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MARCH 1971

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**SODIUM TECHNOLOGY DEVELOPMENT PROGRAM
MASS TRANSFER INVESTIGATIONS
IN LIQUID METAL SYSTEMS
QUARTERLY PROGRESS REPORT NO. 16
DECEMBER 1970-FEBRUARY 1971**

U.S. ATOMIC ENERGY COMMISSION
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DECEMBER 1970 - FEBRUARY 1971**

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1. INTRODUCTION AND SUMMARY

1.1 TECHNICAL HIGHLIGHTS

- *Radioisotope transport scoping tests were initiated on schedule using ^{60}Co . Specimens of Type-316 stainless steel were electroplated with ^{60}Co on their outer surfaces and annealed to diffuse the cobalt to a maximum depth of 0.6 mil into the surface. This procedure resulted in specimens with high surface activity, low total activity, and a negligible increase in cobalt content.*
- *Recently completed runs provide the most reliable data and correlations to date on corrosion rates versus oxygen and downstream. Corrosion rates for Type-316 stainless steel were five times greater in high oxygen sodium (cold trap equilibration at 432°F) than in low oxygen sodium (270°F cold trap). These temperatures are equivalent to 20 ppm oxygen and 1.6 ppm oxygen, respectively, by the Eichelberger correlation. Downstream position had a much stronger effect on the corrosion rates measured in the high oxygen test than in comparable low-oxygen tests.*
- *Carbon in the form of acetylide will form in sodium only if calcium is present. Sodium acetylide is not formed in high-purity sodium at 1200°F in iron, nickel, and stainless steel capsules. However, acetylides were formed upon addition of 1 at. % calcium to the sodium. Calcium may thus affect the rate of carbon transport in sodium systems.*
- *The partial pressure of hydrogen in argon over sodium (as well as hydrogen concentration in the sodium), is controllable by cold trap temperature. At concentrations under 2 ppm hydrogen in sodium, the partial pressure in the cover gas decreases with increasing sodium temperature.*
- *An improvement in confidence limits of corrosion rates for four alloys (Types-304, -316, and -347 stainless steel, and Incoloy-800) has resulted from analysis of results of exposure in recent runs in 1300°F sodium up to 15,726 hours.*
- *The efficiency of an unstirred cold trap was found to increase as throughput was increased from 1 to 1.6 system turnovers per hour.*

1.2 PROGRAM DESCRIPTION AND BACKGROUND

Corrosion of candidate structural materials, the influence of corrosion and of impurities in the sodium on engineering properties of materials, and corrosion and deposition of radioactive isotopes from the core cladding are being studied in test loops designed to simulate typical conditions of sodium-cooled reactors. The program is designed to obtain out-of-pile test data that will (when used in conjunction with irradiation data) support engineering design selections and lead to the establishment of specifications for the monitoring and control of impurities in large plants.

Current work is directed to the near-term requirements for data and the development of sodium technology for application to FFTF and LMFBR demonstration plant designs. Emphasis is placed on deposition studies, design data for materials performance, the associated requirements and methods for plant chemistry control, and data on the transport and buildup of radioactive isotopes.

Work initiated during the quarter includes the preparations for experimental determination of corrosion release rates of long-lived radioactive isotopes from the core regions of FTR and LMFBR and their deposition patterns in the piping and other components in the cold leg of the primary sodium system. Emphasis is placed on development of a calculational model, based on accurate experimental data, for predicting the buildup of radioactivity in the primary coolant system of large sodium cooled reactors.

In an earlier 6-year phase of the program, ending in 1965, corrosion and mass transfer of Type-316 stainless steel and 2% Cr-1 Mo and 5 Cr-½ Mo-½ Ti ferritic steels were studied at sodium temperatures to 1200°F. Average corrosion rates were correlated by statistical analysis from which quantitative relations were derived.

In the current phase of the program, the number of materials studied has been extended to include cladding candidates for oxide fuel (Types-304, -316, and -347 stainless steels and Incoloy 800). The temperature range of testing has been increased to 1300°F, and temperature profiles, loop geometry, and sodium purity controls have been established to better represent environments of large fast reactors. Oxygen concentration has been maintained less than 5 ppm, well within recommended prototypic values.

Increasing emphasis has been placed on improving sodium impurity monitoring and control. In-line devices are used to obtain rapid and, where possible, continuous indications of impurity changes. These include chromatography for cover gas, replaceable flow-through sodium samplers, electrochemical cells for oxygen activity, diffusion cells for hydrogen concentration in sodium and on-line distillation.

Two cold traps for control of sodium purity in test loops have been modified with electromagnetic stirring devices to increase cleanup rate. Automatic controls have been added on all operating loops for continuous and unattended operation with an accuracy of $\pm 2^\circ\text{F}$.

1.3 SUMMARY OF TASK OBJECTIVES

The Sodium Mass Transfer Program is organized into five tasks,* of which three are currently active. These three tasks have the following objectives:

Task I — Corrosion-Deposition Studies

The continuous improvement of empirical and rational models for the accurate prediction of corrosion and mass transport in the primary and secondary sodium systems of large plants. This task is organized in subtasks which include experimental determination of corrosion rates and deposition patterns of stainless steel corrosion products as a function of temperature, velocity, and the chemical composition of the sodium, as well as geometric and hydrodynamic factors. A further objective of this task is the development of calculational models to predict the release, transport, and deposition of radioactive isotopes from the core regions of FTR and LMFBR and the consequent buildup of long-lived radioactivity in primary coolant system piping and components.

Task II — Surveillance and Analysis of Impurities in Sodium

The development of improved methods for sampling of sodium systems, impurity monitoring, collection and identification of particulate material, and chemical and structural analysis. An operating objective is to provide well-controlled operation of loops in support of the other tasks.

Task IV — Corrosion and Mass Transport Effects on Composition, Structure and Structural Integrity of Candidate Alloys for LMFBRs

The determination of the relation between mass transfer and sodium impurities and the long term structural properties of specific LMFBR candidate materials, including Types-304 and -316 stainless steel tubing.

*Quarterly Report No. 11 includes a description of all five tasks.

2. TASK I: CORROSION-DEPOSITION STUDIES

2.1 GENERAL

Current work is directed toward:

- The experimental study of the corrosion rates, transport mechanisms, and deposition of corrosion products of Types-304 and -316 stainless steel in pumped sodium systems at top temperatures of 1150°F and 1300°F, and the specification of corrosion allowances criteria.
- The experimental and analytical study of corrosion rates and transport of activated corrosion products from cladding, and the prediction of radioactive buildup factors that describe their deposition in the primary system and its components (plant maintenance and shielding criteria).
- The understanding of the mechanisms of corrosion, mass transport, and deposition, including boundary layer phenomena and fluid dynamic parameters typical of sodium coolant systems in large reactors.

2.2 OPERATION OF TEST FACILITIES

The experimental work is conducted in seven test loops designed to simulate corrosion conditions in the primary coolant system of a typical LMFBF and FFTF.

2.3 TASK I-1: OXYGEN CONTROL TESTS AND CLEANUP RATE STUDY

Objective

The objective of this task is to investigate the effects of low cold trap throughput rates on oxygen control and system cleanup rates.

Technical Summary

a. Low Cold Trap Throughput Test

The low cold trap throughput test was done after first running the cold trap for several days at 335°F (5 ppm O, Eichelberger), with the hot leg of the loop at 1100°F and the cold leg at 800°F. These operating conditions were chosen to simulate the conditions of FFTF. After establishing these initial conditions, the cold trap flow rate was lowered from the usual value of 18 gal/h to 0.47 gal/h—the equivalent of one system turnover per day. Because a flowmeter capable of measuring such a low flow rate was not available, this reduced average flow rate was accomplished by valving the cold trap out of the system for 22 hours and then running the equivalent of one system turnover (11.3 gal) through the cold trap in 2 hours. This procedure was repeated once every 24 hours for a total of 1 week. After the 7-day period, the cold trap was put on line full time at full flow (18 gal/h—0.3 gpm) and 335°F. Means for controlling low flow rates without intermittent operation are being developed.

Impurity concentrations were measured with the plugging indicator and oxygen meter; however, only the plugging indicator data proved reliable.

The plugging indicator (operated in the oscillating mode) showed evidence of a rise in impurity saturation temperature during the test. This behavior of the oscillating plugging indicator indicated that a certain amount of impurity may be released into the loop by valving in the cold trap, even though the cold trap minimum temperature is held constant. To separate out the effect of intermittent cold trap operation from other possible sources of impurities, it is recommended that the cold trap be operated continuously at one throughput per day. A flowmeter capable of measuring this low flow (8×10^{-3} gpm) is being developed but was not available when this test was run.

The results obtained from the electrochemical oxygen meter during the test were inconclusive; the meter did not recover to its initial voltage at the conclusion of the test when the cold trap was operating continuously. Subsequent changes in cold trap temperature produced only 30 to 50% of the expected theoretical voltage response.

Thus, the test in Loop 9 did not conclusively show the effect on oxygen at low cold trap flow because of nonreproducibility of the oxygen meter readings and the possible introduction of impurities from the intermittently operated cold trap.

b. Cold Trap Cleanup Runs

Two cold trap cleanup runs were monitored with an oscillating plugging indicator to determine cold trap efficiency. The plugging indicator results were converted to solubility data by assuming the impurity to be oxygen, based on the Eichelberger relationship. The efficiency was calculated by plotting the reduced driving force, D_R versus the number of system throughputs (see Figures 2-1 and 2-2).

$$D_R = \frac{C - C_e}{C_0 - C_e} \quad (1)$$

where

- C = ppm oxygen measured by plugging indicator,
- C_e = ppm oxygen in equilibrium with cold trap temperature, and
- C_0 = initial ppm oxygen in system.

Figure 2-1. Cold Trap Cleanup Curve at 0.3 gpm as Monitored by Plugging Indicator

Figure 2-2. Cold Trap Cleanup Curve at 0.2 gpm as Monitored by Plugging Indicator

The slope of this plot is related to the cold trap efficiency by the material balance equation,

$$\ln_e \frac{C - C_e}{C_o - C_e} = \epsilon \lambda, \quad (2)$$

where

ϵ = cold trap efficiency, and

λ = number of system throughputs.

The results of the two cleanup runs are shown in the following table:

Cold Trap Cleanup Run Results

Efficiency of Cold Trap	Flow Rate (gpm)	Throughput Time (Minutes)
59±9%	0.3	38
55±7%	0.2	58

C_o = 19.4 ppm O at 430°F cold trap temperature

C_e = 1.1 ppm O at 250°F cold trap temperature

The increase in efficiency for the higher flow rate is consistent with work by Biery and McPheeters,^{1 2 3} which shows an increase in the rate of sodium oxide precipitation with increasing Reynolds number. The stirred cold trap developed by Roy and Pohl,^{4 5} is based on this same observation.

By using specific impurity meters such as the hydrogen diffusion tube, it is possible to measure the efficiency of species removal. Some cleanup rate data are available from the hydrogen diffusion tube studies and are being analyzed at the present time. The results will be reported in the future.

c. Cold Trap Cleanup Runs

The sodium was equilibrated with the cold trap at 430°F. While loop conditions were maintained constant, the cold trap was valved out, cooled to 250°F, and valved in again. Total soluble impurity level was monitored by an oscillating plugging meter while equilibration at 250°F was achieved. Runs were successfully completed for throughput times of 38 minutes and 58 minutes.

2.4 TASK I-2: RADIOACTIVE MATERIALS SCOPING TESTS

Objective

The objective of this task is to establish the sensitivity and operability of radioisotope techniques for measuring the transfer rate and the deposition rate of corrosion products. The initial tests will be designed to determine where deposition of ^{60}Co and ^{54}Mn occurs in a test loop that simulates FFTF.

Technical Summary

Specimen preparation, Loop 9 modifications, and radiological safety procedures required for the initial scoping test were completed during this quarter. Run 9-3 was initiated February 1, 1971, and is continuing.

Radioactive samples consisted of Type-316 stainless steel rings (1.0 in. long X 0.441 in. o.d. X 0.020 in. wall). The samples were electroplated with ^{60}Co (on the outer surface only) and heat-treated in vacuum at 1000°C for 3 hours. The diffusion cycle was determined from a solution of Fick's Second Law for diffusion into a semi-infinite solid from a nonreplenished source of negligible thickness. Values of the diffusion coefficient of Co in stainless steels were not available; consequently, diffusion coefficients for nickel in stainless steel were used in these calculations.⁶ The penetration profile for ^{60}Co into the samples (calculated on the basis of $D_{\text{Co}} = D_{\text{Ni}}$) is shown in Figure 2-3.

As shown in Figure 2-3, approximately 40% of the ^{60}Co remains within 0.0001 in. of the metal surface when the sample is diffused for 3 hours. Even higher surface activities can be attained by shorter exposures. However, it was observed that smearable contamination was undesirably high for diffusion anneals of 1 hour duration.

The obvious advantage of this type of specimen is that it combines a high specific surface activity with a low total activity. For example, an irradiated sample of the same dimension and specific surface activity would contain 100 times more total activity (i.e., dose rates at 10 cm in the order of 10 R/h). In addition, there is a negligible increase in the total cobalt concentration of the steel because only very small amounts (in the order of 10^{-6} gm ^{60}Co) are diffused into each sample. Weights and activities of the samples currently under test in Run 9-3 are shown in Table 2-1.

Figure 2-3. Calculated Concentration Profile for ^{60}Co Diffusion into Stainless Steel at 1000°C

Table 2-1
WEIGHTS AND ACTIVITY OF PLATED RING SAMPLES

Sample Number	Sample Weights (grams)	Activity ^{60}Co (1-19-71) (millicuries)
R3(571-9-E)	3.2409	0.71
R5(571-9-D)	3.3193	0.48
R4(571-9-C)	3.1538	0.35
R1(571-9-B)	3.3607	0.78
R7(571-9-A)	3.2345	0.68
Total		3.00 mCi

Operations Summary

The modifications required on Loop 9 (see Figure 2-4) for the radioisotope transport and measurement scoping test consisted of replacement of the sample exposure test section, the pipe section between the test section and the mixing tee, and both electromagnetic pump cells, and the installation of 2 in. of lead shielding around the test section and interconnecting pipe. In addition, 3-in.-square replaceable peepholes have been cut out of the lead shielding and pipe insulation at two locations to accommodate the beta-gamma counter. A new duct and blower assembly has been added to the smoke abatement system for safety. All electronics and associated equipment have been received. Counting stations have been selected, and are shown in Figure 2-4.

The operations personnel have been issued film badges and have attended two lectures concerning the use and interpretation of the radiation monitoring equipment. A procedure for operating the e.b. (electron beam) heater has been completed.

A dummy e.b. heater anode was inserted in the test section, and the checkout run of Loop 9 was completed. The loop was cold-trapped at $270^{\circ} \pm 2^{\circ}\text{F}$ and associated instrumentation was verified. The radioactive samples were loaded February 1, 1971, and Run 9-3 was initiated.

2.5 TASK I-3: RADIOACTIVE MATERIALS TRANSPORT TESTS

Objective

The objective of these tests is to determine the distribution, magnitude, and rate of deposition of radioactive corrosion products in flowing sodium systems. Particular emphasis is placed on the transport of ^{60}Co and ^{54}Mn in systems simulating FFTF conditions. Experimental methods to be used in these tests will be based on techniques developed in Task I-2.

Technical Summary

Activities in this task are in a preliminary planning stage, pending results from Task I-2.

2.6 TASK I-4: HIGH OXYGEN CORROSION TESTS

Objective

The objective of these tests is to determine the effects of high oxygen concentrations on the corrosion rate of stainless steels, the transport of minor elements, and the chemistry of sodium systems.

Technical Summary

A high oxygen (432°F C.T. temperature) corrosion test was completed during this period. The cold trap in Loop 8 was stacked with Na_2O in order to maintain a high oxygen level. The resulting corrosion rates of Type-316 stainless steel are shown in Figure 2-5, along with low oxygen (270°F C.T. temperature) corrosion data which were obtained at the same velocity and temperature conditions.

As can be seen from Figure 2-5, there is a considerable increase in the corrosion rate due to the increase in oxygen concentration. The most interesting feature of the results is that downstream (L/D) has a much stronger effect on corrosion rates at higher oxygen levels. This is caused by the higher corrosion rates at upstream positions—and more rapid saturation of the sodium with corrosion products, which decreases the chemical driving force for corrosion at downstream positions. This observation also emphasizes the point that effects of oxygen concentration on corrosion rates of stainless steels are dependent on the downstream position of the sample. For example, at zero downstream (extrapolated value), the corrosion rate of stainless steel is 0.5 to 02.75 mil/year (~ 5 times) for cold trap temperatures of 270 to 432°F , respectively. However, at a downstream position of ~ 540 , the corrosion rate is greater by only a factor of 2 (from 0.25 to 0.5 mil/year). Hence, any equation describing the relationship between corrosion rate and oxygen concentration in sodium is meaningless unless the L/D is specified in the equation. The situation is further complicated by the previous observation⁷ that the effect of downstream on corrosion rates of stainless steels also depends on sodium velocity. Hence the studies under the P.A 15

Figure 2-4. Radioactive Transfer Test Loop Showing On-Line Counting Stations (Numbered)

Figure 2-5. Comparison of Corrosion Rates of Type-316 Stainless Steels in High Oxygen and Low Oxygen Sodium

Phase II Mass Transfer Program results show that effects of oxygen, velocity, and downstream on corrosion are interdependent. As a first approximation, during the Phase I mass transfer studies, an empirical corrosion equation was formulated on the assumption that oxygen, velocity, and L/D were independent variables. The present studies show that these are interdependent variables, and their relationships are complex. An expanded data matrix is needed in order to express the corrosion rates for stainless steels in the form of an empirical equation.

The metallographic examinations and XRF analyses of the samples from the high oxygen run are in progress.

Operations

Because of excessive pressure drop between the surge tanks, Loop 8 was dumped on December 14, 1970, to remove samples in the zone of suspected plugging. The Zone N samples were removed under inert atmosphere and sent to VNC for distillation analysis. After the loop was restarted, it was discovered that the plugging had not been eliminated, so the run was terminated on December 22, 1970. All samples were removed, cleaned, and weighed.

The reason for the plugging was that the sample string in Zone M had been detached from the upper flange holder and had slipped down, causing the lowest cartridge to partially block the 1-in. tee inlet. Deposition of particulates progressively blocked the remaining flow passage until the pressure drop through this section of the loop reached 45 psi, compared with a normal 12 psi.

A photograph (Figure 2-6) of the lowest cartridge from Zone M, after being cleaned in alcohol and water, clearly shows the outline of the incoming sodium and the particulate buildup in the close clearance flow path.

Figure 2-6. Lowest Cartridge from Loop 8, Zone M, Showing Sodium Flow Patterns and Particulate Blockage (Run 8-6)

Loop 8 was restarted without samples in order to obtain a 200-gram distillation sample for particulate analysis. After run conditions were established and the sample was taken, the cold trap temperature was lowered in steps to determine the response of the UNC-type oxygen meter. The oxygen meter results are presented under Task 11-1.

The results of Loop 8 impurity monitoring during Run 8-6 are shown in Table 3-1 in Section 3.

Nine on-line vacuum distillation samples were taken during the course of Run 8-6. The average value for distillation runs for Na_2O concentration in the first eight samples was 15.5 ppm, with a standard deviation (1σ) of ± 3.2 ppm. This average value is in agreement with oxygen solubility data of $15.0 \text{ ppm} \pm 2 \text{ ppm}$ as obtained by Rutkauskas.

The UNC oxygen meter also showed a steady increase in the voltage reading, during the end of the run (Table 3-1, Section 3), indicating oxygen activity was decreasing although the cold trap temperature was kept constant. This observation also suggests that the Na_2O which was added to the cold trap prior to the start of the run was being depleted.

2.7 TASK 1-5: PARTICULATE STUDY

Objective

The purpose of this study is to determine the nature of the particulates found in sodium which flows in stainless steel pipes. The data will provide information for the prediction of local deposition in reactor systems.

Technical Summary

Analysis of particulate samples removed from Loop 1 is in progress. Analysis of experimental studies (initiated in contract year 1970) related to the nature of carbon particulates in sodium has been completed.

Previously, it was proposed that carbon is transferred as C_2^{2-} or Na_2C_2 species in sodium systems. Nevzorov⁹ suggested that carbon is transported in liquid sodium by formation of CO due to the reaction of oxygen ions in sodium and carbon in the steel. Recently,¹⁰ it has been proposed that carbon is transported in liquid sodium systems as atomic carbon. In other words, when carbon diffuses out of the steel as atomic carbon in the hot leg, it remains as atomic carbon, and does not form C_2^{2-} or CO species. This hypothesis was based on thermodynamic calculations, which showed that it is impossible to form CO or Na_2C_2 . Furthermore, an analysis of the electronic state of liquid sodium indicates that it is highly unlikely that carbon atoms diffusing out of the steel will form C_2^{2-} species.

An experiment was designed to verify the above hypothesis; i.e., to determine what happens to the carbon atom when it is released into sodium by a diffusion process. A series of six capsules of Fe, Ni, and stainless steel with 20-mil wall thickness was prepared. All the capsules were filled with loop sodium containing <1 ppm Ca and <5 ppm oxygen. About 1 at. % of calcium was added to the sodium of one of the iron capsules. The capsules were sealed by welding, embedded in very fine graphite powder, and heated to 650°C for about 200 hours. After the heat treatment, the entire Na within the capsules was reacted with water. The gases released by this reaction were analyzed with a gas chromatograph (with a detection limit equivalent to 0.3 ppm carbon in sodium). No acetylene was found in the Na-H₂O reaction off-gas, from the Fe, Ni and stainless steel capsules, indicating that no acetylides were formed when carbon diffused through the capsule wall into the Na. Small amounts (<1 mg) of black deposits, which were observed at the bottom of the capsules, were predominantly elemental carbon as determined by wet combustion. The sodium from the iron capsule containing 1 at. % calcium showed the presence of acetylene in the off-gas, indicating that some acetylide (e.g., Na_2C_2 and/or CaC_2) was formed during the heat treatment. It was not possible to isolate the type of acetylide that was formed in the sodium containing 1 at. % calcium. However, thermodynamic data indicate that formation of CaC_2 is feasible. Addition of calcium could also increase the energy levels of the conduction band electrons, which could in turn ionize the elemental carbon to form C_2^{2-} species.¹¹

The present results are consistent with previous observations of acetylides in sodium systems.¹² If the sodium used in dynamic loops contains appreciable amounts of calcium, it is very likely that deposits in the colder regions (e.g., cold traps) will contain some acetylides. Sodium used to start a new loop could contain appreciable amounts of calcium, which could form CaC_2 during the early part of the operation. In principle, in time all the calcium should precipitate in the cold trap as CaO and CaC_2 , and loop sodium should be virtually free of calcium (< 1 ppm), hence further formation of acetylide in the loop will have very low probability.

Operations Summary

Run 1R-3 was concluded on December 4, 1970. The filter was removed and sent to VNC for examination (see history tabulation, Table 2-2), and the samples were removed, cleaned, dried, and weighed. The samples were green in color after the cleaning, and turned a gold color after oven-drying at ~300°F.

2.8 TASK I-6: CORROSION STUDIES AT HIGH REYNOLDS NUMBER CONDITIONS

Objective

Determine the corrosion rate of stainless steels at various Reynolds numbers by simulating in a small loop the boundary layer conditions in reactor systems.

Technical Summary

It has been proposed⁷ that the velocity dependence of corrosion rates of stainless steels is fundamentally associated with the thickness of the liquid boundary layer of the velocity profile at the steel/sodium interface. To verify this hypothesis, a new loop (3R) was designed for corrosion studies. Rotating mandrels are being used to simulate boundary layer conditions

Table 2-2
LOOP 1R FILTER HISTORY

Filter Change	Location	Exposure Hours At Temperature	Filter Temperature (°F)	Filter Material	Average Pore Size	Material Collected (grams)
I	CFH	149	750	Fe	5 μ	
II	CFH + MFH	603	MF-580 CF-560	Ni	5 μ	~3
III	CFH	1347	600	Ni	5 μ	
IV	MFH	1129	525	Ni	5 μ	~3
V	MFH	1877	540	Ni	5 μ	~3
VI	MFH	1534	520	Ni	10 μ	0
VII	MFH	175	725	Ni	5 μ	0
VIII	MFH	115	600	Ni	5 μ	
IX	MFH*	618	600	Ni	5 μ	~4
X	MFH*	989	600	Ni	5 μ	
XI	MFH*	432	600	Ni	5 μ	
XII	MFH*	716	760	Ni	5 μ	

CFH—Cleanup Filter Housing

MFH—Main Filter Housing

* 20 ppm oxygen operation. Other runs, low oxygen (1.6 ppm).

similar to those of sodium flowing through annuli (previous sample configurations). If, at constant temperature and oxygen concentration, corrosion rates depend only on velocity profile, then corrosion data from Loop 3R should agree with results obtained from other loops (8 or 9).

An 800-hour run at 1300°F and 270°F cold trap temperature has been completed during this period. The boundary layer simulated during this run corresponds to turbulent flow through an annulus. The results will be presented in the next report.

Operations Summary

After the bearings in the sample drive rotor were replaced, Loop 3R was started on November 30, 1970. On December 4, 1970, the loop tripped out due to shorted leads external to the level probe. While the level probe leads were being repaired, the sample drive bearings were checked. Sodium was found in the bearing area due to an overly rapid filling of the loop and the slow response of the level probe. Changes in the filling procedure and the installation of a bypass around the flow-through dump tank have successfully eliminated this problem in subsequent fillings.

The hot leg oxygen meter was removed before starting up the loop because the anticipated operating temperature would have caused catastrophic failure of the ceramic and interfered with the run.

The cold leg oxygen meter (air reference, slip cast electrolyte) failed during operation after ~1500 hours of operation. The cause of failure is not known. There were no transient conditions (flow or temperature) when the failure took place. Similar past failures have been traced to attack of the ceramic by sodium.

The first run was completed on February 12, 1971. The samples are presently being removed for processing. The results of impurity monitoring during the run are shown in Table 2-3.

TABLE 2-3
RESULTS OF LOOP 3R IMPURITY MONITORING

Date	PI Runs* Ts (° F)	Rapid Samplers	Cold Trap Temperature (° F)
1/19/71	288	3R-1-1	270±2
1/20/71	282		270±2
1/27/71	No Breaks	3R-1-2	270±2
2/09/71	245	3R-1-3	270±2

*The plugging indicator showed a gradual flow decrease, typical of high purity sodium, and the Ts listed is a result of operator's best judgment of where flow decrease started.

3. TASK II: SURVEILLANCE AND ANALYSIS OF IMPURITIES IN SODIUM

3.1 TASK II-1: OXYGEN MONITORING AND METERS

Objective

The objective is to monitor the oxygen concentration in sodium in the mass transfer loops, using a combination of on-line devices and techniques.

Technical Summary

This task has concentrated on the monitoring of oxygen activity with electrochemical cells. The work includes: tabulation of the behavior of the cells currently in operation; tabulation and evaluation of attempts to calibrate these cells by such techniques as on-line distillation, adjustment of cold trap temperature, vanadium wire equilibration, and amalgamation determinations; and improvement of the reliability of the cells by utilization of any new technology that is forthcoming. The work is performed to disturb major loop experiments as little as possible. Work in Task II-2 will provide additional direction for this task.

Distillation Results for Oxygen, Loop 8

The on-line distillation system was used to determine the oxygen concentration in Loop 8 during Run 8-6. The data are shown in Table 3-1, along with other pertinent monitoring information.

The average value for the oxygen present as sodium oxide was 15.5 ± 3.2 ppm. This value is in good agreement with the solubility data obtained by Rutkauskas for the 430°F cold trap temperature (15.0 ± 2 ppm) but differs from the Eichelberger value of 19.3 ppm. The oxygen present as carbonate (22 ppm) is higher than that observed in previous runs (~ 14 ppm) and may be due either to the introduction of carbonate along with the sodium oxide used to load the cold trap or to carbonate somehow resulting from the high carbon incident in the loop in the preceding run (8-5).

The data obtained by distillation appear to be the most consistent measure of oxygen in this high oxygen run. Oxygen meter readings have been erratic and amalgamation data have been scattered. This is probably due to segregation along the sample tube.

Operations

Loop 8 and 9 Oxygen Meter Operation

Tests were run in Loop 8 to determine the response of the Loop 8 UNC-type oxygen meter to changes in cold trap temperature. The data, which are listed in Table 3-2, show a response of $\sim 1.1 \times 10^2 \text{ volt}^{-1}$ compared with a theoretical response of $0.39 \times 10^2 \text{ volt}^{-1}$. The reproducibility of the meter was not determined.

An electrochemical oxygen cell was operated in Loop 9 for 430 hours. The output of the cell was about 1.6 volts instead of ~ 1.8 volts expected at cold trap temperatures of 255°F . The cell did give consistent readings at higher oxygen levels (e.g., at 330 and 430°F), but did not yield reproducible values at minimum cold trap temperature.

Calibration by Vanadium Wires

Vanadium wires for use in estimating the oxygen levels in loop sodium have been received from ANL. They have been inserted in the hot leg of Loop 9 to monitor oxygen during the radioactive transport test. At maximum loop temperatures (about 1300°F), the vanadium wire technique will not permit measurements above 2 to 3 parts per million oxygen. If amalgamation data are correct, and oxygen levels exceed this value, the wire data will only confirm this fact. The possibility of using niobium wire, which permits detection of oxygen in a higher range, will be explored.

Table 3-1

LOOP 8 IMPURITY MONITORING: RUN 8-6

Date	CT Temperature (°F)	P I Data		O ₂ Meter (UNC Type) (Volts)	On-Line Distillation Oxygen as		Chemical Analysis	
		Lower Break (°F)	Upper Break (°F)		Na ₂ O (ppm)	Na ₂ CO ₃ (ppm)	Sample Number	Total Alkalinity (ppm)
10/09/70	427±2						8-6-16A	100/25
10/14/70	432±2	414	520					
10/14/70		420	515					
10/16/70		400	500				8-6-17A	49
10/21/70					18	41		
10/23/70		410	480				8-6-18A	42
10/25/70		415	495	Meter Not Operating. Replacement Sensor Installed 11/05/70				
10/26/70		415	492					
10/28/70		427	560		15	30	8-6-19A	
11/01/70		435	500					
11/02/70		435	530					
11/03/70		428	515				8-6-20A	40
11/04/70					13	19		
11/10/70		417	None	1.155				
11/11/70		420	None	1.155	11	19	8-6-21A	
11/12/70		420	None	1.155				
11/18/70		420	None	1.155	19	25	8-6-22A	31
11/19/70		435	None	1.155				
11/24/70		419	488	1.160				
11/25/70		415	475	1.162	17	15	8-6-23A	
12/01/70		415	None	1.167	19	18		
12/02/70		415	None	1.167			8-6-24A	
12/04/70		430	None	1.169	13	17		
12/10/70	432±2	415	455	1.173	9	14	8-6-25A	

Table 3-2

LOOP 8 OXYGEN METER (Cu/CuO Reference Electrode)

Date	Cold Trap (°F)	O ₂ Meter (Volts)
1-24-71	432	1.174
1-26-71	400	1.177
1-27-71	350	1.180
1-28-71	300	1.183
1-30-71	250	1.186
2-01-71	250	1.190

3.2 TASK II-2: HYDROGEN-OXYGEN INTERACTION STUDIES

Objective

The objectives of this task include:

- Calibration of air reference and UNC oxygen cells
- Calibration of hydrogen detector (diffusion type)
- Measurements of the effect of hydrogen on the activity of oxygen and vice versa
- Study of cold trap cleanup rates for oxygen and hydrogen
- Interpretation of plugging temperatures in the presence and absence of hydrogen contamination in the sodium

Technical Summary

The present investigation was undertaken to determine the performance of a diffusion-type hydrogen monitor in a cold-trapped sodium loop. The sodium loop used for this experiment is shown schematically in Figure 3-1. The loop was fitted with two cold traps that could be operated independently. Before the start of the experiment, the first cold trap was operated at 250°F for several days in order to clean up the loop sodium. The first cold trap was then valved out of the system and hydrogen was added to the loop by bubbling a mixture of hydrogen and argon under the sodium surface in the cold-leg surge tank. The hydrogen was then collected in the second (clean) cold trap. The hydrogen concentration of the loop was controlled by varying the temperature of the second cold trap.

The device for measuring hydrogen consisted of a diffusion type sensor made from 0.375 in. o.d. X 0.020 in. wall X 19 in. long Type-304 stainless steel tubing attached to an 8 liter/sec sputter-ion pump. The inside of the tube was first pumped down to $<10^{-8}$ torr. After pumpdown, the detector was flanged into the hot leg of the sodium loop so that the outside of the diffusion tube was in contact with the sodium. The current drawn by the sputter-ion pump from the power supply was used as a measurement of pressure.

The sputter-ion pump current was measured at various sensor temperatures, while the hydrogen concentration in sodium was kept constant by the temperature of the second cold trap. The sputter-ion current is directly proportional to the pressure of hydrogen, and the conversion factor is independent of hydrogen pressures below 5×10^{-5} torr.

A conversion factor recommended by the pump supplier was used to convert the ion current into apparent pressures of hydrogen; the results are shown in Figure 3-2. The data were fitted by least-square method to the equation:

$$\log P_{H_2} = \text{Const.} - \frac{Q_D}{RT} \quad (1)$$

The Q_D values shown in Figure 3-2 are computed to 95% confidence limits. It should be pointed out that although the absolute values of the pressures plotted in Figure 3-3 depend on the manufacturer's conversion factor, the slopes of the lines (i.e., the values of Q_D) are independent of the absolute value of the pressures.

In addition to the diffusion tube measurements, a gas chromatograph was used to measure the hydrogen content in the cover gas of the cold leg surge tank. The surge tank was maintained at $\sim 425^\circ\text{C}$. The results of these measurements are shown in Figure 3-3. The line in Figure 3-3 is the result of correlating the present data with those of a number of other investigators.¹³⁻²⁰ The measurements, which span a range of nearly 9 orders of magnitude, were fitted to the equation,

$$\log_{10} P_{H_2} = 11.6 \pm 0.2 - \frac{6100 \pm 100}{T_C}$$

(2)

where

P_{H_2} is hydrogen pressure in torr.

T_C is cold trap temperature in $^{\circ}\text{K}$, and

$\pm$ indicates 95% confidence limits by the method of least squares.

Figure 3-1. Schematic Diagram of Sodium and Cover Gas System, Loop 9

Figure 3-2. Temperature Dependence of Apparent Partial Pressures of Hydrogen

Discussion

a. Effect of Sensor Temperature on Permeation of Hydrogen

The temperature dependence of the flux of hydrogen "J" permeating through the diffusion barrier can be expressed as

$$J \propto (P_1^{1/2} - P_2^{1/2}) \exp - \frac{Q_D}{RT_s} \quad (3)$$

where

P_1 = partial pressure of hydrogen in sodium,

P_2 = partial pressure of hydrogen in the vacuum side,

Q_D = activation energy for diffusion of hydrogen through the sensor membrane, and

T_s = temperature of the sensor membrane.

Figure 3-3. Sodium Hydride Dissociation Pressure

Since the partial pressure of hydrogen at the vacuum side is considerably lower than in sodium, Equation (3) becomes

$$J \propto P_1^{1/2} \exp \frac{-Q_D}{RT_s} \quad (4)$$

Using Clausius-Clapeyron approximation, the temperature dependence of P_{H_2} in sodium at constant concentration of hydrogen can be expressed as

$$P_{H_2} \propto \exp \frac{-\Delta \bar{H}_s}{RT_s} \quad (5)$$

where $\Delta \bar{H}_s$ is the partial heat of solution of the following reaction,

Hence Equation (4) becomes

$$J \propto \exp - \frac{(\Delta \bar{H}_s + Q_D)}{RT_s} \quad (7)$$

If the partial heat of solution of Equation (6) is zero and Kopps-Neuman²¹ rule is obeyed (i.e., $\Delta \bar{C}_p = 0$); then P_{H_2} in sodium, below saturation, is independent of temperature. Hence, Q_D can be obtained from studies of diffusion of gaseous hydrogen through stainless steel in sodium systems. Obviously, this will be true, provided the diffusion of hydrogen through the membrane is the rate determining step for the process, rather than some surface reaction. As can be seen from Figure 3-2, at higher concentrations of hydrogen (>2 ppm) the Q_D obtained in this experiment is found to be 15.7 kcal, which is in good agreement with gaseous diffusion data.²² This result indicates that at higher concentrations of hydrogen, the diffusion of hydrogen through the membrane is the rate controlling step and that P_{H_2} in sodium is independent of temperature, which is also in agreement with the literature.²⁰ However, at lower hydrogen concentrations (<2 ppm) the Q_D value obtained in this experiment is 13.5 kcal, indicating that P_{H_2} below the saturation concentration of hydrogen decreases with an increase in sodium temperature which contradicts other observations.^{17,18,20} The $\Delta \bar{H}_s$ for Equation (6) calculated from the present data is +2 kcal/g-atom H.

b. Effect of Cold Trap Temperature on Permeation of Hydrogen

Since the cold trap contains liquid sodium and excess NaH, the P_{H_2} of hydrogen in loop sodium will be controlled by the dissociation pressure of NaH in equilibrium with liquid sodium according to the following reaction:

then P_{H_2} in sodium at various cold trap temperatures will be

$$P_{H_2} \propto \exp - \frac{\Delta H_D}{RT_c}$$

where ΔH_D is the heat of dissociation of NaH in equilibrium with liquid sodium, and T_c is the cold trap temperature. If the partial pressure of hydrogen is independent of temperature, the slope of the flux-versus- $1/T_c$ curve should yield $\Delta H_D/2$ even if the diffusion membrane is placed at a higher temperature. The present data indicate that at lower hydrogen concentrations (<2 ppm), the hydrogen partial pressure decreased with increasing sodium temperature. Hence, in principle, if one assumes that $\Delta \bar{H}_s$ of the reaction 6 is independent of temperature, then the partial pressures of hydrogen in Na at any sensor temperature can be calculated as:

$$\log_e \left(P_{H_2}^{T_s} \right) - \log_e \left(P_{H_2}^{T_c} \right) = \frac{-\Delta H_s}{R} \left(\frac{1}{T_s} - \frac{1}{T_c} \right) \quad (9)$$

where

$$\left(P_{H_2}^{T_s} \right) = \text{partial pressures of hydrogen at the sensor temperature } T_s,$$

$$\left(P_{H_2}^{T_c} \right) = \text{partial pressure of hydrogen at cold trap temperature } T_c, \text{ and}$$

$$\Delta \bar{H}_s = \text{partial heat of solution.}$$

If the foregoing relationship is used to correct the partial pressure of hydrogen at the sensor temperatures, then the slope of a plot of hydrogen flux versus $1/T_c$ at any sensor temperature should yield a true value of $\Delta H_D/2$.

The present data indicate that $\Delta \bar{H}_s$ at <2 ppm H in Na is between -1.90 and -2.3 kcal; hence a value of -2.10 kcal/gm-atom H was used to correct the partial pressures of hydrogen at various sensor temperatures, using the Equation (9) relationship. The $\Delta \bar{H}_D/2$ obtained from the slope of P_{H_2} versus $1/T$ plot (Figure 3-4) was found to be 11.5 kcal/g-atom of H. The heat of dissociation of NaH as measured by other investigators¹³⁻²⁰ ranges from 11.0 to 14.5 kcal/gm-atom H. If the P_{H_2} of hydrogen was not corrected for temperature (i.e., $\Delta \bar{H}_s = 0$), then the $\Delta H_D/2$ obtained from the present investigation would be found to be about 16 or 17 kcal/gm-atom of H.

c. *Effect of Cold Trap Temperature on Partial Pressures of Hydrogen in the Cover Gas*

There has been no experimental evidence to show whether, in a dynamic sodium system, the partial pressure of hydrogen in the argon cover gas is controlled by the cold trap. Therefore, the partial pressure of hydrogen over sodium at 800°F was measured by gas chromatography, and is shown in Figure 3-3. The present data indicate that partial pressures of hydrogen in the cover gas are in good agreement with dissociation pressures of NaH in equilibrium with liquid sodium. This observation implies that partial pressures of hydrogen in the cover gas in a dynamic sodium system are controlled by the cold trap temperature.

Figure 3-4. Apparent Partial Pressure of Hydrogen at Various Cold Trap Temperatures

3.3 TASK II-3: ANALYSIS OF SURVEILLANCE SAMPLES

Objective

The purpose of this task is to provide routine surveillance of the purity of loop sodium and cover gas. The emphasis is primarily on the analysis of sodium taken in flow-through samples for total alkaline impurities, by the amalgamation technique.

Technical Summary

a. Total Alkalinity

Recent data are summarized in Table 3-3.

Table 3-3

TOTAL ALKALINE IMPURITIES IN LOOP SODIUM FLOW-THROUGH SAMPLES - AMALGAMATION DETERMINATIONS

Sample Number	Date Taken	Total Alkalinity (ppm)*	Cold Trap Temperature (°F)
1R-3-8A	11-19-70	21	432±2
8-6-18A	10-23-70	42	↕
8-6-20A	11-04-70	40	
8-6-22A	11-18-70	31	
8-6-19A	10-28-70	46**, 39	432±2
6R-17A	10-23-70	12**, 13	270±2
4R-2-29A	11-20-70	13**	270±2

*Total alkalinity is calculated as oxygen and assumes that the amalgamation residue consists only of Na₂O.

**By the interim standards methods described in ANL/ST-6 p. 50, see ensuing discussion.

The results are in the usual ranges for the respective loops.

Two samples were run simultaneously by our usual technique (ASTM-STD) and by the interim standard method specified by ANL. These were samples 6R-17A and 8-6-19A. The results are in good agreement; the estimated precision of the method is ± 30%.

Since there is little obvious difference between the techniques in method, equipment, cost, and resulting data, it is planned to use the ANL technique exclusively henceforth. There are details in the ANL method that are poorer than those in the ASTM procedure, and comments on these will be forwarded to the "standards" committee.

b. Determination of Carbon

Two samples have been analyzed for total carbon. The data are shown in Table 3-4.

The intercomparison of carbon techniques was continued. A sample from WADCO was analyzed. The data obtained indicated a total of 27 ppm carbon, of which 5 ppm arose from easily hydrolyzable species. The WADCO results were 2.0 ppm for total carbon. By a separate technique, WADCO finds the hydrolyzable species to be 1.4 ppm.

Table 3-4
DETERMINATION OF CARBON
IN LOOP SAMPLES

Sample	Total Carbon (ppm)
8-6-17A	19
1R-3-5A	34

A sample of the loop sodium, which has been analyzed by WADCO, EBR-II and GE (data reported in the last quarterly), has been sent to the National Bureau of Standards for activation analysis of carbon.

It is clear from these data, and data reported previously, that there is a functional disagreement between the analytical procedures. We have examined our technique over a number of years and can find no real source of contamination to account for the high values. Similarly, both EBR-II and WADCO can add carbon and recover it, and they are certain they are not losing any to account for the lower values. An overriding point to bear in mind is that neither method gives any indication of the carburization potential of a system. Because of this, changing to the ANL standard technique is of no value, in a practical sense, in our work. (Since the equipment cost to do so is very high, it would be preferable to ship samples to EBR-II or FFTF for analysis as an alternative to setting up the recommended method.) A final point, not emphasized in the last quarterly, is that by definition our sodium conforms to "FFTF quality," since a Loop 5R sample was found to contain 0.5 ppm carbon by the "standard" technique.

3.4 TASK II-4: PARTICULATE ANALYSIS

Objective

The objective of this task is to determine the chemical composition of deposits and in-stream particulates in flowing sodium systems. This task includes the work on the characterization of deposits and particulate materials obtained from filter traps and sample surfaces. It is performed as part of the study to predict local deposition effects and is needed to improve estimates of the radioactivity levels of reactor components.

Operations

a. Filtered Particulates from Loop 1

A fourth filter has been obtained from the Loop 1 high oxygen run (see 15th quarterly for details concerning the first three filters). This filter, No. XII, differed from the preceding ones in that the sampling temperature was 760°F instead of 600°F. The product obtained after distillation was also different in that it was black instead of green, as was true for the three earlier filters.

Filter XI contained 110 mg of residue per filter (330 mg total), while Filter XII contained 85 mg of particulates per filter (245 mg total).

All four filters in this series have been found to contain a larger fraction (75 to 79%) of water-soluble components than was true for the residues from low oxygen runs, which ran in the region of 60 to 70% water soluble.

Analyses for the individual elemental species are in progress. Work completed to date includes the determination of carbon in two filters. No. IX contained 0.86%, while No. XI contained 0.75% total carbon. Their values are about the same as those found for the residues from the low oxygen runs.

Silicon analyses are reported in Table 3-5.

Table 3-5

ANALYSES OF FILTER RESIDUES

Filter Designation	Si (%)
IX	21.4
X	18.8
XI	10.1
XII	----

The silicon values are two to three times higher than observed in the low oxygen runs and indicate an apparent increase in silicate formation because of the high oxygen levels.

Microprobe analyses indicate that iron is more homogeneously distributed in the green particles and at a higher concentration than was observed for the black particles obtained in low oxygen runs at equivalent times of loop operation.

b. Deposited Particulates from Loop 8

Three cartridges taken during Run 6 (high oxygen) from separate zones of Loop 8 have been freed of sodium in preparation for a characterization of the deposited materials.

2

2

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4. TASK IV: MASS TRANSFER EFFECTS ON STRUCTURE AND MECHANICAL PROPERTIES OF AUSTENITIC STAINLESS STEELS

Objective

The objective of this task is to measure the effects of sodium exposure on the composition, microstructure, and high temperature stress rupture properties of stainless steels.

Technical Summary

a. Loop 4R, Run 4R-2, 1300°F

Loop operations were completed during the report period. The run reached a total of 15,726 hours at 1300°F, with 15,248 hours under pressurization. The 16 stress-rupture samples were removed from the test facility and were visually evaluated. The results are shown in Table 4-1. The 15 failed samples all exhibited normal tube failures, which are fine cracks as opposed to the explosive ruptures of a burst test. The 16th sample was slightly expanded (indicating that internal pressurization had occurred), but did not exhibit cracks. Initially, the stress-rupture samples will be evaluated for surface compositional changes (XRF), bulk interstitial and minor element changes, and structure (optical metallography).

Table 4-1

RUN 4R-2, 1300°F IN SODIUM BIAXIAL STRESS-RUPTURE DATA

Material	Initial Hoop Stress (psi)	Time-to-Rupture (Hours)	
		Sample 1	Sample 2
304 MA	5000	6,546	5,187
304 CA	5000	6,228	4,500
304 MA	7000	2,237	2,198
304 CA	7000	2,263	2,188
316 MA	6000	U	10,883
316 CA	6000	14,239	10,852
316 MA	8000	4,415	3,822
316 CA	8000	5,175	5,162

MA - Mill Annealed (ASTM-A213 Grade H)

CA - Carbide Agglomerated, 1650°F for 24 hours

U - Unfailed at end of test (15,276 hours)

Sample 1 is further upstream

The samples for measuring the comparative corrosion of the stainless steels were removed from the test facility, weighed, and are presently being evaluated for compositional and microstructural changes.

The corrosion data from Zone E (maximum upstream area in mechanical property test loops) are shown in Figure 4-1, and the corrosion rates are shown in Table 4-2. These rates are a result of evaluating all the available P.A. 15 corrosion data from the Zone E mechanical property loops (538 to 15,726 hours), and are compared with the previous data (538 to 4,838 hours).

Figure 4-1. Corrosion Rates of Mill-Annealed Stainless Steels from Zone E in 1300°F Sodium

Table 4-2

1300°F CORROSION RATES FROM TEST ZONE E (LOOPS 4R, 5R, 6R)

Material	Rate, Mils/Year, with 95% Confidence Limits	
	Previous Data to 4,838 Hours	Previous and Present Data to 15,726 Hours
316 SS	0.39 ± 0.03	0.33 ± 0.02
304 SS	0.51 ± 0.09	0.40 ± 0.04
347 SS	0.57 ± 0.06	0.48 ± 0.04
Incoloy 800	0.82 ± 0.12	0.60 ± 0.08

The addition of the 15,726 hours data resulted in a reduction of the previously reported corrosion rates and a refinement of the confidence limits for all four alloys. It should be noted that the corrosion data from Run 4R-2 were obtained from tests in which the sodium velocity was limited to 7 ft/sec, as compared with 10 ft/sec in previous runs. This lower velocity may be responsible for the observed decrease in the corrosion rates (Table 4-2). Analysis of corrosion data from Run 5R-5 will add more data for exposures of ~10,000 hours, and will provide further verification of corrosion rates.

Type-321 stainless steel was exposed in Test Zone G, which is somewhat downstream of Zone E (lower corrosion rates). Since this was the initial exposure of Type-321 stainless steel, no cumulative corrosion rate data are available. However, a rate can be calculated on the basis of the 15,726 hours data points. This rate compared with those of the other four alloys in Zone G is shown in Table 4-3. The corrosion rate for Type-321 stainless steel is comparable with that of Type-347 stainless steel and possibly slightly lower.

Table 4-3
PRELIMINARY CORROSION RATES (1300°F)
FROM TEST ZONE G (LOOP 4R)

Material	Rate (Mils/Year)
316 SS	0.12
304 SS	0.21
321 SS	0.28
347 SS	0.30
Incoloy 800	0.53

b. Loop 5R, Run 5R-5, 1300°F

The loop operated at test conditions during part of the report period. The run reached 7,663 hours at 1300°F, with the pressure applied as of February 12. Four samples failed during the report period. The results to date are shown in Table 4-4.

Table 4-4
RUN 5R-5, 1300°F IN SODIUM
BIAXIAL STRESS-RUPTURE DATA

Material	Initial Hoop Stress (psi)	Time-to-Rupture (Hours)	
		Sample 1	Sample 2
304MA	5500	7806*	3939
304CA	5500	U	U
304MA	6000	U	6999*
304CA	6000	7679*	6709
316MA	7000	7780*	U
316CA	7000	U	U
316MA	9000	6727	4676
316CA	9000	U	6124

U - Unfailed

* - Failed this period

Sample 1 is further upstream

c. *Loop 6R, Run 6R-4, 1150°F*

The loop operated at test conditions during part of the report period. The run reached 9,506 hours at 1150°F, with the pressure applied as of February 12. One sample failed during the report period. The results to date are shown in Table 4-5.

Table 4-5
RUN 6R-4, 1150°F IN SODIUM
BIAXIAL STRESS-RUPTURE DATA

Material	Initial Hoop Stress (psi)	Time-to-Rupture (Hours)	
		Sample 1	Sample 2
304MA	18,000	1947	1710
304CA	18,000	5391	6235
304MA	20,000	514	559
304CA	20,000	1662	4006
316MA	20,000	U	639
316CA	20,000	U	U
316MA	22,000	U	7734
316CA	22,000	U	8570

U - Unfailed

Sample 1 is further upstream

d. *Control Tests, Argon, 1150 and 1300°F*

Stress-rupture tests are being run at 1150°F and 1300°F in argon on mill-annealed and carbide-agglomerated Types-304 and -316 stainless steel. The tests are being run for the purpose of supplying comparison (control) data for the tests in flowing sodium. The same heats of materials are being tested in both environments to identify the environmental effects.

The results, plus those for Runs 4R-2 and 5R-5, and the previously reported data from Loop 5R^{2,3} are plotted in Figures 4-2 and 4-3. On the basis of comparison with the control tests, there is a definite decrease in long-term stress-rupture strength due to exposure in 1300°F flowing sodium in a nonisothermal loop.

Samples of stainless steel (Types 304, 316, 321, 347, and Incoloy 800), 0.250 in. o.d. X 0.015 in. wall seamless tubing, have been exposed to 1300°F sodium for various lengths of time in the unstressed condition. Biaxial stress-rupture testing of these samples in argon at 1300°F is in progress. Results from these tests are being reported as failure occurs and compared with results for previously unexposed material. Two new data points have been obtained from the Type-347 stainless steel test and are shown in Figure 4-4. Prior results of these tests^{2,4} were reported in Quarterly Progress Report No. 15.

e. *Loop Operations*

A summary of loop operations is given in Table 4-6.

Figure 4-2. *Biaxial Stress-Rupture Properties of Type-316 Stainless Steel in Sodium and Argon at 1150°F and 1300°F*

Figure 4-3. *Biaxial Stress-Rupture Properties of Type-304 Stainless Steel in Sodium and Argon at 1150°F and 1300°F*

Figure 4-4. Biaxial Stress-to-Rupture Properties of Type-347 Stainless Steel in Argon at 1300°F

Table 4-6
SUMMARY OF LOOP OPERATIONS
November 13, 1970 to February 12, 1971

		Temperature (°F)	ΔT (°F)	Flow (gpm)	CT (°F)*	Time (Hours)
Run	8-6 (Final)	1300	400	5.0	432±2	2,000
	Three-Month Operation					1,028
	Final Run 8-6					1,768
	Total Loop Age					28,087
Run	9-3	1200	400	5.2	270±2	
	Three-Month Operation					911
	Run 9-3					216
	Total Loop Age					23,286
Run	1R-3 (Final)	1300	800	2.0	432±2	2,000
	Three-Month Operation					495
	Final Run 1R-3					2,664
	Total Loop Age					8,993
Run	3R-1	1300	670	0.3	270±2	800
	Three-Month Operation					1,149
	Final, Run 3R-1					819
	Total Loop Age					1,804
Run	4R-2 (Final)	1300	500	7.0	270±5	10,000
	Three-Month Operation					576
	Final Run 4R-2 — 1000°F and Above					15,726
	1300°F and Pressure Applied					15,248
	Total Loop Age					17,628
Run	5R-5	1300	500	10.0	270±5	5,000
	Three-Month Operation					1,245
	Cumulative, Run 5R-5 — 1000°F and Above					7,981
	1300°F and Pressure Applied					7,663
	Total Loop Age					23,473
Run	6R-4	1150	400	7.10	270±5	10,000
	Three-Month Operation					1,194
	Cumulative, Run 6R-4 — 1000°F and Above					9,943
	1300°F and Pressure Applied					9,506
	Total Loop Age					18,618

* CT —Temperature of on-line cold trap.

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