again may be due to the longer exposure time. It was previously noted that the worth of some of the radiochemical data generated became of marginal value with long exposure.

While the counts reported in the tables appear to be of a high order of magnitude, they were corrected to June 1, 1961. The observed counting rates were on the order of 2 to 3 times counter background. This was especially true of the shorter lived, $^{27}\text{d. Cr}^{51}$ and $^{45}\text{d. Fe}^{59}$ activities.

The Fe^{59} data was not obtainable in the examination of the gross gamma spectra of coupons from runs 1-2, 2-3, and 4-2. The Co^{60} present, about 100 to 500 times the Fe^{59} , masked the Fe^{59} photopeak to the degree that meaningful data could not be developed. The Cr^{51} data obtained with low counting rates was still believed to be meaningful.

In all cases the activity of the wash solutions had been at least one order of magnitude too large to be accounted for by the activity of the sodium. It was therefore evident that some of the surface of the sample was being removed during the washing process. The amount of material removed must be small, however, since the activity of the wash was significantly lower than the sample activity. In the coldest portion of the cold leg, however, the wash activities became large, indicating that the deposits formed at 700-800 F in a 1200 or 1300 F loop were not very adherent. Or, there may be significantly larger deposits of "corrosion" products on the low temperature samples than post cleaning weight change data would indicate. Finally, the activity levels of the hot leg samples which were losing weight were only about one order of magnitude lower than the corresponding activities in the cold leg. This seems to indicate that random interchange of atoms was playing a significant role in the mass transfer process.

The activation data from run 1-2 showed none of the correlation between activity and weight change which had been observed for the other runs. This is especially true in the hot leg where the correlation had been excellent in the past. In the cold leg there was an increase of activity with increasing weight gain. Sufficient scatter existed in the data, however, so that sample weight change could not be predicted within a factor of two by activity readings.

FIGURE V-5 MANGANESE ACTIVITY Vs. SAMPLE WEIGHT CHANGE

FIGURE V-6 ACTIVITY Vs. WEIGHT CHANGE - RUN 4-2 COLD LEG SAMPLES

FIGURE V-7

ACTIVITY VS. WEIGHT CHANGE - RUN 4-2 HOT LEG SAMPLES

G. Conclusions

- (1) In the bimetallic loops the Type 316 stainless steel will lose chromium and manganese from the H3R position.
- (2) The data clearly shows that some of the ingredients of stainless steel are moving selectively.
- (3) The selective loss of elements may be a surface effect and not cause severe damage to the base structure of thicker material.
- (4) The attack may be less selective in other parts of the loop since the foil samples were exposed in a region of extremely low corrosion rate.
- (5) Random interchange of atoms is evidently playing a role in the mass transfer process.
- (6) Wash fluid activity from the samples exposed in the coldest sample holder is sufficiently high to cast some doubt on the validity of weight change data as a measure of deposition ("crud" buildup) in this region.
- (7) Future runs could be made with activated foils located in a more corrosive region, such as in the H3 holder, however, until more satisfactory equipment is available to measure the foil activity before and after sodium exposure or, preferably, if the steel foils were prepared with only one activated isotope, the use of radioactive tracers does not appear to be as useful as other techniques for our work. It is, therefore, recommended that further work along this line be discontinued at present. The use of radio-tracer techniques should be reconsidered at the end of the five-run fractional replicate test plan to determine if more information on transfer of select elements around the sodium system is required.

VI. CORROSION DATA ANALYSIS by D. W. Gaylor, E. G. Dunn and L. F. Epstein

- VI A Introduction
- VI B Experimental Design
- VI C Models and Analysis Techniques
- VI D Results: Hot Leg
- VI E Results: Cold Leg
- VI F Discussion and Analysis

A. Introduction

With the large number of factors affecting corrosion rates and the volume of data necessary to study these factors, it was considered essential to use statistical techniques to analyze the data in an effort to summarize the results in a simple and useable form. The primary statistical tool used here was a least squares multiple regression analysis. Good data analysis implies that data must exist which lend themselves to analysis. Hence, statistical requirements have been considered in planning future runs in an effort to separate the effects of the many factors influencing corrosion rates.

B. Experimental Design

It was presumed from theory and experience that the following factors might influence corrosion: loop material, oxygen concentration in the sodium, time (length of exposure), sodium velocity, and temperature, including $\Delta T = T_{\max} - T_{\min}$ and $T_{\max}$. Analyses of the data collected to date have indicated the presence of two additional factors: age and downstreamness (or loop position in an isothermal region). The age factor was discovered by observing that samples exposed during the first half of a run consistently showed lower corrosion rates than samples exposed during the last half of the run. Downstreamness is a function of the location of a sample in the loop. It was observed that samples in H3R showed consistently lower corrosion rates than H3 samples which were located upstream. Also, within the same sample holder, outlet samples consistently show lower corrosion rates than inlet samples.

The values for oxygen concentration, $T_{\max}$, and ΔT must be set at the beginning of a run and maintained as nearly constant as possible throughout a run. An experimental plan (Table VI-1) which includes a one-half fractional replicate of a 2^3 experiment, is being used to estimate the linear effects of the factors ΔT , $T_{\max}$, and oxygen level, which are varied on a run to run basis.

The plan in Table VI-1 provides experimental symmetry of the primary variable conditions to prevent confounding the results among these three factors. For example, confounding would occur between oxygen and $T_{\max}$ if they were always increased or decreased simultaneously from run to run. Then, it would not be possible to determine how much of an observed difference between runs to assign to oxygen or $T_{\max}$.

TABLE VI-1

FRACTIONAL REPLICATE PLAN FOR STUDYING ΔT , $T_{\max}$, AND OXYGEN

Oxygen	45 ppm		12 ppm	
ΔT	500 F	250 F	500 F	250 F
$T_{\max} = 1200 \text{ F}$	Run Type I	*	Run Type II	Run Type IV
$T_{\max} = 1100 \text{ F}$	*	Run Type V	Run Type III	*

*Conditions that may be needed to check interactions among these three factors.

When the five runs are completed for each loop, the linear effects of ΔT , $T_{\max}$, and oxygen level can be estimated. Meanwhile, run Type I versus run Type II data gives an estimate of the effect of oxygen. This estimate will be more precise when the five runs are completed. Run II versus run III will provide a direct estimate of the $T_{\max}$ effect. Run Type II versus run Type IV will give an estimate of the effect of ΔT . The order of operating runs Types III, IV, and V, could be interchanged. Runs Types I and II have been completed on all loops except for run Type I in loops 1 and 2. The three blanks (*) in Table VI-1 would have to be completed to check for interactions among these three variables. For example, the presence of an oxygen - ΔT interaction would mean that the effect of oxygen is different for different values of ΔT .

Secondary variables, time, velocity, internal temperature, age, and downstreamness are varied and their effects are estimable within each run. Different times and ages are obtained by removing and replacing the three-slot samples after different lengths of exposure during a run. See the sample removal pattern in Table VI-2. As a first approximation to age, the mid-point of the time the sample was exposed was used. For example, sample No. 2 is inserted in the slot vacated by sample No. 1 after 700 hours of a run. Sample No. 2 is removed at the end of the 2800 hour run giving a mean run age of $(700 + 2800)/2 = 1750$ hours for sample No. 2. Age is defined as

$$a = (t_1 + t_2)/2 \quad (1)$$

where the run has been conducted for t_1 hours when the sample is inserted and for t_2 hours when the sample is removed.

Different velocities are obtained from one-slot holders, three-slot holders and pipe samples. Sample holders are located at three different temperatures in each leg of the loops. Different downstream values are obtained from the inlet-outlet samples within a holder, H3 versus H3R samples, and pipe samples.

Runs in addition to those listed in Table VI-1 are necessary to study age, temperature profile and downstream effects to help interpret and extrapolate the results obtained from the runs presented in this table.

TABLE VI-2

SAMPLE INSERTION-REMOVAL PATTERN FOR THREE-SLOT SAMPLE HOLDER

<u>Slot</u>	<u>Sample</u>	<u>Inserted at</u>	<u>Removed at</u>	<u>Exposure Time</u>	<u>Age</u>
1	No. 1	0 hrs	700 hrs	700 hrs	350 hrs
	No. 2	700 hrs	2800 hrs	2100 hrs	1750 hrs
2	No. 3	0 hrs	1400 hrs	1400 hrs	700 hrs
	No. 4	1400 hrs	2800 hrs	1400 hrs	2100 hrs
3	No. 5	0 hrs	2800 hrs	2800 hrs	1400 hrs

Some special "replicate" runs described in Table VI-3 were conducted on loops 2 and 3 with run Type II conditions. These "replicate" runs had a three-fold purpose.

1. To obtain high velocities in the cold leg by using the 7 sample loading pattern. Thus, the cold leg velocity effect could be estimated. Then, corrections to the same velocity can be made when comparing results from different loops or different runs.
2. To determine loop-to-loop reproducibility of results. This was possible since loops 2 and 3 are made of the same material. This information is necessary when comparing different materials from the different loops.
3. To determine run to run reproducibility of results on the same loop. Obviously, reproducibility is necessary when comparing different test conditions from different runs.

TABLE VI-3

REPLICATE TYPE II RUNS

(12 ppm oxygen, $T_{\max} = 1200$ F, and $\Delta T = 500$ F and seven sample loading pattern)

<u>Loop</u>	<u>Run</u>	<u>Time</u>	<u>Velocity</u>
2	2	700 hrs	21 fps
2	3	700 hrs	7 fps
2	4	2800 hrs	7 fps
3	3	700 hrs	7 fps
3	4	700 hrs	21 fps
3	5	700 hrs	30 fps
3	6	2800 hrs	21 fps

Some special "Downstream" or "HC" runs have been initiated on the $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$ loop (No. 4). The purpose of these runs was to expose as many samples as possible in one temperature zone. Using Type II test conditions (of $T_{\text{max}} = 1200\text{ F}$, $\Delta T = 500\text{ F}$ and 10 ppm oxygen level) the entire hot leg was operated at T_{max} and the entire cold leg was operated as close as possible to 700 F . This provided the normal 14 samples in the hot leg at one temperature and more than doubled the "downstreamness" of the samples in H3R. Cooler capacity limitations resulted in the C1 sample holder operating at about 800 F but 9 of the 12 cold leg samples operated at 700 F .

As other variables become apparent and/or test scheduling permits some additional special runs will be required to evaluate the significance of recognized and suspected variables affecting mass transfer.

C. Models and Analysis Techniques

The general analytical technique has been to subject the data to least squares multiple regression analyses. This makes it possible to analyze the data where the target values for oxygen, velocity, time, and temperature were not and could not be strictly adhered to. This technique simply fits the best curve or surface through the data points, relating corrosion rates to exposure time, velocity, oxygen, temperature, and position effects. The factorial type experimental plan, Table VI-1 was followed to prevent confounding of the ΔT , T_{max} , and oxygen effects.

Employing the Philco 2000 Computer has made it possible to try a large number of different types of models recently. This computer is particularly well adapted to matrix inversion required by the least squares multiple regression analysis. Mr. C. L. Peterson, Technical and Business Computation Services, APED, developed a versatile program for handling the data very quickly and simply.

The model currently being considered is

$$|R_i| = \beta_0 (\Delta T)_i^{\beta_1} (t_i)^{\beta_2} (v_i)^{\beta_3} (a_i)^{\beta_4} \exp \left[\frac{\beta_5}{T_{m_i} + 460} + \frac{\beta_6}{p_i + 460} + \frac{\beta_7}{T_i + 460} + \beta_8 D_i \right] \quad (2)$$

where the β 's are parameters to be estimated from the data, R_i is the absolute value of the rate of weight change for the i th sample expressed in $\text{mg}/\text{dm}^2\text{-mo}$, $(\Delta T)_i$ is the difference between the maximum and minimum temperature in the loop while the i th sample was exposed, t_i is the length of time in hours the i th sample was exposed in the loop, v_i is the average sodium velocity in ft/sec past the i th sample, a_i is the age of the i th sample defined by equation (1), T_{m_i} is the maximum temperature in the loop in which the i th sample was exposed, p_i is the average plugging temperature in $^{\circ}\text{F}$ during the exposure of the i th sample, T_i is the temperature in $^{\circ}\text{F}$, and D_{k_i} is a downstream value. Several downstream functions were tried.

$$D_{2_i} = \sum_j (\text{Re})_j S_j / A_{c_j} \quad (3)$$

$$D_{3_i} = \sum_j (Re)_j^{0.8} S_j / A_{c_j} \quad (4)$$

$$D_{4_i} = \sum_j (Re)_j^{0.6} S_j / A_{c_j} \quad (5)$$

$$D_{5_i} = \sum_j S_j v_j \quad (6)$$

where A_c is the flow cross-sectional area, S the wetted surface area, v is the coolant velocity and Re is the Reynolds Number.

For Type I and II runs on loops 4, 5, and 6; the data were analyzed using D_2 , D_3 , and D_4 . None of these factors appeared to be better or worse than either of the other two. There is a high correlation between these three factors. The results were similar regardless of the power of Reynold's number. Hence, only D_4 and D_5 were used in subsequent analyses. D_1 was similar to D_5 but nominal (0.5 gpm) rather than actual velocities (flow rates) were used. D_1 is no longer used.

In the mathematical model, equation (2), ΔT , t , and v enter the model raised to powers that were predictable by theory*. As described in Chapter II, the maximum corrosion rate is expected to be proportional to an Arrhenius term in T_{max} . It is important to realize that this form arises from theory for the maximum temperature, T_m , in a loop. The same form has been used here for studying the effects of varying temperature internally within the loop from the various sample holders. Plugging temperature, p , is the basic measurement taken which can be converted to oxygen concentration. This plugging temperature is the temperature at which sodium oxide in solution at a concentration, C , is in equilibrium with solid Na_2O . From the theory of such solution equilibrium** the relation between C and p is given by

$$C = K \exp \left[B / (p + 460) \right] \quad (7)$$

where K and B are constants. Epstein suggests that the relationship between corrosion rate, R , and oxygen concentration, C , is of the form

$$R = K' C^{B'} \quad (8)$$

Substituting the expression for C from (7) into (8) gives

$$R = k \exp \left[\beta / (p + 460) \right] \quad (9)$$

as p appears in the model.

The mechanism of age is not well understood. It is not known how age should be defined or how it should be introduced into the model. However, a simple definition of age has been

*Epstein, L. F., Chemical Engineering Progress Symposium Series No. 20, Vol. 53, p 67. Presented AICHE Meeting, April 1956.

**Hildebrand, J. H. and Scott, R. L. The Solubility of Nonelectrolytes. Reinhold (New York, 1950)

described in equation (1) and is used in the model. It is known that such a term is necessary since large and consistent differences exist between data collected during the first part and last part of a run. The age effect cannot be ignored in predicting long term service, even if it persists for only a few hundred hours after start-up. Long-term corrosion rates will be extrapolated from these 2800-hour runs. Hence, the corrosion rates from these short runs must be corrected for aging effects during the first 2800 hours before extrapolation to long time periods. More and longer runs are needed to investigate this behavior.

Three recurring results in the data necessitated the introduction of a downstream factor into the model. First, the ratio of inlet-to-outlet corrosion rates from H3, both at high velocity, was about two. Secondly, the ratio of H3-to-H3R corrosion rates corrected to the same conditions was about four to one. Finally, the ratio of C3-inlet-to-C3-outlet deposition rates was about two. Hence, a factor in the model describing specimen location is needed. The correct form of this effect is not necessarily represented by any of the downstream factors tried to date. These factors have been adequate for correlating corrosion rates at the sample positions in these loops.

As written, the model expressed in equation (2) is a first approximation from which corrosion rates can be predicted within the range of experimental data used. Extrapolation from 2800-hour runs to three year corrosion rates is extremely imprecise, particularly with the presence of aging. Each factor enters equation (2) independently. For example, we obtain the effect of velocity averaged over temperature. Interaction terms will be introduced later to determine if and how the effect of velocity is dependent on temperature. This will be done for all pairs of factors.

Type I and II runs have been conducted at a constant ΔT and T_m , with only one exception. Hence, the effects of temperature cannot be estimated at this time and appear as part of the constant term b_0 . The data have been analyzed by loops for each run. Thus, the value of b_0 also includes the oxygen level effect. In order to apply a least squares analysis, the logarithm of equation (2) is taken giving

$$\ln |R_i| = b_0 + b_1 \ln t_i + b_2 \ln v_i + b_3 \left(\frac{1}{T_i + 460} \right) + b_4 \ln a_i + b_5 D_i. \quad (10)$$

This results in a simple linear additive model in the unknown coefficients. The errors in the $\ln |R_i|$ have a fairly constant standard deviation with slightly better precision as the length of exposure time is increased. The coefficient of variation (standard deviation expressed as percent) is about 40 percent in the hot leg and about 50 percent in the cold leg. The data from each leg were analyzed separately. In order to use logarithms, only the negative corrosion rates (weight losses) in the hot leg and only the positive corrosion rates (weight gains) in the cold leg were used. Taking the absolute value of corrosion rates is necessary to avoid logarithms of negative numbers. When data from the five runs, listed in Table VI-1, are available, the effects of oxygen, as well as, T_m and ΔT will be separated from the constant term and estimated individually. At the present time, the effects of oxygen can be observed graphically by comparing Type I and Type II plots. However, part of the observed differences may be due to aging of the loops. The results for equation 10 apply for specified T_m , ΔT , and p . The

value of b_0 helps establish the magnitude of the corrosion rate. No physical significance can be attached to the estimate of b_0 since it is a mathematical extrapolation far outside the range of data for which the model applies; corresponding to $t = 1$ hour, $v = 1$ ft/sec, $T = \infty$, $a = 1$ hr, and $D = 0$.

Examination of Table VI-2 shows that as "t" increases "a" also increases. Thus, there is some question that the age effect might be manufactured by the method of analysis. Most of the correlation between time and age comes from the 700 hour data which gives both the lowest value of t and a . Removing the 700 hour data eliminates this correlation. The data were analyzed without the 700 hour data resulting in independent estimates of time and age effects. The results were similar to results employing all data. Hence, the age effects were not generated by the method of analysis and they represent a real effect.

Analysis on weight changes, ΔW , rather than corrosion rates $R = \Delta W/t$ gives identical results with this model (10). For a given set of conditions with only t varying, we may write

$$R = kt^b \quad (11)$$

or

$$\Delta W = kt^{b+1} \quad (12)$$

where k is a constant. The least squares estimate of b is exactly the same whether the analysis is performed on R or ΔW . Taking the logarithm of equation (11)

$$\ln R = \ln \Delta W/t = \ln \Delta W - \ln t = \ln k + b \ln t$$

or

$$\ln \Delta W = \ln k + (b + 1) \ln t$$

which is exactly the same as the logarithm of equation (12). Hence, the two models are identical and will give identical results. Further, let the variance of $\ln \Delta W$ measurements be σ^2 ,

$$V(\ln \Delta W) = \sigma^2.$$

Consider the variance of $\ln R$ measurements,

$$V(\ln R) = V(\ln \Delta W/t) = V(\ln \Delta W - \ln t) = V(\ln \Delta W) + V(\ln t).$$

Since t is essentially measured without error, its variance is zero. That is, $V(\ln t) = 0$.

Hence

$$V(\ln R) = V(\ln \Delta W).$$

That is, a data point is given the same statistical weight whether ΔW or R is analyzed.

Significant carbon changes occur in loops containing $2\frac{1}{4}$ Cr-1Mo steel (loops 2, 3, and 4). The results from these loops differ considerably from the other loops in which no significant carbon transfer occurs. The amount of carbon transfer depends primarily on temperature and occurs almost entirely during the first 700 hours. Since we are interested primarily in long time corrosion rates, it is desirable to correct for these short term carbon effects, making our results more applicable to long term effects. In some cases, data on carbon changes were available for only about half of the samples. In these cases, the average carbon weight changes available for a particular loop-run at each temperature were used. After carbon corrections, results for loops 2, 3, and 5 were similar to the other loops.

In this program, as in many experimental situations, a small proportion of the data vary markedly from the remainder of the data in an unexplainable manner. Individual data points may be far out-of-line due to numerous causes such as: nonhomogeneous material for any important constituent, variation in sample preparation and handling, variation in loop operation, local effects in the loop, laboratory measurements, data recording, data transcribing, and computational errors. It is highly desirable to discard these extremely variable random points that bias the results by tending to pull the fitted curve through themselves. The bias due to these bad points can be particularly large if the point happens to be at the low or high level of any factor. A statistical test based on the reduction in the scatter, standard deviation, due to discarding suspicious values was used. In doing this, a few representative but extreme values are discarded. This penalty is undoubtedly worth absorbing in order to obtain unbiased estimates of the parameters in the model. The test is devised to discard only a small proportion of the good data points. Also, keeping extreme data points which are not in agreement with the remainder of the data will cause over-estimates of the variation of the results.

As mentioned before, only weight losses were analyzed in the hot leg and only weight gains were analyzed in the cold leg. In many cases, the carbon correction made a data point usable. Negative corrosion rates from all loops (carbon corrected in loops 2, 3, and 5) in the hot leg and positive rates (carbon corrected in loops 2, 3, and 5) in the cold leg which failed to agree with the remainder of the data were discarded and are listed below for future review.

Sample Number:	1503	3483	4705
	1506	3504	4912
	1913	3977	5103
	2576	4475	5106
	2703	4501	6505
	2901	4578	6508

Weight changes for approximately 500 samples have been collected to date and less than 4 percent of the values were discarded on the basis of extreme variation from the remainder of the data within the respective run. Of the 18 values discarded, 12 were in the hot leg and 6 in the cold leg. Of the 12 hot leg values discarded, 8 were in H3R. This is a larger proportion than would be expected for random events. This may simply illustrate that there is more variation in H3R results or it may indicate a deficiency in the model (particularly in the downstream and temperature corrections).

When weight gains occurred in the hot leg, they appeared predominantly at those locations where corrosion rates are low. When weight losses occurred in the cold leg, they appeared at location C3 and for pipe samples.

Some data from the hot leg of run 4-2 are presented here to show the individual effects of each factor as determined by a least squares multiple regression analysis. The observed corrosion rates are plotted against the reciprocal of absolute temperature. Considerable variation of the raw data about the line is observed, see Figure VI-1. This line is the least squares prediction for somewhat average conditions, $t = 1400$ hrs, $v = 8$ ft/sec, $a = 1400$ hrs, and $D_4 = 1.0$. When the variation of velocities is accounted for, less variation about the line is obtained (Figure VI-1). For example, the observed corrosion rate of $14.4 \text{ mg/dm}^2\text{-mo}$ for the pipe sample at 0.84 ft-sec is predicted to be $59.5 \text{ mg/dm}^2\text{-mo}$ had the velocity been 8 ft/sec. Hence, correction for the velocity effect brings the pipe sample result into line with the other sample. Figure VI-2 shows a further improvement when the downstream factor is included. For example, the velocity corrected corrosion rate at $8.2 \text{ mg/dm}^2\text{-mo}$ (Figure VI-1) is adjusted to $28.3 \text{ mg/dm}^2\text{-mo}$ (Figure VI-2) when the downstream effect is considered for the H3R specimen. The corrosion rate of $2.2 \text{ mg/dm}^2\text{-mo}$ (Figure VI-2), corrected for velocity and downstream, is still low for this specimen exposed during the first half of the run. Adjusting this rate to an age of 1400 hours gives $3.6 \text{ mg/dm}^2\text{-mo}$ (Figure VI-2) which is closer to the line. For this set of data, the correction for time effects was negligible. Hence, estimating the effects of velocity, downstream, and age reduced the variation considerably in the raw data, with one exception. For the data point with all corrections giving $28.3 \text{ mg/dm}^2\text{-mo}$ (Figure VI-2), the predicted value is 2.5 times higher at $72 \text{ mg/dm}^2\text{-mo}$. This point is one which was discarded from the analysis for having considerably more variation than the remainder of the data. The least squares prediction for the 4-2 hot leg data was

$$|R| = 9.59 \times 10^7 t^{0.02} v^{0.63} e^{-34,260/(T + 460)} a^{0.80} e^{-0.52 \times 10^{-5} D_4} \quad (13)$$

To adjust an observed corrosion rate at v ft/sec to 8 ft/sec, the rate was multiplied by $(8/v)^{0.63}$. For example, if the corrosion rate was $14.4 \text{ mg/dm}^2\text{-mo}$ at 0.84 ft/sec, the predicted corrosion rate at 8 ft/sec is $14.4 \times (8/0.84)^{0.63} = 59.5 \text{ mg/dm}^2\text{-mo}$, as obtained above (Figure VI-1). Similarly, adjustments to common conditions were made for all factors on each data point in Figures VI-1 and VI-2.

To further illustrate the magnitude of the effects of the various factors, three typical samples were selected. The effects of normalizing each factor to a set of average values: $v = 8$ ft/sec, $D_4 = 10^5$, $t = 1400$, and $a = 1400$, are given in Table VI-4.

The corrosion rate on the first line of Table VI-4 gives the observed corrosion rate at the test temperature of the sample. The second line shows the corrosion rate that would be predicted at $v = 8$ ft/sec instead of the indicated velocity. The third line shows the adjustment of the observed data point to a D_4 value of 1.0×10^5 in addition to the velocity adjustment, etc. The last line of Table VI-4 gives the final multiple regression prediction of the corrosion rate at the indicated temperature and the average values of the other factors given above. These results are plotted on Figure VI-3.

FIGURE VI-1 MULTIPLE REGRESSION ANALYSIS ILLUSTRATION

FIGURE VI-2 MULTIPLE REGRESSION ANALYSIS ILLUSTRATION

TABLE VI-4

MULTIPLE REGRESSION ADJUSTMENTS

	Sample No. 1478		Sample No. 4480		Sample No. 6483	
	Test Conditions	Adjusted Corrosion Rate (mg/dm ² -mo)	Test Conditions	Adjusted Corrosion Rate (mg/dm ² -mo)	Test Conditions	Adjusted Corrosion Rate (mg/dm ² -mo)
T	1297	546	1197	38.6	1197	27.4
v	12.1	381	12.1	29.7	13.4	15.5
D ₄ (10 ⁻⁵)	1.158	459	1.597	40.4	0.631	12.9
a	2230	537	745	67.1	1758	12.7
t	1520	524	1490	67.2	2095	16.4

The least squares procedure in itself does not provide the best form of a model for a given set of data. It can be helpful in choosing the best form for the model by comparing the precision of the fit to different types of models for a given set of data. Least squares provides estimates of the b's (unknown parameters) such that the sum of squares of deviations of observed values from predicted values at each point is minimized.

D. Results: Hot Leg

The weight change data which were analyzed are given in Volume 3 of this report. Several different types of models were tried, as discussed in the previous section. The final model used to date which appears to do a fairly good job of prediction is given by equation (10). The use of the Philco Transact Computer made it possible to try a variety of models before selecting this one. The least squares estimates for hot leg coefficients are given in Table VI-5. Each loop-run resulted in weight change data for 15-25 samples. Ninety-five percent confidence limits are attached to the estimates. The data were analyzed with both D₄ and D₅ as downstream factors. The replicate run data were originally analyzed separately for loops 2 and 3. More consistent results were obtained by combining the data from loops 2 and 3 for the replicate runs. The combined results are given in Table VI-5. An analysis of the results in this table shows that the choice of downstream factor between D₄ and D₅ has no influence on velocity or age. The coefficients for time average 0.063 ± 0.047 higher when D₅ is used. All limits are 95 percent confidence limits unless stated otherwise. The effect of temperature is less when D₅ is used. The average difference between temperature coefficients for D₅ and D₄ is 1.76 ± 1.02.

For many estimates in Table VI-5, the ± confidence limits are larger than the value of the exponent. For example, the estimate of b₄ for the age term in loop 1-run 1 was -0.34 ± 0.45. We are 95 percent confident that based on this set of data the interval -0.79 to + 0.11 contains the true value for the age exponent. It might be argued that since the interval indicates the exponent could be positive or negative, it is not estimated very precisely and the age factor should be dropped from the model. This indeed would be the proper action if this were the only

FIGURE VI-3 MULTIPLE REGRESSION ADJUSTMENTS FOR H3 SAMPLES

information available about age. It is quite possible where a series of results are obtained, that no single result may differ significantly from zero. If the results are predominantly greater than zero, say, this consistency may indicate the presence of a factor, when averaged over all of the results available. This is the case here. Whereas only a few of the estimates of the age exponent differ significantly from zero, the average age exponent over all loops in the first run was 0.49 ± 0.41 . Hence, in this run, age was a significant factor and cannot be ignored. For those cases where the limits are larger than the estimate, no significance can be attached to those individual results. But consistency of the estimates over the different loops or between the two runs may indicate the presence of a factor. Hence, time and age have been included in the analyses to determine if any pattern is detectable after several sets of data have been studied. It must be remembered that failure to detect the presence of a factor does not mean the factor is not important. It may only mean that the experimental variation is so large that an effect is not detectable from a few data points from one run of a single loop. One remedy to this situation is to obtain more data by estimating the average effect from several runs or several loops.

The effects of time did not differ among loops. The exponent for time was not significantly different from zero for Type I runs, (0.03 ± 0.38) . For Type II runs, the average time exponent was different from zero, (-0.70 ± 0.38) .

Table VI-2 illustrated how age is defined, except for the replicate runs. For the replicate runs, age was determined from the beginning of the replicate runs rather than from the beginning of each individual run. In Type II runs, the exponent for age was not significantly different from zero, (0.13 ± 0.41) . In Type I runs however, the corrosion rate increased significantly during the run where the average age exponent was 0.49 ± 0.41 . The exponents did not vary significantly among the six loops.

Data from more runs will be required in order to determine if the exponents for time and age are converging to constant values. Extrapolation of the results for Type I and II runs to long term service should not be attempted at this time. A further complication in the interpretation of time and age effects now is the role of oxygen. The change in the exponents for time and age between runs 1 and 2 may in part reflect the change in oxygen concentration from approximately 45 ppm in run 1 to about 12 ppm in run 2. After completion of the 5 fractional replicate runs, it will be possible to separate the effect of oxygen from time and age.

The exponent for velocity did not vary significantly among loops or between runs 1 and 2. The average velocity exponent is 0.76 ± 0.18 . For example, if the velocity is increased by a factor of 10, then the corrosion rate is predicted to increase from $10^{0.58} = 3.8$ to $10^{0.94} = 8.7$ times with 95 percent confidence. Early analyses were run with and without the low velocity pipe samples included. There appeared to be no statistically significant differences in the results. Subsequently, all analyses included the pipe samples.

Again, more runs are needed in order to estimate the temperature coefficients with enough precision to determine the effects of different combinations of materials. The coefficients in Table VI-5 show the effect of changes of temperature within the loop. The effect of changing the maximum temperature and ΔT will be studied in future runs, as previously discussed (para. B).

TABLE VI-5

HOT LEG ESTIMATES OF THE COEFFICIENTS WITH 95 PERCENT CONFIDENCE LIMITS FOR R

$$\text{WHERE } R = -b_0 t^{b_1} v^{b_2} e^{b_3} 10^3 / (T + 460)^{b_4} a^{b_5} D_i$$

Coef. Variable	$\ln b_0$ Constant	b_1 (Time, hrs)	b_2 (Vel., fps)	b_3 (Temp, °F)	b_4 (Age, hrs)	b_5 ($D_4 \cdot 10^{-5}$)	b_5 ($D_5 \cdot 10^{-2}$)
Loop-Run							
1-1	26.19 ± 4.91	+ 0.29 ± 0.53	0.87 ± 0.24	-35.75 ± 5.77	-0.34 ± 0.45	-1.18 ± 0.22	
1-1	20.76 ± 4.53	+ 0.51 ± 0.54	0.88 ± 0.24	-30.90 ± 5.38	-0.29 ± 0.45		-2.24 ± 0.41
1-2	40.46 ± 19.46	-1.44 ± 1.47	0.34 ± 0.55	-40.61 ± 26.92	-0.31 ± 1.64	-0.21 ± 0.56	
1-2	39.28 ± 18.29	-1.34 ± 1.50	0.35 ± 0.54	-40.79 ± 24.30	-0.25 ± 1.62		-0.41 ± 0.86
2-1	23.85 ± 6.28	-0.20 ± 0.58	0.27 ± 0.22	-30.41 ± 9.11	-0.02 ± 0.50	-0.40 ± 0.16	
2-1	22.98 ± 5.05	-0.21 ± 0.50	0.24 ± 0.19	-29.23 ± 7.33	-0.01 ± 0.43		-0.63 ± 0.21
2&3-Rep.	18.37 ± 4.99	-0.66 ± 0.50	0.95 ± 0.44	-22.23 ± 6.35	+ 0.20 ± 0.35	-0.27 ± 0.25	
2&3-Rep.	17.99 ± 4.96	-0.67 ± 0.50	0.97 ± 0.45	-21.80 ± 6.29	+ 0.21 ± 0.36		-0.38 ± 0.36
3-1	20.13 ± 8.89	+ 0.04 ± 0.95	0.97 ± 0.55	-43.12 ± 14.80	+ 1.16 ± 0.89	-0.52 ± 0.28	
3-1	18.07 ± 9.01	+ 0.08 ± 1.04	0.92 ± 0.60	-40.16 ± 15.20	+ 1.11 ± 0.97		-0.79 ± 0.48
4-1	22.23 ± 2.25	-0.42 ± 0.31	0.90 ± 0.09	-34.53 ± 3.67	+ 0.72 ± 0.25	-0.42 ± 0.09	
4-1	20.85 ± 2.47	-0.37 ± 0.36	0.92 ± 0.11	-32.92 ± 4.10	+ 0.69 ± 0.29		-0.72 ± 0.18
4-2	18.38 ± 5.77	+ 0.02 ± 0.58	0.63 ± 0.26	-34.26 ± 7.16	+ 0.80 ± 0.49	-0.52 ± 0.28	
4-2	15.72 ± 4.42	+ 0.14 ± 0.49	0.75 ± 0.21	-31.85 ± 5.05	+ 0.76 ± 0.41		-1.11 ± 0.45
5-1	13.38 ± 6.20	+ 0.45 ± 0.83	0.57 ± 0.25	-24.48 ± 9.26	+ 0.39 ± 0.61	-0.73 ± 0.24	
5-1	11.12 ± 5.16	+ 0.48 ± 0.75	0.58 ± 0.23	-21.92 ± 7.98	+ 0.40 ± 0.55		-1.39 ± 0.40
5-2	21.41 ± 4.77	-0.97 ± 0.55	0.42 ± 0.28	-16.27 ± 6.00	-0.21 ± 0.48	-0.42 ± 0.18	
5-2	19.23 ± 4.72	-0.89 ± 0.60	0.46 ± 0.30	-14.12 ± 6.11	-0.22 ± 0.52		-0.71 ± 0.35
6-1	9.36 ± 2.60	-0.19 ± 0.24	1.12 ± 0.17	-21.10 ± 3.40	+ 1.05 ± 0.22	-0.64 ± 0.09	
6-1	7.29 ± 2.16	-0.10 ± 0.21	1.05 ± 0.14	-19.20 ± 2.79	+ 1.04 ± 0.19		-1.06 ± 0.13
6-2	17.36 ± 15.78	-0.64 ± 0.69	1.11 ± 1.00	-20.13 ± 18.27	+ 0.08 ± 0.50	-0.49 ± 0.48	
6-2	17.88 ± 16.49	-0.66 ± 0.70	0.94 ± 1.15	-20.62 ± 19.00	+ 0.08 ± 0.51		-0.96 ± 0.96

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The temperature effect was greater in the loops with 316 SS in the hot leg (loops 1, 2, 3, and 4) than in the ferritic loops (loops 5 and 6). The average value of b_3 in the 316 SS loops is 32.04 ± 5.58 and 19.73 ± 7.89 in the ferritic loops. The difference is $32.04 - 19.73 = 12.31 \pm 9.76$.

Average values of b_5 for the downstream factors are presented in Table VI-6. Again more runs are needed to determine if the downstream effect is changing.

TABLE VI-6

AVERAGE DOWNSTREAM EXPONENTS, b_5

	Run 1	Run 2	Difference
D_4	-0.65 ± 0.27	-0.36 ± 0.27	-0.29 ± 0.38
D_5	-1.14 ± 0.56	-0.66 ± 0.56	-0.48 ± 0.79

To illustrate the effect of downstream, the value of D_4 at the H3 inlet is approximately 0.7×10^5 and 4.5×10^5 at the H3R outlet. The corrosion rate at the H3 inlet for run 2 is predicted to be $e^{-0.38 \times 0.7} / e^{-0.38 \times 4.5} = 4.2$ times the corrosion rate at the H3R outlet, due to location. For run 1, the ratio of H3 inlet to the H3R outlet corrosion rate is about 12.

The precision of the estimate of the oxygen effect depends upon the precision of the results among runs on the same loop. The precision of estimates of material effects depends upon the precision of the results from different loops. One purpose of the replicate runs was to determine the reproducibility among results from different runs on the same loop. A second purpose was to determine the reproducibility of results from different loops of the same material. Data from the remainder of the five runs, Table VI-1, will be required before definite statements can be made about the precision of comparisons between runs, and between loops. Based on only the replicate run data, average corrosion rates from two runs on the same loop at identical conditions will generally agree within a factor of two. Average corrosion rates from runs on different loops of the same material at identical conditions will generally agree within a factor of three.

Velocity, temperature, oxygen, and downstream have been demonstrated to be important factors. Data from the remaining types of runs are necessary to determine how the effects of these factors may differ for the materials under study. The effects of time and age changing between runs 1 and 2 are particularly puzzling. It is especially important to determine the effects of time and age with good precision, since a long extrapolation from these four-month runs to long times will be necessary to predict performance in a central station type system. Very little can be said about time and age, now, other than that they may be important variables. Hence, additional runs are necessary to estimate the effects of these important factors.

The necessity of a precise estimate of the time and age effects can be illustrated by a mathematical example. Suppose the corrosion rate after 2800 hours is $1000 \text{ mg/dm}^2\text{-mo}$. If there is no time effect, $b_1 = 0$, the corrosion rate remains constant, if $b_1 = -0.5$, at the end of 10 years

the corrosion rate is 180 mg/dm²-mo. Hence, the corrosion rate is highly dependent upon the exponents for time and age.

Graphical presentation of the results of applying multiple regression analysis to the existing corrosion data are provided in Figures VI-4 through VI-7. These results will be discussed in the following sections of this chapter. Figures VI-4 and VI-5 present the hot leg and cold leg corrosion rate data as a function of temperature for the high oxygen (TypeI conditions) runs. Figures VI-6 and VI-7 provide the same type data for the low oxygen (TypeII conditions) runs. Each leg of each loop of each run are analyzed separately to minimize confoundment and the difficulty of analyzing negative corrosion rate data.

Analyzed test data are presented rather than the observed data points in order to normalize some of the test variables so that the effect of others can be described. It should be noted that the results were evaluated at zero downstream; that is at the maximum corrosion rate zone.

Figure VI-8 presents another way of describing the magnitude of the "downstream" effect. In this example all test variables and their respective coefficients are held constant except velocity and the diameter of the pipe. Then, the length of pipe predicted to reduce the corrosion rate by a factor of ten (10) was calculated from the equation for predicting the corrosion rate:

$$R = b_n \exp b_5 D_4 \cdot 10^{-5}$$

where R = the hot leg corrosion rate

b_n = a new constant containing all terms in equation (10) except downstream = $\sim 0.5 \cdot 10^{-5}$

b_5 = exponent for downstream derived from the multiple regression analysis of existing data.

D_4 = the previously defined arbitrary downstream factor $\frac{S}{A_c} Re^{0.6} \cdot 10^{-5}$

$$\text{Thus } R = b_n \exp \frac{b_5 \pi d L}{\frac{\pi d^2}{4}} \left(\frac{\rho v d}{\mu} \right)^{0.6} \cdot 10^{-5}$$

Simplifying and taking the natural logarithm of both sides

$$\ln R = \ln b_n + 4b_5 L d^{-0.4} \left(\frac{\rho v}{\mu} \right)^{0.6} \cdot 10^{-5}$$

Substituting 10 for the change in corrosion rate and letting L_{10} symbolize the pipe length to reduce the corrosion rate by a factor of ten this equation becomes

$$\ln 10 - \ln 1 = 4b_5 L_{10} d^{-0.4} \left(\frac{\rho v}{\mu} \right)^{0.6} \cdot 10^{-5}$$

Substituting the constants (sodium properties evaluated at 1200 F) yields

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FIGURE VI-4 CORROSION RATE VS RECIPROCAL TEMPERATURE FOR HIGH OXYGEN RUNS

277-29A

FIGURE VI-5 CORROSION RATE VS RECIPROCAL TEMPERATURE FOR HIGH OXYGEN RUNS

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FIGURE VI-6 CORROSION RATE VS RECIPROCAL TEMPERATURE FOR LOW OXYGEN RUNS

277-30A

FIGURE VI-7 CORROSION RATE VS RECIPROCAL TEMPERATURE FOR LOW OXYGEN RUNS

FIGURE VI-8 DOWNSTREAM EFFECT AT 1200F

$$2.3026 - 0 = 4 (-0.5 \times 10^{-5}) L_{10} d^{-0.4} \left(\frac{49.6}{1.33 \times 10^{-4}} \right)^{0.6} v^{0.6} \times 10^{-5}$$

or

$$L_{10} = 52.4 d^{0.4} / v^{0.6}$$

where L and d are in feet and v is in feet per second.

if ID is measured in inches this equation becomes

$$L_{10} = 19.4 ID^{0.4} / v^{-0.6}.$$

The graph (Figure VI-8) was plotted from this relationship. It is striking to note that if allowable corrosion rates exist at the start of an isothermal region, the rate will be negligible (less than 1/10 of the initial rate) in a short distance. For instance, the rate will be negligible in a 5-foot length of 1-inch diameter pipe with a 10 ft/sec flow rate. This example emphasizes the effect of downstreamness. Work was derived from preliminary data and analysis and should not be used for design as yet.

Figure VI-9 shows the relative corrosion rate effects in the hot leg for runs Type I and II for the ranges of variables studied. Velocity, temperature, and downstream are important factors. Corrosion rates increase slightly with age and with time. Extrapolation beyond the limits of the factors studied is not advisable at this time.

E. Results: Cold Leg

With the current analytical approach, it is necessary to analyze the data* separately for the hot and cold legs. Certainly, the weight changes in the two legs are not independent. It is most likely that higher weight losses in the hot leg will result in higher weight gains in the cold leg. If corrosion rate decreases with time in the hot leg, it is possible that the deposition rate in the cold leg might also decrease with time. Hence, the model for the cold leg should probably contain some of the same factors in the same form as they appear in the hot leg. For lack of any better approach, the same form of the model was used in the cold leg as was used in the hot leg, equation (10).

The estimates with 95 percent confidence limits are given for each run in Table VI-7. From 15-25 sample weights were obtained in each run. The blank spaces in Table VI-7 indicate that the proper type of data were not available to estimate these exponents. Generally, these factors were approximately constant in a run and therefore not estimable. Downstream effects were primarily estimated from a comparison of C3 inlet versus C3 outlet samples. In the first run locations of samples as to inlet or outlet were not generally recorded. Hence, the downstream effect could not be estimated in run 1, nor in the replicate runs. In these cases an average downstream coefficient from the second run was used.

*Operating condition and weight change data for each sample are reported in Volume 3 of this report.

FIGURE VI-9 AVERAGE RELATIVE CORROSION RATE FACTORS OVER RANGES OF VARIABLES STUDIED IN THE HOT LEG.

TABLE VI-7

COLD LEG ESTIMATES OF THE COEFFICIENTS WITH 95 PERCENT CONFIDENCE LIMITS FOR R

$$\text{WHERE } R = b_0 t^{b_1} b_2^{b_2} 10^3 b_3 / (T + 460)^{b_4} b_5 D_i^{b_5}$$

Coef. Variable	$\ln b_0$ Constant	b_1 (Time, hrs)	b_2 (Vel., fps)	b_3 (Temp, °F)	b_4 (Age, hrs)	b_5 ($D_4 10^{-5}$)	b_5 ($D_5 10^{-2}$)
Loop-Run							
1-1	9.28 ± 3.50	-0.72 ± 0.63	0.64 ± 0.25	-7.52 ± 2.12	+ 0.88 ± 0.46	-1.56	-
1-1	8.42 ± 3.52	-0.74 ± 0.63	0.63 ± 0.25	-7.12 ± 2.13	+ 0.97 ± 0.46	-	-1.71
1-2	14.88 ± 10.39	-0.42 ± 0.89	0.19 ± 1.03	-8.51 ± 4.24	-0.29 ± 0.79	-0.23 ± 1.27	-
1-2	15.12 ± 9.08	-0.42 ± 0.86	0.00 ± 1.04	-7.50 ± 3.98	-0.35 ± 0.77	-	-0.88 ± 1.78
2-1	3.59 ± 2.13	-0.80 ± 0.35	-	+ 6.25 ± 1.26	+ 0.28 ± 0.29	-1.56	-
2-1	2.86 ± 1.96	-0.81 ± 0.32	-	+ 7.21 ± 1.15	+ 0.30 ± 0.26	-	-1.71
2&3 Rep.	1.54 ± 4.37	-0.61 ± 0.59	1.66 ± 0.49	-0.54 ± 2.28	+ 0.55 ± 0.38	-1.56	-
2&3 Rep.	0.49 ± 4.36	-0.63 ± 0.58	1.67 ± 0.49	+ 0.24 ± 2.27	+ 0.58 ± 0.38	-	-1.71
3-1	10.66 ± 6.47	-0.43 ± 0.79	-	-3.38 ± 3.34	-0.16 ± 0.63	-1.56	-
3-1	11.67 ± 4.46	-0.60 ± 0.54	-	-3.50 ± 2.30	-0.08 ± 0.44	-	-1.71
4-1	6.67 ± 8.92	+ 0.13 ± 1.20	1.07 ± 0.35	-6.98 ± 4.31	+ 0.07 ± 0.85	-1.56	-
4-1	6.27 ± 8.89	+ 0.13 ± 1.20	1.05 ± 0.35	-6.58 ± 4.29	+ 0.08 ± 0.85	-	-1.71
4-2	6.83 ± 3.12	+ 0.02 ± 0.49	-	-3.06 ± 1.75	+ 0.03 ± 0.36	-2.63 ± 1.02	-
4-2	6.00 ± 3.13	+ 0.00 ± 0.49	-	-2.45 ± 1.84	+ 0.09 ± 0.35	-	-3.21 ± 1.23
5-1	4.84 ± 3.26	-0.33 ± 0.48	0.54 ± 0.18	-1.78 ± 1.78	+ 0.39 ± 0.42	-1.56	-
5-1	4.41 ± 3.31	-0.31 ± 0.49	0.52 ± 0.18	-1.45 ± 1.81	+ 0.38 ± 0.43	-	-1.71
5-2	2.05 ± 4.59	+ 0.44 ± 0.75	1.36 ± 0.66	+ 6.56 ± 2.34	-1.28 ± 0.59	-1.37 ± 1.36	-
5-2	1.64 ± 4.51	+ 0.44 ± 0.74	1.35 ± 0.66	+ 6.92 ± 2.45	-1.27 ± 0.59	-	-1.63 ± 1.60
6-1	10.31 ± 3.03	-0.50 ± 0.40	0.50 ± 0.14	-5.30 ± 1.41	+ 0.06 ± 0.31	-1.56	-
6-1	9.73 ± 3.06	-0.55 ± 0.40	0.53 ± 0.14	-4.55 ± 1.43	+ 0.11 ± 0.31	-	-1.71
6-2	-0.76 ± 2.47	-0.20 ± 0.24	-	+ 6.28 ± 1.65	+ 0.17 ± 0.27	-1.42 ± 0.94	-
6-2	-1.15 ± 2.45	-0.16 ± 0.24	-	+ 6.67 ± 1.73	+ 0.14 ± 0.27	-	-1.65 ± 1.09

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Time, velocity, and age were not influenced by the choice of the downstream term, D_4 or D_5 . The average temperature coefficient was more positive when D_5 was used, $b_5(D_5) - b_5(D_4) = 0.53 \pm 0.23$.

The average exponent for time was -0.35 ± 0.24 . The exponent for age did not differ significantly from zero during runs Types I and II, 0.11 ± 0.30 . Extrapolation of the results for runs Types I and II to long-term service should not be attempted at this time. More runs are necessary to obtain precise estimates of the effects of time and age for deposition rates.

The estimates of the effects of velocity within the cold leg are particularly poor. All of the samples in the cold leg are in three-slot sample holders or in-pipe samples. Thus, most of the samples analyzed were exposed at about the same velocity. The estimate of the velocity effect depends on the comparison of in-pipe samples with the remainder of the regular corrosion samples, all at relatively low velocities. The average exponent for velocity was, 0.70 ± 0.42 .

The velocity exponent from the replicate runs, 1.66 ± 0.49 , represents a different phenomenon. In this case, the velocity (loop flow rate) was changed from run to run. At higher velocities there was more corrosion in the hot leg. Hence, at higher velocities more corrosion products were available for deposition in the cold leg. The exponent for velocity possibly reflects, in part, this phenomenon.

In the cold leg, run Type II samples located in decreasing temperature locations sometimes resulted in increases of deposition rate and sometimes in decreases. See Figure VI-7. However, the average temperature exponent, b_3 , for runs Types I and II did not differ significantly from zero (-1.27 ± 3.45).

Similar to velocity effects, the estimates of downstream effects in the cold leg are particularly weak due to the lack of data. The estimate of this effect depends primarily on the comparison of C3 inlet versus C3 outlet deposition rates. In Type I run this effect could not be estimated as there was no record of the location of samples exposed in C3. Hence, the downstream exponent was estimated from Type II data only. The effect of age or oxygen on the downstream effect cannot be estimated at this time.

In order to compare the pipe samples with C3 sample data, for velocity effects, a downstream correction for the pipe samples was necessary. Rather than ignore this effect in the run Type I data analyses, average values for the downstream effect from Type II run were used. A preliminary analysis indicated that $b_5(D_4) = 1.56$ and $b_5(D_5) = 1.71$. These estimates of the downstream exponent will improve as more data are collected. Hence, the Type I and replicate run data will be reanalyzed when the final estimates of b_5 are available at the conclusion of the five fractional replicate runs. The best estimates of the downstream coefficients at this time are $b_5(D_4) = -1.41 \pm 1.20$ and $b_5(D_5) = -1.84 \pm 1.20$.

Based on the replicate run data, average deposition rates from two runs on the same loop at identical conditions will generally agree within a factor of 1.7. This large variation between runs must be considered when comparing different runs to estimate oxygen effects. Average

deposition rates from runs on different loops of the same material at identical conditions will generally agree only within a factor of ten. Hence, it is very difficult to compare deposition rates of different materials at this time.

The results from the cold leg samples are much less definitive than those from the hot leg. Cold leg samples show slightly more variation than hot leg samples. Due to the lack of high velocity samples in the cold leg, the velocity effect is not estimated as well. Most of the estimates of the effects in the cold leg border on being non-significant. Data from the five-run test program will be necessary to obtain more precise estimates of the effects under study.

Figure VI-10 shows the average effects in the cold leg for the first two runs over the ranges of variables studied. Velocity and downstream are important factors. Deposition rates increase slightly with age and decrease slightly with time. Extrapolation beyond the limits of the factors studied is not advisable at this time.

F. Discussion and Analysis

The data obtained from the first few runs in the present study, which are reported above, have revealed no basically new phenomena and appear to be consistent with the previously developed models extrapolated into the new regions of operating conditions. As previously mentioned, corrosion in sodium appears to take place either by oxidation of the metal by Na_2O that is present as an impurity in the sodium, resulting in weight gains in the sample; or by a process of simple solution of the solid in the liquid metal. The rates for the two kinds of phenomena have different temperature coefficients however; and the relative importance of the possible processes (which may take place simultaneously) changes as the temperature increases. An interesting example of this effect is the corrosion pattern for austenitic stainless steel. In lower temperature work, this material gains weight, and the increase has been attributed to a reaction with the oxygen in the system to form an adherent oxide film, probably richer in chromium than the basic metal. In the current series of tests, in contrast to this behavior, stainless steel as well as all the other steels investigated, lose weight in the hot region. While the picture is somewhat complicated by the curious role which carbon plays, it appears that the oxidation process which is predominant at lower temperatures is relatively less important than the solution phenomenon as temperature is raised. This concept is entirely in agreement with the thermodynamic and thermochemical data on the oxides derived from stainless steel, in particular those of chromium, but has not, so far as is known, previously been recorded.

For the solution mechanism, where diffusion or solid-liquid transfer is rate-determining, the theory leads to the result, in systems of this sort, that

$$R_{\text{Max}} = - (\alpha/2) (S_{\text{H}}^{\circ} \rho_{\text{H}} - S_{\text{C}}^{\circ} \rho_{\text{C}})$$

where R_{Max} is the maximum corrosion rate (grams per cm^2 per sec. lost), observed at or near the hottest point in the system, where the equilibrium solubility is S_{H}° in g. Fe/g. Na, and the density of the Na is ρ_{H} g./ cm^3 .

Similarly S_{C}° is the equilibrium solubility of the metal and ρ_{C} the density of Na at the coldest point in the loop, and α is the specific solution rate constant (cm/sec). There is some

FIGURE VI-10 AVERAGE RELATIVE DEPOSITION RATE FACTORS OVER RANGES OF VARIABLES STUDIED IN THE COLD LEG.

question about the temperature at which α is to be computed: in the original analysis which led to this theory, ΔT was taken to be small, so that it was a matter of indifference whether α was determined at the maximum T_H or average temperature $\bar{T}$. Horsley⁽¹⁾ recommended that when ΔT was not small, it was better to compute its value at the maximum temperature.

The accuracy of this theory can be evaluated from a comparison with the corrosion rate derived from the statistical analysis above. Introducing the requirements that to obtain the maximum corrosion rate, the "age" $a_i = t_i/2$, and the "downstream function" $D_i = 0$, the multiple regression analysis yields the result

$$R_{\text{Max}} (\text{M.R.}) = -e^{b_0} (1/2)^{b_4} t^{(b_1 + b_4)} v^{b_2} \exp \left[1000 b_3 / (T + 460) \right] \quad (2)$$

where T is in degrees F, and values computed from this equation for three particular sets of conditions are shown in Table VI-8.

R_{Max} (Theory), on the other hand, is given by equation (1) above. In order to use this, the values of the equilibrium solubility S° and the constant α , as functions of temperature, must be known. These have not been determined experimentally for the alloys considered here; but as a very crude first approximation, the results obtained for pure iron may be used. According to these data

$$S^\circ = (-8.7 + 0.0277 T) \times 10^{-6} \quad (3)$$

The constant α was measured as a function of temperature in sodium containing 100 to 300 ppm oxygen, at a mean value of 200 ppm, say; and at an undetermined effective flow velocity v , which was known to be small. The effect of the oxygen constant p (in ppm) on α is by no means certain, but again, to a first approximation, α is linear in p . Combining these concepts, it is found that

$$\log_{10} \alpha (200/p) = -0.007 - 2.338/T \quad (4)$$

In these relations, S° is in g. Fe/g. Na, α is in cm/sec., and T is in degrees Kelvin. For the density of sodium required in equation (1) above the relation

$$\rho = 1.0167 - 2.392 \times 10^{-4} T \quad (5)$$

may be used.

The maximum corrosion rate for three sets of conditions, identical with those examined on Figure VI-3 are given in Table VI-8, and compared with the results of the multiple regression analysis. In this tabulation α , and the corresponding value of R_{Max} , have been computed at both $\bar{T}$ and T_H .

It will be seen that the two theoretical values, in each case, bracket the multiple regression R_{Max} 's, which themselves may be in error by a factor of two or more. It is clear from Table VI-7 that the values of α taken at T_H are somewhat better than those at $\bar{T}$, thus

(1) Horsley, C. W., Journal of Nuclear Engineering, B1 84 (1959)

TABLE VI-8

COMPARISON OF THEORY AND MULTIPLE REGRESSION
ANALYSIS IN COMPUTING R_{Max}

System	Units	316 SS - 316 SS		316 SS - 5 Cr		5 Cr - 5 Cr	
T_H	$^{\circ}\text{F}$	1297		1197		1197	
ΔT	$^{\circ}\text{F}$	550		550		550	
Oxygen content, p	ppm	45		12		12	
Time, t	hours	1520		1490		2095	
Velocity, v	ft./sec.	12.1		12.1		13.4	
$-R_{\text{Max}}$ (M.G.)	mg/dm ² -mo	2640		111		43	
T_H	$^{\circ}\text{K}$	976		920		920	
T_c	$^{\circ}\text{K}$	670		615		615	
$\bar{T}$	$^{\circ}\text{K}$	823		767		767	
S_H°	ppm	18.3		16.8		16.8	
S_C°	ppm	9.9		8.3		8.3	
ρ_H	g/cm ³	0.783		0.796		0.796	
ρ_C	g/cm ³	0.856		0.870		0.870	
$\alpha \times 10^4$	cm/sec	At $\bar{T}$	At T_H	At $\bar{T}$	At T_H	At $\bar{T}$	At T_H
$-R_{\text{Max}}$ (Theory)	mg/cm ² -mo	2.781	7.909	0.4573	1.500	0.4573	1.500
		950	2701	37	121	37	121

confirming Horsley's conclusion for systems with large ΔT 's. The agreement is nothing short of astounding, considering the crudeness of both experiment and the theory used here.

For the important case of diffusion-limited solution

$$\alpha = 0.023 (\underline{D}/D) (vD/\nu_o)^{0.8} (\nu_o/\underline{D})^{0.4}$$

where

D = average loop diameter (cm.)

$\underline{D}$ = diffusion coefficient of solute (cm²/sec) in the liquid sodium

v = linear flow velocity of Na in loop (cm/sec)

ν_o = Kinetic viscosity of the sodium (cm²/sec) i.e., ν/ρ_o where ν_o is the absolute viscosity (poises) and ρ_o the density (gm/cm³) of the Na.

The grouping (vD/ν_0) in this equation is the dimensionless Reynold's number, and (ν_0/D) is also dimensionless. These equations predict that R_{Max} should be independent of the time of operation of the loop, which is not in disagreement with the results given above. The length of the loop does not enter into the evaluation of R_{Max} , and the average loop diameter D only as $(1/D)^{1/5}$; thus the maximum corrosion rate depends only slightly on loop size and geometry, and it appears possible to extrapolate from laboratory observations on relatively small systems to full scale reactor assemblies. The theory predicts that R_{Max} should vary as $v^{0.8}$, which is in excellent agreement with the value of (0.76 ± 0.18) found in the multiple regression analysis of these experiments presented in a previous section. The temperature coefficient of the corrosion process should follow an Arrhenious type equation of the form

$$\ln R_{Max} = A - Q/RT$$

and the constant Q should be approximately equal to the energy of activation for the diffusion process. While $\ln R$ is in fact linear in $1/T$, as is to be expected from this equation, it is not entirely clear that the slope of the resulting line, i.e., Q , is completely consistent with what would be expected theoretically. The lack of experimental data on the diffusion of metals in liquid sodium, however, makes this point somewhat indeterminate at the present time.

Thus, so far as theory goes, it seems to be consistent with the experimental observations. There are a number of areas where the model requires further extension and improvement however:

1. In addition to the maximum corrosion rate R_{Max} , a complete theory that can predict local corrosion rates for a sample located at any point in the loop, is needed. Although the picture may be somewhat clouded by entrance and exit effects in the sample holders (analogous to similar hydrodynamic phenomena observed in heat transfer studies) and by the influence of system geometry and sample shape on the observations, there appears to be evidence for variation of R with distance from the hot zone which is not accounted for in the simple theory outlined above. It has been found, for example, that there is a variation of corrosion rate in an isothermal region (the so-called "downstream effect".)
2. Similarly, the theory outlined above is based on a system with a uniform circular cross section, of diameter D , so that v is a constant. Extension of the equations to more realistic systems in which the hydraulic diameter, and consequently the flow velocity vary around the loop, is needed.
3. The equations presented are based on steady-state operation of the system, and initial transients have been neglected. There is reason to believe that these may be of rather more importance than has been expected ("aging effect") and that periods of nearly 1000 hours of undisturbed operation are required before steady-state conditions are achieved.
4. The theory developed is inadequate to explain weight changes and other phenomena in the cold zone of the system other than to say that the ΔW 's observed should be opposite in sign and equal to or less in magnitude from the weight losses in the hot

zone. Since a substantial part of the difficulties encountered in loop operation is associated with deposition and plugging in the cold zone, it appears evident that it will be necessary to devote much more attention to this end of the system.

5. The above equations are derived for a pure metal in contact with pure sodium. In an alloy, it is entirely conceivable that one or more constituents could be selectively extracted from the solid phase, and transported at different rates through the solution. There is good evidence that this happens with carbon, and some indirect indications that it may occur with other alloying constituents as well. It is possible that an adequate theory will require separate consideration of each component of the alloy. Similarly there is older data suggesting that α is directly proportional to the concentration of sodium oxide present in the Na. There is insufficient information, either from the completed tests of this program or other studies, to determine the validity of this linear relation, and the effect of system variables on the parameters.

The behavior of carbon, mentioned above, unfortunately acts to somewhat obscure the effects observed. Carbon is known to have a finite, although small, solubility in Na. In low-temperature work, iron and low-alloy steels tend to lose C; and prolonged exposure in hot, pure, sodium is an extremely effective way to decarburize these materials almost completely. Stainless steel, on the other hand, gains weight due to carbon pick-up, and this has been explained by the stability of the carbides of Cr and other alloying constituents in the material. When there are weight gains in a sample, therefore, there is always the question of whether these result from reaction with oxygen or carbon, or both. In some of the current experiments, there have been some indications that a substantial part of the weight gain observed in the lower temperature region in the bimetallic 316 SS - 2 $\frac{1}{4}$ Cr loops is due to carbon; and the form of the curves of carbon content of the metals versus time seem to be entirely consistent with a solid phase diffusion controlled mechanism. The question of the interactions of carbon and oxygen with steels, and each other, is however still unclear, and must await further experiment for clarification.

Another interesting observation has been made with stainless steel. This material in contact with some liquid metals appears to dissolve non-uniformly; and the various constituents of the alloy are selectively extracted by the solvent. The nickel, for example, is dissolved out preferentially in contact with Hg or liquid Li. Austenitic stainless steel is a metastable phase normally; and its transition to the equilibrium ferrite form is prevented only by a careful balance of the Fe-Ni-Cr ratio resulting in a kinetic situation unfavorable to transformation. When this balance is upset by differential solution (or any similar process), the solid composition may be altered enough so that the γ to α transition can take place. This effect has been seen often with other liquids, and has in fact been reported previously with sodium. Apparently, at the higher temperatures of the present experiments the phenomenon takes place to a greater extent than at lower temperatures; and in this work to date, a number of examples of the austenitic to ferritic transformation have been seen. The role, if any, which carbon and oxygen play in this process is not yet definite, however.

In summary, it will thus be observed that the present experimental program results lend some assurance to the belief that the basic phenomena in these systems have been correctly understood

and interpreted. The difficulties of the experiments are such that corrosion rates are seldom good to better than a factor of two; and prediction of R_{Max} using values of α and S° obtained some years ago for pure iron agree with experiment just about as well. The nature of the experimental facilities and equipment available, and the results obtained to date, support the expectation that by operating the loops under conditions somewhat different and more severe than have previously been extensively explored with sodium systems, and with careful control of process variables possible with those components, a greater understanding of the phenomena of corrosion and mass transfer effects will be forthcoming in the future. To complement such observations, the extensions to theory along the lines described above, will be most important and useful.

CONCLUSIONS AND RECOMMENDATIONS

1. It is apparent that from the available preliminary data that as expected, the corrosion and metallurgical effects on candidate steels are more severe at elevated temperatures than at lower temperatures. However, no large departures from existing theory and models have been observed. Various geometric conditions have been explored since there is indication of a velocity and "downstream" effect on the corrosion rates. Deposition and flow fouling or plugging of small passages has been observed.
2. Except for changes in the carbon content of the materials, no critical deterioration in material properties has been observed. However, relatively little emphasis on physical properties has been made under this program, so substantial differences in properties changes may yet be found. Thus, in respect to uniform corrosion effects only, any of the materials or material combinations tested could be utilized in a large system within the temperature, oxygen, and flow ranges tested with appropriate allowance for the change in carbon content.
3. Additional experimental work will be necessary to apply the information obtained in this program, (also from other related work) to large scale systems. The scale-up of geometry and velocity effects requires further testing.
4. With proper care in design and assembly, small scale sodium systems can be operated and maintained with flanged joints operating at temperatures above 1000 F in air. However, even a small sodium leak will cause serious damage to the steel structure, as reported in the Liquid Metal Handbook (NAVEXOS P-733 Rev. , June, 1952, p. 151). The corrosion of steel at high temperature by sodium exposed to air is very rapid.
5. The activated-foil tracer technique has not provided unequivocal data. Its routine use should be discontinued until improved counting and discrimination techniques are developed.

Based on the above observations and the reported information, it is recommended that:

1. The "five run fractional replicate test program" for the primary variables appears likely to attain the planned objectives and should be completed.
2. Additional tests be performed with test conditions for oxygen at an extra low (3rd level) and also for a change in flow rate with all other variables the same as one of the runs in the test plan.
3. A vigorous attempt to be made to determine the composition of the depositing material and means of limiting or removing this deposition.
4. A much greater emphasis is needed on tests designed to obtain quantitative data on the deterioration of the physical properties of the steel alloys, subjected to exposure in realistic "Reactor System Sodium" environment.
5. Additional test loops should be designed and operated to make possible the extension or scale-up of geometrical effects, which so far as we now know may be peculiar to the existing equipment.
6. Long time exposures comparable at least to the life of removable reactor components (20,000-30,000 hours) should be scheduled and monitored in detail for improved confidence in extrapolation of the existing data.

7. Corrosion study of well-controlled and re-insertable samples should be made in large scale reactor-size or test loop systems.
8. Experimental data should be obtained on operation in various controlled atmospheres to establish the most practical conditions for minimizing the catastrophic corrosion of steel in the vicinity of a sodium leak at high temperatures.

APPENDICES

Three appendices follow. They contain the detailed information and data listed below.

Appendix A - Test Loop Geometry, Temperatures and Flow Characteristics

Appendix B - Operating Data and Analysis of Sodium Samples

Appendix C - Metallurgical Examination of Two Failed Components

APPENDIX A

TEST LOOP GEOMETRY, TEMPERATURE AND FLOW CHARACTERISTICS

August 1961

Prepared By D. R. Rice

This appendix provides a ready reference for representative geometry data, temperature distribution, and flow characteristics throughout the six identical test loops. The graphs and tables are based upon a loop operating over the temperature range of 700 F to 1200 F. In Figures A-1 through A-6, sodium temperature, velocity, and wetted area are plotted as a function of centerline distance measured from the pump inlet in the direction of flow. Graphs of sodium temperature, velocity, and Reynolds number versus wetted area are shown in Figures A-7 and A-8. Table A1 lists temperatures, velocities, and selected geometry factors for individual components and pipe section in sequential order of flow around the loop, beginning with the pump. The range of sodium velocities and the percentage of total loop volume at these velocities are shown in Table A2.

The wetted area at a specified location is defined as the total internal surface area in contact with the sodium between the pump inlet and that point (measured in the direction of flow). Indicated velocities are based on a volume flow rate of 0.50 gallons per minute of sodium at 700 F.

In calculating the centerline distances, areas, Reynolds numbers, and velocities it was assumed that the sample holders were loaded as shown in the standard loading pattern (see Figure III-4) and that the surge tank was 33 percent (or 4 inches) filled with sodium. Loop assembly drawing No. 197E782 and referenced component drawings (not attached, but most of which are included in GEAP-3725) were used to calculate the geometric parameters.

The hydraulic diameter ($D_H = 4 \times \text{cross sectional area} / \text{wetted perimeter}$) was used as the characteristic length throughout the loop in the calculation of Reynolds numbers. In the surge tank where the flow stream is not clearly defined, it was assumed that the velocity and Reynolds number would be relatively small. Estimated values are shown on the graphs (Figures A-1 through A-8).

NOMENCLATURE

T = Temperature, °F

L = Length, inches

S = Wetted surface area, in.²V = Sodium volume, in.³* All values based upon flow rate = 0.50 gpmA = cross, sectional area, in.²

v = velocity, ft/sec

t = residence time, sec.

Sv* = possible downstream correlating factor

FIGURE A-1 TEMPERATURE vs CENTERLINE DISTANCE

FIGURE A-2 WETTED AREA vs CENTERLINE DISTANCE

FIGURE A-3 VELOCITY vs CENTERLINE DISTANCE

FIGURE A-4 SODIUM VELOCITY IN SAMPLE HOLDERS H1, H2 & H3

FIGURE A-5 SODIUM VELOCITY IN SAMPLE HOLDER H3R

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FIGURE A-6 SODIUM VELOCITY IN SAMPLE HOLDERS C1, C2 & C3

FIGURE A-8 VELOCITY & REYNOLDS NUMBER VS WETTED AREA

TABLE A-1

TEST LOOP GEOMETRY, TEMPERATURE AND FLOW TABULATION

Based upon standard sample loading pattern and
temperature distribution

COMPONENT	T	L	S	V	S/V	A	v*	t*	Sv*
1. Pump	675	94.3	82.4	5.70	14.5	0.0604	2.64	2.98	2.18
2. Pump-H1 Htr Line	675	4.34	8.5	1.32	6.44	0.304	0.521	0.69	4.42
3. H1 Heater	850	13.0	370.0	121.0	3.05	9.35	0.018	60.2	6.68
4. Htr-H1 Inlet	1000	3.84	7.5	11.7	0.64	0.304	0.550	0.58	4.11
5. H1 Sample Holder									
a. 3/4 in. D. Hole	1000	2.80	6.00	1.24	4.84	0.442	0.380	0.61	2.28
b. 9/16 in. D. Hole	1000	1.80	3.10	0.45	6.88	0.250	0.670	0.21	2.08
c. Inlet slot	1000	2.94	9.20	0.092	100.0	0.0311	5.38	0.045	49.6
d. Crossover	1000	1.26	10.4	0.583	17.8	0.357	0.470	0.22	4.88
e. Outlet slot	1000	2.94	3.06	0.030	102.0	0.0103	16.3	0.015	49.8
f. Outlet	1000	1.80	1.75	0.374	4.67	0.208	0.806	0.19	1.49
6. H1 Outlet-Htr In.	1000	10.4	21.2	3.18	6.68	0.304	0.550	1.58	11.6
7. H2 Heater	1050	7.00	207.0	65.5	3.31	9.35	0.018	32.4	3.73
8. Htr-H2 Inlet	1100	3.84	7.50	1.17	6.42	0.304	0.560	0.57	4.20
9. H2 Sample Holder									
a. 3/4 in. D. Hole	1100	2.80	6.00	1.24	4.84	0.442	0.387	0.61	2.32
b. 9/16 in. D. Hole	1100	1.80	3.10	0.45	6.88	0.250	0.683	0.21	2.12
c. Inlet slot	1100	2.94	9.20	0.092	100.0	0.0311	5.48	0.045	50.3
d. Crossover	1100	1.26	10.4	0.583	17.8	0.357	0.478	0.21	4.96
e. Outlet slot	1100	2.94	3.06	0.030	102.0	0.0103	16.5	0.015	50.5
f. Outlet	1100	1.80	1.75	0.374	6.68	0.208	0.821	0.18	1.44
10. H2 Out-Htr. In.	1100	10.4	21.2	3.18	6.68	0.304	0.560	1.55	11.8
11. H3 Heater	1150	7.00	207.0	65.5	3.31	9.35	0.018	32.4	3.73
12. Htr-H3 Inlet	1200	3.84	7.50	1.17	6.40	0.304	0.567	0.56	4.25
13. H3 Sample Holder									
a. 3/4 in. D. Hole	1200	2.80	6.00	1.24	4.84	0.442	0.392	0.69	2.35
b. 9/16 in. D. Hole	1200	1.80	3.10	0.450	6.88	0.250	0.694	0.21	2.14
c. Inlet slot	1200	2.94	3.06	0.030	102.0	0.0103	16.8	0.015	51.4
d. Crossover	1200	1.26	10.4	0.583	17.8	0.357	0.487	0.21	5.06
e. Outlet slot	1200	2.94	3.06	0.030	102.0	0.0103	16.8	0.015	51.5
f. Outlet	1200	1.80	1.75	0.374	4.68	0.208	0.821	0.18	1.44
14. H3 Out-H3R In	1200	24.0	54.2	7.30	7.42	0.304	0.567	3.52	30.8
15. H3R Sample Holder									
a. 9/16 in. D. Hole	1200	3.16	5.63	0.790	7.13	0.250	0.694	0.38	3.91
b. Inlet slot	1200	2.94	9.20	0.092	100.0	0.0311	5.57	0.045	51.2
c. Crossover	1200	1.26	10.4	0.583	17.8	0.357	0.487	0.21	5.08
d. Outlet slot	1200	2.94	2.72	0.032	85.0	0.0105	16.5	0.015	44.8
e. Outlet	1200	1.80	1.75	0.374	6.68	0.208	0.821	0.18	1.44
16. H3R Out-Cooler In	1200	53.9	106.0	16.4	7.09	0.304	0.567	7.91	60.1
17. C1 Cooler	1150	20.0	66.0	17.3	3.81	0.864	0.199	8.43	13.1
18. Cooler-C1 Inlet	1100	2.0	6.6	1.73	3.81	0.864	0.198	0.85	1.30

TABLE A-1 (Continued)

COMPONENT	T	L	S	V	S/V	A	v*	t*	Sv*
19. C1 Sample Holder									
a. 3/4 in. D. Hole	1100	2.65	6.24	1.17	6.17	0.442	0.386	0.57	2.79
b. 9/16 in. D. Hole	1100	1.75	3.10	0.438	7.08	0.250	0.683	0.21	2.12
c. 5/8 in. D. Hole	1100	2.94	5.80	0.906	6.40	0.308	0.553	0.44	3.21
d. Crossover	1100	1.26	10.4	0.583	17.8	0.357	0.447	0.22	4.96
e. Outlet slot	1100	2.94	9.20	0.092	100.0	0.0311	5.41	0.045	50.3
f. Outlet	1100	1.75	3.10	0.438	7.08	0.250	0.683	0.21	2.12
20. C1 Out-Cooler In	1100	4.60	13.7	3.99	3.45	0.864	0.198	1.94	2.72
21. C2 Cooler	1050	20.0	66.0	17.3	3.81	0.864	0.197	8.48	13.0
22. Cooler-C2 In	1000	2.0	6.6	1.73	3.81	0.864	0.195	0.84	1.29
23. C2 Sample Holder									
a. 3/4 in. D. Hole	1000	2.65	6.24	1.17	6.17	0.442	0.380	0.58	2.75
b. 9/16 in. D. Hole	1000	1.75	3.10	0.438	7.08	0.250	0.670	0.22	2.07
c. 5/8 in. D. Hole	1000	2.94	5.80	0.906	6.40	0.308	0.545	0.44	3.16
d. Crossover	1000	1.26	10.4	0.583	17.8	0.357	0.470	0.22	4.88
e. Outlet slot	1000	2.94	9.20	0.092	100.0	0.0311	5.40	0.045	49.6
f. Outlet	1000	1.75	3.10	0.438	7.03	0.250	0.670	0.22	2.08
24. C2 Out-Cooler In	1000	4.60	13.7	3.99	3.45	0.864	0.195	1.97	2.68
25. C3 Cooler	850	78.6	26.1	68.2	3.81	0.864	0.191	34.3	51.0
26. Cooler C3 In	700	2.00	6.60	1.73	3.81	0.864	0.186	0.90	1.23
27. C3 Sample Holder									
a. 3/4 in. D. Hole	700	2.65	7.24	1.17	6.17	0.442	0.362	0.61	2.52
b. 9/16 in. D. Hole	700	1.75	3.10	0.438	7.08	0.250	0.640	0.23	1.98
c. Inlet slot	700	2.94	9.20	0.092	100.0	0.0311	5.15	0.047	47.4
d. Crossover	700	1.26	10.4	0.583	17.8	0.357	0.447	0.24	4.55
e. Outlet slot	700	2.94	9.20	0.092	100.0	0.0311	5.15	0.047	47.4
f. Outlet	700	1.75	3.10	0.438	7.08	0.250	0.690	0.23	1.98
28. C3 Outlet-FM	700	2.60	8.56	2.25	3.81	0.864	0.186	1.16	1.59
29. PM Pipe	700	17.0	19.4	1.77	11.0	0.104	1.54	0.92	30.0
30. Surge Tank**	100	4.0	151.0	199.0	0.76	49.8	0.003	103.0	0.45
31. Surge Tk-EM Pump	675	52.0	101.0	15.9	6.37	0.304	0.522	8.30	52.8
32. Bypass Line									
a. Inlet	675	14.0	27.3	4.26	6.40	0.304	0.521	2.23	14.2
b. PM Pipe	675	18.0	20.5	1.87	11.0	0.104	1.54	0.98	31.6
c. Aux. Cooler	675	17.0	55.8	14.7	3.80	0.864	0.184	7.70	10.2
d. 5/8 in. D. Line	675	41.0	61.8	7.48	8.31	0.181	0.885	3.86	54.6
e. PI Cooler	675	17.0	55.8	14.7	3.80	0.864	0.184	7.70	10.2
f. Return Line	675	16.0	31.2	4.86	6.28	0.304	0.521	2.56	16.3

**Based upon 4 in. sodium level in ST.

TABLE A-2

SODIUM VOLUME & VELOCITY DISTRIBUTION

(Based upon Table A1 Data)

<u>Location</u>	<u>Velocity ft/min.</u>	<u>Volume</u>	
		<u>in. ³</u>	<u>% of Total</u>
Surge Tank	0.2	194.4	25.6
Main Heaters	1.0	368.2	48.5
Main Coolers	11.0	120.0	15.8
1/2 in. & 1 in. Piping	11-50	67.3	8.9
Pump-FM Lines	>60	7.5	1.1
Sample Passages	>300	0.79	0.10
Total Loop		758.2	100.0

APPENDIX BOPERATING DATA AND CHEMICAL ANALYSIS OF SODIUM SAMPLES

March 1962

Prepared by G. Billuris and W. W. Sabol

This appendix contains a tabular listing of the impurities in the sodium samples and a graphical representation of the loop flow and plugging temperature (oxygen content) as a function of test time. All data and samples were taken prior to December 31, 1961. The flow and plugging temperature data were obtained from the operating log data sheets. The chemical analyses were performed and reported by the Analytical Chemistry Unit, Vallecitos Atomic Laboratory.

Appendix B contains:

Chemical Analysis of Sodium Samples - Table B1.

Sodium Flow versus Test Time	Loop No. 1	Figure B-1
Sodium Flow versus Test Time	Loop No. 2	Figure B-2
Sodium Flow versus Test Time	Loop No. 3	Figure B-3
Sodium Flow versus Test Time	Loop No. 3 (Runs 3-4, 3-5, and 3-6)	Figure B-4
Sodium Flow versus Test Time	Loop No. 4	Figure B-5
Sodium Flow versus Test Time	Loop No. 5	Figure B-6
Sodium Flow versus Test Time	Loop No. 6	Figure B-7
Plugging Temperature versus Time Test	Loop No. 1	Figure B-8
Plugging Temperature versus Time Test	Loop No. 2	Figure B-9
Plugging Temperature versus Time Test	Loop No. 3	Figure B-10
Plugging Temperature versus Time Test	Loop No. 4	Figure B-11
Plugging Temperature versus Time Test	Loop No. 5	Figure B-12
Plugging Temperature versus Time Test	Loop No. 6	Figure B-13

TABLE B-1

CHEMICAL ANALYSIS OF SODIUM SAMPLES

Sample No.	Run	Test Hrs.	Concentration, ppm								
			Ca	Cr	Fe	K	Li	Mn	Mo	Ni	C
S6	1-1	100	67	<2	16.5	-	-	-	-	<16	-
S14		700	50	0.8	177	-	-	-	-	1.1	-
S22		780	<22	0.3	2.0	-	-	-	-	2.5	-
1-1-7		1470	<8	1.1	3.5	229	<14	<1.7	0.3	2.8	-
1-1-12		2989	<9	1.4	1.5	203	<13	<1.8	0.2	1.4	-
1-2-1	1-2	0	<10	0.8	3.7	129	<9	<1.6	0.2	2.1	4
1-2-2		692	<10	0.8	3.5	280	<11	<1.5	0.4	1.9	15
1-2-3		1400	<11	1.0	2.9	-	-	-	-	1.5	9
1-2-5		2165	<17	0.3	2.0	193	-	-	-	1.8	9
S7	2-1	0	67	2.7	15.0	-	-	-	-	<10	-
S18		650	16	0.2	12.8	-	-	-	-	2.5	-
S23		1440	<13	0.5	3.6	-	-	0.4	<0.5	2.5	-
2-1-5		2780	<14	5.4	3.2	-	-	3.3	<0.6	3.1	-
2-1-6		2780	<10	4.9	3.2	-	-	1.3	<0.3	1.5	-
2-2-5	2-2	70	<19	0.9	3.3	261	<14	3.3	1.1	1.4	68
2-2-6		680	<14	0.3	1.7	151	<10	<1.7	<0.2	1.2	8
2-3-1	2-3	22	<14	0.3	1.0	270	<10	<1.5	<0.2	1.8	22
2-3-2		693	26	0.2	7.9	246	<11	<1.6	0.6	1.4	5
2-4-1	2-4	1430	<7	0.3	1.2	228	-	-	-	0.4	11
2-4-2		2799		0.8	5.7	-	-	-	-	0.7	-
S11	3-1	700	90	0.1	1.0	-	-	-	-	3.4	-
S16		1410	40	1.0	329	291	<9	-	-	1.1	-
S21		2730	<12	0.2	0.6	-	-	<1.3	0.4	1.1	-
3-2-3	3-2	0	<15	2.5	0.8	187	<9	<2.4	1.5	1.7	30
3-2-4		0	<14	1.3	0.9	173	<8	<2.2	0.4	0.4	-
3-2-6		660	<10	0.8	0.7	175	<11	<2.4	0.4	0.5	6
3-2-7		660	<10	1.2	5.0	165	<5	<2.0	<0.3	1.2	-
3-2-8	3-3	0	<10	0.7	0.8	217	<8	<1.6	<0.2	0.8	-
3-3-1		150	<9	0.8	2.6	210	<13	<1.9	0.3	1.3	58
3-3-2		650	<16	0.6	9.8	245	<11	<1.7	0.2	2.6	17
3-4-1		694	<13	0.1	1.0	366	<12	<1.4	<0.2	1.3	20
3-5-1	3-5	696	17	0.5	1.1	295	<10	<1.7	0.3	1.1	5
S8	4-1	120	35	1.6	15.6	-	-	-	-	11	-
S12		700	90	0.8	4.9	-	-	-	-	2.4	-
S17		1410	80	1.8	3.2	-	-	-	-	0.9	-
S25		2802	16	0.3	1.4	-	-	1.1	<0.6	1.6	-

TABLE B-1 (Continued)

<u>Sample No.</u>	<u>Run</u>	<u>Test Hrs.</u>	<u>Concentration, ppm</u>								
			<u>Ca</u>	<u>Cr</u>	<u>Fe</u>	<u>K</u>	<u>Li</u>	<u>Mn</u>	<u>Mo</u>	<u>Ni</u>	<u>C</u>
4-2-3	4-2	79	<15	0.1	3.5	334	<8	<1.6	0.3	1.6	17
4-2-4		700	<10	<0.1	0.8	331	<10	<1.1	0.2	1.5	23
4-2-6		1922	15	1.0	8.5	99	<10	<1.6	0.4	1.8	10
4-2-7		2759	10	0.7	6.1	270	<10	<1.7	<0.2	1.3	15
4-3-1	4-3	673	<10	1.8	1.3	359	<32	<1	<0.2	1.9	<5
S13	5-1	630	80	1.2	5.2	-	-	-	-	-	-
S20		1250	1972	0.7	156	-	-	-	-	1.1	-
5-1-7		2600	<10	0.7	31.4	-	-	4.9	0.9	4.2	-
5-1-10		2700	<10	0.2	1.9	314	<9	0.8	<0.2	1.4	19
5-2-3	5-2	138	<27	0.9	4.2	167	<12	<1.5	<0.2	2.3	29
5-2-4		664	23	0.4	8.5	250	<10	<1.7	<0.2	2.9	4
5-2-5		2766	<17	0.4	2.8	95	-	-	-	0.8	5
S15	6-1	700	50	0.8	3.8	-	-	-	-	0.8	-
S19		1400	19	0.6	0.2	-	-	-	-	1.8	-
S28		2680	13	0.6	10.8	-	-	5.7	<0.5	5.1	-
S29		2730	16	0.6	3.6	-	-	0.4	0.6	4.1	-
6-2-5	6-2	70	<10	0.8	2.6	261	<9	<0.3	<2.0	2.7	23
6-2-6		680	<11	<0.1	1.7	331	<11	<0.2	<1.2	1.2	17
6-2-7		1383	19	0.6	3.5	185	<10	<0.2	<1.7	2.2	14
6-2-8		2788	<15	0.2	2.2	319	-	-	-	1.6	21

FIGURE B-1 FLOW vs TEST TIME - LOOP #1

FIGURE B-2 FLOW vs TEST TIME - LOOP #2

FIGURE B-3 FLOW vs TEST TIME - LOOP #3

FIGURE B-4 FLOW vs TEST TIME - LOOP #3

FIGURE B-5 FLOW vs TEST TIME - LOOP #4

FIGURE B-6 FLOW vs TEST TIME - LOOP #5

FIGURE B-7 FLOW vs TEST TIME - LOOP #6

FIGURE B-8 PLUGGING TEMPERATURE vs TEST TIME - LOOP #1

FIGURE B-9 PLUGGING TEMPERATURE vs TEST TIME - LOOP #2

FIGURE B-10 PLUGGING TEMPERATURE vs TEST TIME - LOOP #3

FIGURE B-11 PLUGGING TEMPERATURE vs TEST TIME - LOOP #4

FIGURE B-12 PLUGGING TEMPERATURE vs TEST TIME - LOOP #5

FIGURE B-13 PLUGGING TEMPERATURE vs TEST TIME - LOOP #6

APPENDIX CMETALLURGICAL EXAMINATION OF TWO FAILED COMPONENTS

October 1961

Prepared by F. J. Hetzler

This appendix discusses the results of the metallurgical examination of the pump cell and heater tube failures which were referenced in Section IV. These investigations were made to determine the cause of the failures and thereby facilitate remedial action to minimize the risk of recurrent sodium leaks.

C1. FOUR PASS PUMP CELL FAILUREA. Discussion

A material failure and resulting sodium leak occurred in the $2\frac{1}{4}$ Cr-1Mo, electromagnetic pump, four pass cell during loop No. 2 start-up operation. Figure C-1 shows the configuration of the original cell with the electrodes attached. The cell was fabricated from 3/8-inch o.d. tubing with 0.049-inch wall thickness. The nickel electrodes and the $2\frac{1}{4}$ Cr-1Mo tubing were furnace brazed with 82.5 percent Au-17.5 percent Ni braze.

The sodium fire which accompanied the failure, partially obscured determination of the cause of the leak. Examination of the failure produced several significant factors however, which can be seen in Figures C-2 through C-5. Figure C-2 shows a top view and a side view of the faulty tube section with an attached electrode. Figure C-3 illustrates a cross section of the leak. The material adjacent to the leak is fully martensitic. Corrosive action created by the escaping and burning sodium has obliterated the structure adjoining the original leak. A section adjacent to the leak, shown in Figure C-4, indicates alloying at the i.d. of the tube and a martensitic microstructure.

The cross section of tubes in the vicinity of the leak can be observed from Figure C-5. Grain growth is evident in tubes adjacent to the leak, suggesting that very high temperatures had been reached in that section.

B. Conclusions

The size of the leak was probably increased by excessive heat in the area of the failure. High electrical resistance from a poor braze joint or a discontinuity in sodium flow (gas) could have caused excessive local heating. The leak appeared as that of a melting action, which could have been the actual cause, however, a fracture and accompanying fire could have achieved the same result. In either case, temperatures in excess of 2100 F were probably reached in the local area.

FIGURE C-1 FOUR PASS CELL FOR EM PUMP

FIGURE C-2 TOP & SIDE VIEW OF PUMP CELL TUBE FAILURE

250X

PICRAL

FIGURE C-3 CROSS SECTION OF LEAK

250X

PICRAL

FIGURE C-4 SECTION ADJACENT TO LEAK

277-27

277-26

FIGURE C-5 CROSS SECTION OF FAILED TUBE AND ADJACENT TUBE

C. Non-Destructive Testing of Pump Cells

X-ray inspection of braze areas and the adjoining tubes were initiated to help insure satisfactory pump cell material and joints. The method employs a 160 KVP x-ray unit with a focal spot of 1.5-mm. Subject distance was 24 inches; exposure time 15 minutes. Two x-ray films were provided for each shot, which were packed in the same cassette. Film type was Gavaert D-2 Structurix Single Emulsion and Galvaert D-2 Structurix Double Emulsion. The fine definition achieved with the films allowed magnification of $10\times$ to $20\times$, so that fine details could be observed.

C2. 5Cr HEATER TUBE FAILURE

A. Discussion

A heater tube failed in loop No.6, the 5 Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti monometallic loop, after 5530 hours of operation. The tube was one of a 7-element bundle which contained cartridge heaters inside and contacted sodium on the outer tube surface. Inlet sodium temperatures had been 1000 F while the outlet temperatures were 1100 F. Approximately eight cycles, from room temperature to full operating temperature had been experienced by the tube, prior to failure.

Figure C-6 illustrates the design of the heater and positioning of the heater tubes. Nominal diameters are 0.625-inch o.d. and 0.500-inch i.d. for the tube and 0.495-inch o.d. for the heater element. Investigation of the cause of failure necessitated removal of the entire heater and sectioning of the tube wall. Tubes were removed from the tube sheet by machining. The actual failure is illustrated in Figure C-7.

Failure occurred approximately 1/2-inch above the area where the tube entered the tube sheet (shiny portion on lower end of tube represents the tube to tube sheet location). The failure appeared as a longitudinal rupture. Sectioning through the failure produced the results shown in Figure C-8.

The failure illustrated in Figure C-7 is positioned at the extreme left of the photograph in Figure C-8. Three other partial ruptures appear counterclockwise to the main rupture. Just clockwise of the main rupture, a fracture appears in the Inconel. An annulus up to 20 mils wide is evident between the 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti sheath and the Inconel, where nominal less than 2-mils clearance exists. Fabrication practice calls for a shrink fit of the Inconel covered heater into the 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti tube. Sodium had saturated the MgO insulation inside the Inconel tube.

A partial rupture in the 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti tube is illustrated in Figure C-9. This rupture appears in the outer shell approximately at the bottom of the illustration in Figure C-8. Vicker's Diamond Pyramid Hardness readings indicate a marked increase in hardness in the area immediately adjacent to the rupture. The increased hardness at the inside surface of the 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti tube is illustrated in Figure C-10. An intergranular attack also appeared, similar to the rupture pattern.

An intergranular crack was present in the Inconel which appears at approximately 10 o'clock in Figure C-8. The photomicrograph in Figure C-11 shows the intergranular crack with several branches and indicates evidence of alloying at the outside diameter of the tube in the annulus region.

GEAP-3726

FIGURE C-6 HEATER

277-33

FIGURE C-7 HEATER TUBE FAILURE

B. Conclusions

The actual cause of failure has not been determined; however, a hypothesized cause can be constructed from observations.

1. The annulus between the tubes probably resulted from gas pressure which could have been created by a foreign material collected at the lower end of the annulus. The foreign material could have been introduced during the shop operations and contained by the close fitting heater-tube configuration.
2. Failure at the $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$ tube was probably caused by stress corrosion.
3. Failure of the Inconel tube appears to have been caused by intergranular attack similar to that of sulfur attack. This could have occurred either before or after the $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$ failure.
4. An exothermic reaction took place in the annulus. The martensitic formations in the rupture and surrounding the i.d. of the $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$ indicate that localized temperatures of approximately 2000 F existed.
5. The failure was not caused by the sodium environment.
6. No action is evident to decrease the likelihood of a future leak on assembled units.

277-24

FIGURE C-8 CROSS SECTION OF HEATER TUBE THROUGH FAILURE

FIGURE C-9 SECTION THROUGH POTENTIAL RUPTURE IN 5 Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti TUBE

FIGURE C-10 AIR SIDE ID OF 5 Cr- $\frac{1}{2}$ -Mo- $\frac{1}{2}$ -Ti HEATER TUBE
FROM AREA ADJACENT TO RUPTURE

277-21

5% H_2 SO_4 ELECTROLYTIC

250X

FIGURE C-11 INCONEL HEATER SHEATH CRACK

F

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