QUARTERLY TECHNICAL PROGRESS REPORT ON AEC-SPONSORED ACTIVITIES

United States Atomic Energy Commission
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ERRATA

The following change should be made in APDA-242:
In Table 11.17, page 11.42, the last entry

Average Weight Gain, g/hr

should be changed to read

Average Weight Gain, $\mu\text{g/hr cm}^2$

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ABSTRACT

This report is a summary of the technical progress made by Atomic Power Development Associates, Inc., in the period March through June 1969 under AEC Contract No. AT(11-1)-865. The individual projects are covered under the following headings:

Sodium-Water Reaction Test Program
Project Agreement No. 10

Sodium Technology Project
Project Agreement No. 11

Compilation of Fermi Experience
Project Agreement No. 15

Fast Flux Test Facility Consulting and Engineering Assistance
Project Agreement No. 16

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Sodium Technology Project - Project Agreement No. 11 - <u>E. C. Kovacic and J. E. Meyers</u>	11.1
<p>Detailed results are reported for a 155-day multitest program conducted in the sodium technology loop starting September 1968 and ending in February 1969, along with a history of the chemistry changes and operating modes of the loop during the test. Also reported are the continuing evaluation of the Rhometer, centrifuge method of sodium purification, hydrogen and uranium gettering, and results of the ASTM round robin on oxygen in sodium.</p>	
Compilation of EFAPP Experience - Project Agreement No. 15 - <u>E. C. Kovacic</u>	15.1
<p>APDA-309, "Primary Sodium Pumps in the Enrico Fermi Atomic Power Plant," was published, as were APDA-CFE-27 through 31.</p>	
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Several discussions were held between PNL and APDA personnel to provide PNL with information concerning the Fermi reactor system.

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SODIUM-WATER REACTION TEST PROGRAM

PROJECT AGREEMENT NO. 10

SENIOR PROJECT ENGINEER: E. C. KOVACIC

PROJECT ENGINEER: H. V. CHAMBERLAIN

I. PREFACE

A. Background Information

Achievement of the LMFBR program objectives is highly dependent on the development of reliable system components. With regard to the current status of development, the steam generator is recognized to be the weakest link in the heat transport system. The question of what occurs in a steam generator during a sodium-water reaction and how to accommodate such a reaction in the design has been a problem since the inception of sodium- and NaK-cooled reactors. The damage resulting from a reaction may be caused by water leaks of two general categories: large leaks and small leaks. Until recently designers have been concerned with large leaks such as those resulting from the complete circumferential rupture of a water tube. However, in late 1962 a steam generator in the Enrico Fermi Atomic Power Plant (EFAPP) experienced a sodium-water reaction due to a tube failure produced by vibration. Subsequent examination of the unit indicated that a general corrosion had occurred in the area of the sodium-water reaction, producing wastage and failure of some of the adjacent tubes. It is not known at what stage in the propagation of the leaks the wastage occurred; that is, did the wastage occur as a result of the initiating leak, or did it occur as a result of the subsequent leaks? The questions raised by this experience clearly indicate that a sodium-water reaction program should consider both small and large leaks. Subsequent to the EFAPP experience, the British conducted some tests in which they found that significant tube wastage rates can be incurred with small leaks.

B. Scope

As part of the AEC program of steam generator development, a project was initiated at APDA for the purpose of investigating both small and large leaks. An initial series of tests is being run to

- Establish a means of estimating the extent of tube wastage during a small leak of water into sodium in a sodium-heated steam generator.

- Determine the dominant parameters of tube wastage during a small water leak so that these parameters may be taken into account in the design of a unit.

C. Relationship to Other Projects

This program is applicable to the development of sodium-heated steam generators and to the technology of sodium-water reactions in the LMFBR program. Data generated in the program will be used to prepare design criteria and design analysis procedures for steam generators from the standpoint of sodium-water reactions.

Information on the test program and the results to date are available in the proceedings of the Sodium Components Development Program Information Meetings held on April 14, 1964, in Hollywood, California, and on June 17, 1965 in Chicago, Illinois; APDA quarterly technical progress reports on AEC-sponsored activities; and in a paper, "An Investigation of Tube Wastage Caused by Small Leaks of Water in Sodium-Heated Steam Generators," presented at the LAEA Symposium on Alkali Metals - Corrosion Studies and Systems Operation Experience, held at Vienna, Austria, November 28 to December 2, 1966.

II. TECHNICAL PROGRESS

A. Rig 10 Tube Wastage (Task 20)

J. A. Ford
G. H. Reicks

1. Testing (Job 03)

The scope of the FY-69 program is to conduct six tests at water injection rates larger than approximately 0.01 lb/sec, approaching 1 lb/sec, to (1) clearly confirm the phenomenon of a decreasing wastage rate that was observed beyond the injection rate of 0.01 lb/sec¹ and (2) determine the effect of gas versus water as the target coolant on wastage rate at the larger leak rates. Because of operating problems, only five of the six scheduled tests were completed. A summary of the data obtained in the five tests is given in Table 10.1 and the wastage results are shown in Figure 10.1. The following includes a discussion of wastage rates, hydrogen behavior in the cover gas, post-test plugging temperatures, and cold-trapping operation.

a. Wastage Results

The five tests reported herein were conducted with a leak-to-target spacing of 1 inch and with 2-1/4 Cr-1 Mo steel as the target material. The first four tests were made with nitrogen as the coolant in the target tube and the fifth test was made with water as the coolant in the target tube. The leak rates studied ranged from 0.009 to 1.74 lb/sec.

Inspection of the data in Table 10.1 shows that the wastage rate continued to increase with increasing leak rate up to approximately 0.07 lb/sec, and that negligible wastage was obtained in those two tests with leak rates greater than approximately 0.3 lb/sec. In these two tests there was only a slight polishing of the tube in the target area. The results from these five tests seem to confirm the trend observed in tests with the 1/4-inch leak-to-target spacing, where the wastage on the target tube reached a peak and then decreased significantly as the wastage rate increased further.

The wastage observed on the specimens from Tests No. 41 and 44 show a definite toroidal-shaped pattern (Figure 10.2). The wastage pattern for Test No. 40, however, does not appear to have the toroidal-shape, although it is difficult to determine because the wasted area is rather large in diameter. It should be noted that in the tests conducted with a 1/4-inch leak-to-target spacing the boundary between the pit and the toroidal-shaped wastage pattern appears to occur just beyond a leak rate of approximately 10^{-2} lb/sec. It would be expected that the toroidal-shaped wastage pattern for the 1 inch leak-to-target spacing would occur at a lower leak rate than for the 1/4-inch spacing because the jet diameter becomes larger as the distance out from the nozzle increases. However, insufficient data exists at this time to state where the transition between a pit and a toroidal-shaped wastage pattern occurs for the 1 inch leak-to-target spacing.

Test No. 44 was conducted with essentially the same conditions as that for Test No. 41 except that water instead of nitrogen was used as the target coolant. The purpose of the test was to verify the previous experience which showed no significant difference in wastage rate at lower leak rates on target tubes containing either nitrogen or water as the coolant. In Test No. 44, a leak developed in the Swagelok fitting that attaches the injection nozzle to the injection water system, and the leak rate could not be determined in the normal manner, i. e., by knowing the amount of water injected into the system and the duration of the injection; instead the leak rate through the nozzle was estimated based on the measured diameter of the capillary. Comparison of the wastage rates for test No. 41 and 44 indicate that a 25% lower wastage rate was obtained with water in the tube. Because of estimating the leak rate and because there is no estimate of the normal variation to be expected in the wastage measurements at this range of leak rate, it can be concluded that the effect on wastage rate of target coolant at a leak rate of approximately 0.07 lb/sec is not significant.

In all Rig 10 tests reported in previous quarterly progress reports no wastage had been observed other than that on the target tubes. However, in several tests reported herein wastage was observed on adjacent tubes and on tube support straps. In Tests No. 41 and 44 wastage was observed on adjacent tubes as well as on the target tubes. In Tests No. 42 and 43 wastage was observed on support straps in the B and C rows, respectively, but not on the target tube or on the tubes behind the support straps. For instance, in

TABLE 10.1 - SUMMARY OF PRELIMINARY DATA FROM RIG-10 TESTS

	Test No.				
	40	41	42	43	44
<u>SODIUM SYSTEM</u>					
Flowrate, gpm	400	400	400	400	400
Velocity past target tube, ft/sec ~	2	2	2	2	2
Bulk temperature, F	615	617	600	610	585
Sodium level above injection point, ft	8.4	8.1	8.3	8.2	8.3
Plugging temperature, F					
Before injection	395	400	385	320	400
After injection	460	425	>600	>630	>580
<u>INJECTION WATER SYSTEM</u>					
Water added, lb	0.22	2.0	10.0	20.0	2.0
Temperature, F	615	617	600	610	585
Pressure, psig	2650	2650	2650	2650	2650
Orifice size, in.	0.010	0.036	0.085 ^N	0.0128	0.038
Orifice length, in.			0.285		
Injection point-to-target spacing, in.	1	1	1	1	1
Injection duration, sec	24.5	28	16*	11.5	14.5
Injection rate, lb/sec	0.009	0.071	0.63	1.74	0.077 (Est.)
<u>RECIRCULATING WATER SYSTEM</u>					
Pressure, psig	100	100	100	100	2650
Coolant	N ₂	N ₂	N ₂	N ₂	N ₂
<u>WASTAGE</u>					
Target material	2-1/4 Cr-1 Mo				
Target tube					
Wastage pattern	(1)	(2)	None	None	(2)
Depth of penetration, mils	14	31	Neg	Neg	12
Wastage rate, mils/sec	0.57	1.11	---	---	0.83
Wastage rate, mils/lb H ₂ O	63.6	15.5	---	---	6
Max measured tube temp, F	1380	1887	1901	ND	1749
Time of max temp, sec	15.9	3.3	15.0	ND	4.4
Depth of penetration, mils					
Target row					
Tube above target tube	---	---	7	---	---
Row behind target row					
Second tube above target $\mathcal{C}$	---	10	---	---	---
First tube below target $\mathcal{C}$	---	15	---	---	7
5-mil-thick SS T/C straps	---		Some Destroyed		
7-mil-thick SS T/C sheaths	---		Some Thinning		
60-mil-diameter CS tube supports	---	---	Wasted 13 at Weld		
Carbon steel cutter assembly	---	---	---	---	~41
<u>COVER GAS</u>					
Pressure, psig					
Before injection	6	5	5	5	5
During injection, peak	16.5	19	20	32	12
After injection	16.5	13	12	11	12
Hydrogen Concentration					
Before injection, ppm	5,000	~0	5,000	2,000	~0
Peak during test, ppm	44,000	370,000	282,000	198,000	275,000
Change in concentration	39,000	370,000	277,000	196,000	275,000
Rate of change, ppm/sec	201	4,017	5,719	4,020	3,630
Elapsed time between leak initiation and initial H ₂ change, sec	75	75	86	59	60
Elapsed time between leak initiation and peak conc., sec	270	206	150	150	196

* Most of water injection appeared to be complete by this time as evidenced by sharp drop in reaction zone temperatures.

(1) Broad shallow wasted area

(2) Toroidal-shaped with central plateau showing some wastage

Test No. 42 wastage was observed on the inside surface of one of the straps, whereas there was no wastage or polishing observed on the tube surface located behind the strap. Figure 10.3 shows schematically the location and general shape of the wastage patterns. The wastage observed on adjacent tubes and support straps in Tests No. 41 through 44 are not plotted in Figure 10.1, mainly because it is not possible to estimate the equivalent leak rate where the wastage occurred. At the 1-inch spacing, the jet fans out more than it does at the 1/4-inch spacing and a portion of the main stream can impact on adjacent tubes; however, it is impossible to estimate what fraction of the main stream impacts on the adjacent tubes. It should be pointed out that where wastage was obtained on both the target and on adjacent tubes, the target tube had the greater wastage. Significant wastage rates were obtained on the adjacent tubes; for instance, in Test No. 44 the wastage rate observed on a tube in row B behind the target tube was approximately 0.4 mil per second, and in Test No. 41 the wastage rate on the adjacent tube behind the target tube was 0.3 mil/sec. Thus, as the leak rate increases above approximately 10^{-2} lb/sec wastage can occur on areas of the tube bundle located either adjacent to or behind the main target area. These results confirm the experience discussed by J. Bray of the UKAEA, wherein he has stated that wastage occurred on tubes away from the target area in tests where leak rates were greater than 10 lb/sec.² Bray concludes that the wastage is caused by a breakup of the main jet of water into smaller jets and that in some cases an optimum-sized jet or sub-jet was formed, resulting in wastage of the tubes.

Inspection of the tube bundles following each test has shown a wide variation in the quantity of products that remain behind on the tube bundle. The normal test procedure is to make a plugging run very soon after the injection of water is completed and then drain the sodium to the dump tank via the filter tank. Thus, the system is usually completely drained within an hour after a water injection. The reaction vessel is then allowed to cool for a period of at least 24 hours, after which the tube bundle is removed from the reaction vessel, the inspection plate is removed from a shroud surrounding the tube bundle, and the target area is inspected. The shroud is then removed and the tube bundle stream-cleaned to remove the products of the reaction. Shortly after the tube bundle is removed from the reaction vessel, observations are made of the target area and of the quantity of reaction product material remaining in the tube bundle. As would be expected, it was found that the quantity of reaction products remaining on the tube bundle increased as the quantity of water increased from 0.2 to 20 pounds. For instance, in Test No. 40, where 0.22 pound of water were added, there was only a slight buildup of reaction products in the target area. In Test No. 41, where two pounds of water was injected, the front half of the tube bundle had a light coating of reaction products. In Test No. 42, where 10 pounds of water was injected, the entire tube bundle was covered with a large buildup of products. In Test No. 43, where 20 pounds of water was injected, there was a uniform coating of

reaction products over the front 300 degrees of the tube bundle, with essentially no products in the back 60 degrees. Both the target and adjacent tubes were clear of products over approximately a three-inch-diameter circle. There was an approximate 1/4-inch-deep layer of reaction products over most of the top of the tube bundle with some areas one to two inches deep; there was approximately a 1/2-inch-deep layer of products on the bottom guides of the tube bundle. In Test No. 44, where two pounds of water was injected, the front half of the tube bundle had a light coating of reaction products. As usual, the target area in all tests except Test No. 44 was essentially free of deposits; in Test No. 44, there was a small conical shaped deposit right at the target area.

b. Hydrogen Behavior in Cover Gas

The Rig-10 system consists of a reaction vessel which, under normal operating conditions, has a cover gas volume of approximately 50 cubic feet. The pressure relief system for the reaction vessel consists of two parallel paths: a 2-inch-diameter line containing a relief valve that begins to open at 11 psi pressure and a 24-inch-diameter line containing a rupture disc with a centrifix separator downstream of it. In all of the tests conducted to date in Rig 10, the relief of gaseous products from the sodium-water reaction have passed through the 2-inch-diameter line containing the relief valve, and at no time has the rupture disc burst. Included in the cover gas system is a nitrogen purge into the pump casing to maintain gas cover in the pump. To prevent pressure surges in the pump from sodium-water reactions in the reaction vessel, a pressure-equalizing line is installed between the pump casing and the reaction vessel. Because of gas leakage out of the system, primarily through the pump case, there is a continuous nitrogen flow into the system. Prior to conducting the sodium-water reaction tests, the cover gas pressure is adjusted to 5 psig and the nitrogen purge rate adjusted to maintain the 5-psig pressure. Immediately upon initiating the water injection, hydrogen produced from the reaction passes either up through the sodium or flows out through the bottom nozzle on the reaction vessel, through the piping to the pump and then into the gas space of the reaction vessel, thereby causing an increase in the pressure in the cover gas space. As soon as this pressure reaches 11 psig the relief valve in the 2-inch-diameter line begins to open and the cover gas flows out of the reaction vessel. This gas passes through a reaction products tank containing transformer oil and then passes out into the atmosphere. Calculations indicate that essentially all of the purged nitrogen escapes from the system through the pump case and not through the reaction vessel.

In the tests reported this quarter, the leak rates are sufficiently high to produce a very rapid increase in pressure in the cover gas space, thereby causing the relief valve to open almost immediately after the injection has been initiated; thus, it is difficult to determine the actual rate of hydrogen increase in the cover gas as the injection is occurring. This determination is further complicated by the fact that there is approximately a 1-minute delay

in the response of the hydrogen detection system after the leak has been initiated. A thermal conductivity cell is used to measure the hydrogen concentration in the cover gas. A review of the hydrogen behavior in the cover gas, as shown by the data in Table 10.1, shows that a period of approximately 60 to 86 seconds occurs before the detector begins to indicate a change in the hydrogen level and that another 60 to 190 seconds elapses before the peak hydrogen concentration occurs. Inspection of the test data shows that there is essentially no difference in the time from the first indication of the leak until the peak hydrogen concentration occurs as the leak rate increased from 0.07 to 1.74 lb/sec. Since the relief valve is opening during the injection, the rate of hydrogen increase shown in Table 10.1 could be somewhat low. Further analysis of this data as well as that of the previous test results will be analyzed and reported at a later date.

c. Plugging Temperature

The normal test procedures call for a plugging temperature of less than 400 F in the sodium prior to initiating the injection of water. Inspection of the data in Table 10.1 shows that the pre-test plugging temperatures ranged from 320 to 400 F. Shortly after the injection period has been terminated, a post-test plugging temperature is obtained. In the tests reported here, these post-test plugging temperatures were obtained over a range of 5 to 18 minutes after the injection periods were completed. To determine accurately the disposition of the products of the reaction requires continuous monitoring of the impurity level of the sodium and of the cover gas. Thus, a plugging temperature taken several minutes after the end of the injection period may not indicate much about the impurity level immediately after initiation of the leak and up through the injection period. However, use of the change in plugging temperature might be useful in making a rough estimate of the reaction product disposition.

An estimation of the reaction product disposition was made using the plugging temperature data from Test No. 40. A calculation was made assuming that the solid products of the reaction consisted of only sodium hydroxide. The increase in sodium hydroxide concentration in the sodium for a plugging temperature change of 395 to 460 F is equivalent to approximately 0.4 lb water, but since only 0.22 lb water was added to the system there cannot be very much additional sodium hydroxide in the sodium. Assuming that the solid reaction products in the sodium are sodium oxide, the calculations indicate approximately 0.18 lb water equivalent added to the sodium; on this basis, approximately 82% of the oxygen in the injection water went into solution. Examination of the plugging temperatures shown for Test No. 41, assuming that sodium oxide was the main reaction product, shows that only approximately 3% of the oxygen went into solution. In Test No. 41 the post-test plugging temperature was not obtained until 18 minutes after the end of the injection period, yet the change in plugging temperature was less than that obtained in Test No. 40, where only approximately one-tenth of the quantity of water was injected. This lower plugging temperature in Test No. 41 does not make sense

because the impurity level of the sodium would be expected to increase with time, resulting in an increase in the plugging temperature. The development of the leak detection criteria and preliminary evaluation of leak detection systems has pointed out the need for knowledge of the reaction product disposition in order to make a proper assessment of the damage that may have occurred. These plugging temperature results point out the need for experimental tests to determine the disposition of the reaction products during sodium-water reactions; however, to obtain meaningful data in Rig 10 installation of additional impurity monitoring devices is required.

d. Cold Trap Operation

Some difficulties were encountered during this report period in cold-trapping the system sodium down to the required test condition of a 400 F maximum plugging temperature. Difficulties were expected before Test No. 40, because the system had been in a shutdown condition for approximately one year. However, cold-trapping periods were also approximately twice as long as normal before Tests No. 41, 42, and 44 could be conducted. The design of the cold trap is such that collection of the sodium impurities appears to occur primarily in the annulus of the cold trap, resulting in almost complete plugging of the annulus and essentially stopping flow of sodium through the cold trap. Because of this, it has been found necessary to periodically heat up the contents of the cold trap to approximately 700 F, circulate sodium through it, and then drain the system to the dump tank. This procedure was followed prior to conducting Test No. 43, which was the last test conducted this report period, and very little time was required to cold-trap the system down to the required 400 F plugging temperature.

2. Test Analysis (Job 04)

M. K. Deora
H. V. Chamberlain

The scope of this job is to perform additional detailed analyses of all Project 10 wastage data to (1) provide a better understanding of the material wastage phenomena, (2) determine the most meaningful correlation of the data, and (3) provide guidance for the experimental program to be conducted in FY-70.

Additional statistical analyses of the material wastage data obtained in the Rig 10 tests were made to verify the conclusions drawn with respect to the significance of leak rate, leak-to-target spacing, sodium velocity past the target tube, and duration of the leak on material wastage rates. Previous analysis had shown that only leak rate and leak-to-target spacing exerted a significant effect on material wastage.³

For a given spacing (1/4 inch or 1 inch), the dependency of material wastage rate on leak rate is shown in Figure 10.1, i. e., an increase

in the leak rate produces an increase in wastage rate up to a point. The wastage rate continues to increase as leak rate is increased up to 10^{-2} lb/sec; there is then a decrease in the wastage rate as the leak rate is increased beyond 10^{-2} lb/sec. This was not observed in the tests conducted in the static systems; hence, many questions have been raised regarding the representation of the data in the form shown in Figure 10.1. The most important question is related to the possibility of a variation in penetration rate with time. The results shown in Figure 10.1 assume no variation in penetration rate with time; if this is not true, it may lead to a wrong conclusion. The duration of injection covered in the leak rate study beyond 10^{-2} lb/sec, ranged from 2.5 to 42 seconds; this is a very short range when compared to the study of leak rates below 10^{-2} lb/sec, for which the duration of injection ranged from 100 to 300 seconds.

For the leak rates beyond 10^{-2} lb/sec, where the injection for some of the tests is even as low as 2.5 and 11 seconds, the wastage rate expressed as mils/sec (which assumes no variation in penetration rate with time) may be misleading if an initial induction time is required for the wastage to begin.

However, one of the most significant observations on the effect of duration, is the high wastage rates obtained in the very short duration tests. For example, in one UKAEA test made in NaOH with the sodium circulating, the penetrations exceeded 0.062 inch in a 2-sec test.² This result indicates that essentially zero induction time is required for wastage to begin.

In APDA Rig 10 Tests No. 38 and 39, complete penetration of the tube was observed. The most significant observation for these tests is that complete penetration occurred in 58 seconds for both tests, even though the leak rates for the two tests were significantly different, 0.0033 lb/sec and 0.0066 lb/sec. The detailed statistical analysis of the data consisted of three phases. The first two phases determined the significance of the four variables using the analysis of variance technique, and the third phase determined the best fit of the data using multiple regression analysis.

A complete factorial experiment of four factors each at two levels requires 16 observations, each under a different set of experimental conditions; however, for the present set of data a full factorial two-level analysis was not possible because of the lack of appropriate experiments required for the analysis. Therefore, only a half replicate design analysis was possible.

Examination of the experimental data showed that the statistical analysis of the effect of the four variables — leak rate, leak-to-target spacing, sodium velocity past the target tube, and the duration of the leak — could be established only over a relatively small range of the variables. These were

leak rate	0.0012 to 0.0023 lb/sec
spacing	1/4 and 1 inch
sodium velocity	1 and 2 ft/sec (nominal)
leak duration	76 to 266 seconds

Table 10.2 presents a summary of the pertinent data for the eight tests used in the analysis. For the analysis, the wastage (in mils) was normalized to durations of 100 and 200 seconds. Use of statistical analysis techniques for evaluating the significance of these four variables requires knowledge of the variance of a given set of data, i. e., the spread of the measured value for tests run at the same conditions. The variance in a measured value consists of two parts, one due strictly to random events that are not controllable and the other due to the variance in the controlled variables. In the Rig-10 wastage program, no duplicate tests were made to establish the expected variance for a given set of data; therefore, in the statistical analysis of the data using the analysis of variance technique, the three- and four-factor interactions were assumed to be a measure of the variance in the expected result. This assumption should not be made when there is prior knowledge that such interactions are or may be significant. For the present case, there was no prior knowledge that these interactions were significant.

TABLE 10.2 - SUMMARY OF WASTAGE DATA USED
IN STATISTICAL ANALYSIS

Target Material		2-1/4 Cr-1 Mo Steel				
Sodium Temperature		600 F				
Injection Water Pressure		2650 psig				
Test No.	Leak Duration, sec	Nominal Sodium Velocity, ft/sec	Leak-to-Target Spacing, in.	Leak Rate, lb/sec	Wastage	
					Penetration*, mils	Rate, mils/sec
13	100	1	1/4	23	0.23	0.0013
9	200	1	1/4	114	0.57	0.0019
7	100	2	1/4	80	0.80	0.0023
12	200	2	1/4	42	0.21	0.0013
6	100	1	1	18	0.18	0.0023
10	200	1	1	0	0	0.0012
11	100	2	1	3	0.03	0.0015
4	200	2	1	69	0.35	0.0022

* Normalized; wastage values normalized to 100 and 200 second durations.

The first analysis, made to establish the significance of four variables (leak rate, target spacing, injection duration, and sodium velocity),

indicated that at the 95% confidence level leak rate and leak-to-target spacing exerted a very significant effect on material wastage rates, while the injection duration and target spacing effects are not separable from the scatter in the test data due to controllable and noncontrollable variables.

A second analysis was made with the same eight pieces of data used in the first analysis except that, since the previous analysis clearly showed that the effect of sodium velocity was negligible, the data were used to set up complete replicate experiments of three factors at two levels each. The statistical analysis of this complete replicate experiment showed that all three variables, i. e., leak rate, leak-to-target spacing, and duration, exerted a significant effect on material wastage although the effect of both leak rate and leak-to-target spacing was much more significant than that of leak duration.

Thus from all these analyses, it can be concluded that for the existing Rig-10 data, a complete analysis of the data to study the effect of all the four factors on material wastage rate is not possible. However, it is evident that the significance of only leak rate and target spacing can be separated from the estimated variance for experimental error, and they are very significant at the two levels of study, while the effects of duration (100 to 200 seconds) and sodium velocity (1 to 2 ft/sec) on wastage are not separable from the effects of scatter in experimental results due to controllable and uncontrollable variables.

A further analysis of the wastage data was conducted to study the possibility of a variation in penetration rate with time. The test data for the wastage (total penetration in mils) used in this analysis is expressed as wastage rate (mils/sec). The results of this analysis again indicate that the effect of leak rate and target spacing are of more significance in the study of wastage rate (mils of penetration). The effects of sodium velocity (1 to 2 ft/sec) and injection duration (100-200 seconds) on material wastage rate are less significant than that of experimental error. Therefore, a correlation between leak rate and wastage rate without including the duration effect is a valid one. In order to further demonstrate that the effect of duration on material wastage rate is insignificant, a multiple regression analysis for two independent variables (leak rate and duration versus a dependent variable of total wastage) was done. The results showed that the effect of leak rate is the controlling factor in material wastage rate and that the effect of leak duration, i. e., a change in penetration rate with time, is generally very minor. No further work on the effect of leak duration is planned although test conditions will be chosen where possible to obtain significant wastage.

3. Design, Procurement, Fabrication
and Installation (Job 12)

J. A. Ford
G. H. Reicks

The installation of the larger capacity water injection system and the new five-bottle header including regulator for the 6000 psi nitrogen supply were completed early in this report period. Also, all the stainless steel and the 5 Cr-1 Mo tubes in row B of the tube bundle were replaced with 2-1/4 Cr-1 Mo tubes prior to conducting Test No. 41.

B. Leak Detection (Task 22)

The scope of the acoustic detection program is to establish the feasibility of this technique for detecting small leaks of water or steam into sodium. Preliminary acoustic data obtained in Rig 10 tube wastage tests conducted during FY-68 did show that a detectable noise level above the background noise existed during the injection of the water into sodium. The scope of the FY-69 program is to perform a detailed analysis of the acoustic data obtained in Rig 10 during the wastage tests that are to be conducted in FY-69.

1. Testing (Acoustic) - Job 13

C. C. Scott
S. Barkhoudarian

During wastage Tests No. 40 through 44 (see Table 10.1) acoustic data were recorded with various high-frequency transducers mounted in various positions. Details of the mountings and transducers are given below. Schematic diagrams of the test equipment are shown in Reference 4.

2. Test Analysis (Acoustic) - Job 14

C. C. Scott
S. Barkhoudarian

a. Low Frequency (2 kHz)

To compare the low-frequency results of Tests No. 30 through 39, taken with the Endevco transducer, with those of Tests No. 40 through 44, the output of the Endevco transducer (low-frequency 15-kHz resonance) was recorded along with the Edo transducers (flat ± 2 db to 100 kHz) in Tests No. 40, 43, and 44. In these tests, the Endevco transducer was mounted on a 1/2-inch-diameter, 15-inch-long rod welded to the vessel at the same elevation as the reaction zone and adjacent to the 1-inch-diameter, 13-inch-long rod used for Tests No. 30 through 39.

The amplitude of the acoustic signal versus the leak rate is plotted in Figure 10.4, along with similar data from Tests No. 30 through 39. Considering the higher gain of the Ampex recorder versus that of the Sangamo recorder and the difference in transducer mountings, the results of Tests No. 40 and 44 substantiate the straight-line relationship of around 2 kHz. However, the acoustic result of Test No. 43 (leak rate 1.7 lb/sec),

where the backup pressure momentarily dropped to 1960 psi from 2650 psi, was substantially below the straight line. When audio-monitoring this test, the sound of a very large explosion (compared to other lower leak rate tests) could be heard, followed by a quiet period and then intermittent explosions. The above phenomenon and the fact that in Test No. 43 more liquid-phase was present in the jet are possible explanations for the lower amplitude detected. The backup gas flow noise analysis produced results similar to those obtained from Tests No. 30 through 39. The amplitude of the peaks was smaller and exhibited a larger spread from 2 kHz than did the water-injection spectrum.

The resonances of various modes (transverse, longitudinal, and standing-wave) for various mounting fixtures and vessel walls and tanks were calculated to correlate them with the observed experimental spectra. The transverse-mode resonance frequencies of various mountings were calculated using the equation of one-end-clamped rod frequency to determine whether this 2-kHz spectrum is due to the resonance of the mounting fixtures.⁵

$$f_n = \frac{0.392 c_b D}{L^2} \beta_n^2 \quad (1)$$

where

- f = frequency, Hz
- n = integers 1, 2, 3, ...
- L = length of the rod, cm
- D = diameter of the rod, cm
- c_b = bar velocity, cm/sec
- β_n = constants 0.597, 1.494, 2.5, ... (n-1/2)

First resonance for the 1/2-inch x 15-inch rod (side): $f_1 = 60$ Hz

First resonance for the 1-inch x 13-inch rod (side): $f_1 = 160$ Hz

First resonance for the 1-1/2-inch x 10-inch rod (side valve): $f_1 = 420$ Hz

First resonance for the 1-1/2-inch x 6-inch rod (bottom valve): $f_1 = 1100$ Hz

First resonance for the 1-1/2-inch x 11-foot rod (top): $f_1 = 3$ Hz.

The longitudinal resonances of the above one-end-clamped rods using

$f = \frac{c_b}{4L}$ are respectively, 3.3; 3.5; 5; and 8 kHz.

The standing-wave frequencies in a cylindrical fluid container were studied.⁶ The general form of solution to the wave equation $\nabla^2 p = \ddot{p}/c^2$ in symmetrical cylindrical coordinates, when assuming a nonrigid wall where the pressure instead of the velocity is zero at walls, is

$$p \sim \sin \left(\frac{2 \pi f_z z}{c} \right) J_0 \left(\frac{2 \pi f_r r}{c} \right) \exp (-2 \pi i f_{mn} t)$$

where

$$\begin{aligned} f_z &= \frac{m c}{2 L} \\ f_r &= \frac{c \gamma_n}{\pi D} \\ f_{mn} &= \sqrt{f_z^2 + f_r^2} = \frac{c}{\pi D} \sqrt{\left(\frac{m \pi D}{2 L} \right)^2 + \gamma_n^2} \end{aligned} \quad (2)$$

∇^2 = Laplacian operator

p = pressure

c = acoustic velocity of fluid

$\ddot{p}$ = second time derivative of pressure

J_0 = zero-order Bessel function

$i = \sqrt{-1}$

L = length of vessel

D = diameter of vessel

m = integers 1, 2, 3, ...

γ_n = n^{th} root of $J_0 = 0$; 2.4, 5.52, 8.65, ... (Ref. 7)

f_{mn} = the standing-wave frequency

For Rig 10 filled with 600 F sodium, where $D = 3$ feet, $L = 10$ feet, and $c = 2.5 \times 10^5$ cm/sec, the fundamental standing-wave resonance frequency is $f_{11} = 2160$ Hz. Next, the longitudinal wave in the wall of the vessel (with a bar velocity of the wall = C_w) is considered

$$f_w = \frac{C_w}{\pi D} = \frac{5.1 \times 10^5}{3.14 \times 90} = 1800 \text{ Hz} \quad (3)$$

Since the 2-kHz sound has been heard and spectrally detected from various locations and since the resonance frequencies of the mountings at these locations are so different from each other (as low as 3 Hz and as high as 1100 Hz), it can be concluded that the 2-kHz spectrum heard is not due to the mounting fixture resonances. Since the vessel has only one configuration and consequently only one fundamental resonance frequency, the same argument cannot be used for the 2 kHz signal. However, there are three facts which indicate that the 2 kHz spectrum heard is actually the sound of the reaction. First, the background noise spectrum from Rig 10 tests has a peak at less than 500 Hz and decreases sharply beyond 500 Hz without any noticeable peak at 2000 Hz, as shown in Figure 10.5; but during the water-injection period, a broad peak is produced around 2 kHz. When the background noise spectrum is subtracted (analytically by the computer) from the injection spectrum, the peak at 500 Hz vanishes and the broad peak around 2 kHz is seen (Figure

10.6). Second, a laboratory chemical reaction test in a 7-inch-diameter beaker was conducted; the results indicated the presence of 2-kHz spectrum (refer to paragraph d). Third, an audible distinction can be made between the tone (a measure of spectrum distribution) of the sound of sizzling water injection and that of bubbling backup gas flow or that of background noise.

b. High Frequency (up to 80 kHz)

Acoustic data from Tests No. 40 through 44 were acquired essentially to study the existence of higher frequency characteristic spectra. To achieve this, three high-temperature, high-frequency broadband (300 F, 100 Hz to 100 kHz, ± 2 db) Edo transducers, three 15-kHz high-pass filters (Krohne-Hite and APDA fabricated), a 100-kHz acoustic tape recorder, and high-frequency amplifiers (Hewlett-Packard 465 and General Radio 1560) were acquired and arranged as shown in Figure 10.7 for Test No. 40; Figure 10.8 for Tests No. 41 and 42; Figure 10.9 for Tests No. 43 and 44.

Due to a limited number of transducers, amplifiers, and recording channels during Tests No. 43 and 44, recordings were taken from the bottom valve located near the sodium filter and approximately 20 feet away from the reaction zone (see Figure 10.8). Only during Tests No. 40, 41, and 42 were recordings taken from the mounting at the top of the vessel (see Figures 10.6 and 10.7). Also, during Tests No. 40, 43, and 44, recordings were taken from the 1-inch-diameter, 15-inch-long rod mounted on the side of the reaction vessel adjacent to the reaction zone. Efforts were made to obtain a relationship in high-frequency spectra. Specifically, various amplitude-versus-leak rate and amplitude-versus-frequency graphs were studied; all indicated no conclusive relationship.

c. Jet Noise

An experiment to determine the fluid dynamic effect of the water injection was conducted. In this test the nozzle from Test No. 40 (orifice size, 10.2 mils) was connected through a pressure gauge and regulator to a 6000 psi nitrogen bottle, as shown in Figure 10.10. A predetermined amount of room-temperature distilled water was poured in the pipe and capped off. The valve was opened and water at 2650 psi was injected in the air. In this test the liquid phase of the jet was about 1 foot long and the spray was about 10 feet long. Subsequently, the nozzle was immersed in a 20 x 12 x 10-inch glass tank filled with room-temperature water containing a dye. An Edo SN103 hydrophone and the Endevco 2217E accelerometer were mounted on the side of the tank with cloth tape and magnetic clamp, respectively. A layer of grease was applied between the transducers and the wall of the tank. The output of the Edo transducer was recorded both ahead of and after the 15-kHz filter.

Photographs of the water jet into water taken at a shutter speed of 1/1000 sec showed a liquid phase less than 1 inch long and a spray phase about 12 inches long in a small-angle conic form with small (0.020-inch-diameter) bubbles (see Figure 10.11). When the backup nitrogen flow was photographed, a wide-angle cone with larger (1/8-inch-diameter) bubbles was seen (see Figure 10.12). In addition, peaks around 5, 6, and 14 kHz were observed during the flow of the water jet with no noticeable 2 kHz resonance.

The spectrograms, shown in Figure 10.13, indicated a large amount of high-frequency noise beyond the filtered 15 kHz with a general linear drop in amplitude and peaks at 17 kHz, 38 kHz, and 50 kHz for the water flow test. The backup gas flow was much quieter, with a major peak at 17 kHz.

d. Chemical Reaction Test

Subsequently, a laboratory experiment was conducted to isolate the jet noise and the chemical reaction noise.

A 7-inch-diameter, 8-1/2-inch-long stainless steel beaker wrapped with cloth tape to make it relatively nonreflecting acoustically was filled with 5 inches of distilled water. An argon atmosphere was maintained on the water surface to prevent oxygen from reacting with the generated hydrogen. An upside-down mesh funnel was used to keep sodium chips from bouncing to the wall of the beaker. The Edo and Endevco transducers that were used in the jet test were similarly mounted to the bottom of the beaker (see photograph in Figure 10.14). The output of the Edo transducer was recorded ahead of and after filtering and amplifying (see schematic on Figure 10.14).

A 120-Hz vibrator was used to check the resonant frequency of the beaker. A spectrogram (refer to Figure 10.15a) was obtained which has resonances at 5.6 kHz, 8 kHz, and 12 kHz, but no noticeable resonance at 2 kHz.

When chips of solid sodium were dropped into the beaker, a fizzing sound similar to that of frying was heard; when the chips were submerged, a deep frying sound was heard. Again, the spectrogram obtained with the low frequency Endevco transducer indicated a 2-kHz peak (refer to Figures 10.15b and 10.15c). Higher frequencies with peaks around 11, 19, 30, 40, and 48 kHz in addition to a large high-frequency white noise were also observed (refer to Figures 10.14d and 10.14e). The first standing-wave resonant frequency of the beaker using Eq. (2) is

$$f_{11} = \frac{1.5 \times 10^5}{3.14 \times 7 \times 2.5} \sqrt{(2.4)^2 + \left(\frac{3.14 \times 7}{2 \times 5}\right)^2} = 9 \text{ kHz}$$

The next symmetric overtones are $f_{22} = 19$ kHz; $f_{33} = 29$ kHz; $f_{44} = 40$ kHz; and $f_{55} = 50$ kHz. The wall resonance using Eq. (3) is

$$f_w = \frac{5.1 \times 10^5}{\pi \times 7 \times 2.5} = 9 \text{ kHz}$$

Although neither the wall resonance nor the tank standing-wave first resonances coincide with the observed value (11 kHz), the next symmetric overtones coincide with the observed values. Further work is necessary to clearly establish the frequency spectra from the chemical reaction.

e. RMS Measurement

To verify the audio-detected duration of the injection in Tests No. 40 through 44, a chart recording of the RMS output of each acoustic tape recorder channel was made. The output of a tape recorder channel was fed to a Model 873 Fluke a-c differential voltmeter. The output of the true RMS meter (at 20 mvdc, 100 kHz) was applied to a Sanborn preamplifier and galvanometer. Although no absolute calibration was made because the Sanborn preamplifier was loading the Fluke meter d-c output, a relative measurement was achieved by maintaining the same maximum gain and the same sensitivity in all instruments (see Figure 10.16).

During plotting, the sound was audio-monitored. It was with the help of audio monitoring that the termination of the water injection was distinguished on the chart from the backup gas flow in Tests No. 40 and 43, but in the other tests the termination was very readily determined by visually observing the RMS recordings. The former is due to the capability of the human ear to hear tones, in addition to amplitude, which enables the listener to distinguish between the sizzling and splashing sound of water injection versus the damped bubbling sound of the backup gas flow.

In Tests No. 40 through 44, the acoustic duration of the injection was in excellent agreement with temperature readings of the reaction zone. In Test No. 44, where the nozzle was leaking, the acoustic duration coincided with the reading of five out of six thermocouples, in spite of the fact that the after-test visual observation of the orifice verified the nominal size of the orifice. The nominal injection duration would have been approximately twice that of the actual observed value (see Table 10.1).

f. Summary

- Although the chemical reaction test in the beaker confirmed the existence of a 2-kHz spectrum and a sound similar (to the ear) to that of Rig 10, the possibility of the vessel actually acting like a Helmholtz resonator amplifying its resonant sound should not be overlooked. Even so, this

can enhance the detection of a leak in a working steam generator which has its unique resonant frequency.

- The extremely low-amplitude, nonconsistent high-frequency spectrum observed in Tests No. 40 through 44, with the present external mounting technique, is of no convincing value to the detection of high-frequency spectrum.
- Further simple laboratory tests in a completely nonreflecting container with an immersed pressure transducer are needed first to check the results of the chemical reaction tests and second to verify the existence or lack of high-frequency spectrum associated with the combined presence of water and sodium excluding the high-pressure jet noise.
- A high-temperature pressure transducer immersible in liquid sodium is needed to confirm the existence or non-existence of high frequency caused by sodium-water reactions in existing APDA operating facilities.
- The RMS readings of the tape recorded outputs can be helpful in many cases, although they cannot solely determine the occurrence of leak due to the nonstationary background sound.
- The result of the study of the effect of the transducer location on the output signal during the sodium-water reaction indicated that the spectrograms from the top, side, and outlet valve of the vessel, and another valve located downstream (next to the sodium filter, 20 feet away from the reaction zone) possessed 2-kHz band spectrum. However, the acoustic output of the transducers located on these heavy 4-inch valves indicated louder background noise in the lower-frequency range (less than 1000 Hz) and a weaker signal than that obtained from the side mounting (adjacent to the reaction zone). The transducer located on the valve farthest away from the reaction zone had the loudest background noise because it was picking up the additional noise of sodium flowing through the filter.

3. Background Noise Measurements (Job 17)

C. C. Scott
S. Barkhoudarian

It has been previously reported⁸ that although analysis of the acoustic data obtained in the Rig 10 tube wastage tests showed that the sodium-water reaction did have a detectable noise level, evaluating acoustic detection as a practical leak detection method requires knowledge of the normal back-

ground in real systems. Because of this need, a draft of test plans for obtaining the background noise at EBR-II and SCTI were submitted to the AEC for approval.

During this report period a visit was made to both EBR-II and SCTI to discuss the problems, possible solutions, and requirements in obtaining background noise measurements from their steam generators. The results of the meetings can be summarized as follows:

- The personnel were very enthusiastic about the acoustic leak detection program.
- A test procedure will be required from APDA specifying in detail the (1) instrumentation requirements with detailed specifications for each, (2) desired operating conditions for the steam generators, and (3) a detailed operating procedure.
- Most of the required instrumentation may be available at SCTI while none except a tape recorder and an oscilloscope would be available at EBR-II.
- Review of plant schedules at both facilities indicated that the acoustic tests could probably be conducted sometime within the next nine months.
- Strong justification with assurance that any proposed installation is satisfactory from a safety standpoint will be required to internally mount acoustic transducers on the EBR-II and SCTI steam generators.

4. Leak Detection Criteria (Job 20)

J. H. Coleman
J. A. Ford

The scope of this job is to establish a basis for evaluating methods of detecting sodium-water reactions in sodium-heated steam generators. The need is for early detection of leaks to prevent damage to the unit. Included in this effort are (1) definition of leak detection criteria for the LMFBR program, (2) description of a reference LMFBR secondary sodium coolant system, (3) determination of the behavior of the reaction products throughout the coolant system, (4) assessment of candidate detection systems, and (5) recommendations for a leak detection system for an LMFBR plant.

Work was initiated in FY-68 on establishing the criteria, describing a reference secondary system, and a preliminary evaluation of the candidate leak detection systems. Internal review of a revised draft of the report written this year (to include more up-to-date information) has been completed and efforts are underway to incorporate some additional comments.

C. Sodium-Water Reaction Data Correlation (Task 28)

1. Topical Report (Job 12)

H. V. Chamberlain

The scope of this task was to make a review of the literature pertaining to reactions of water with sodium, with particular emphasis on the factors influencing wastage of materials in such reactions. The main objective of the review was to determine if sufficient experimental data exist to develop a correlation of the various factors which affect materials wastage rates. A draft of the topical report covering this review was submitted to the AEC for review and comments in FY-68.

During this report period the comments received from the AEC last quarter were incorporated into the report and the report was printed. The report will be issued early in FY-70.

D. Hot Erosion Studies (Task 60)

J. A. Ford
G. H. Reicks

The scope of this task was to establish the relative contribution of erosion and corrosion to the wastage process by injecting water, sodium, and sodium hydroxide against targets made of the materials of interest to the LMFBR program. Construction of test equipment and initial operation was completed in FY-68. Several operating problems including failure of the injection tube were encountered during the initial operation period.

In FY-69 the scope of the work was (1) to make an evaluation of the design and performance of the hot erosion test equipment, including analysis of the failed injection tube, and (2) to make recommendations for corrective action.

1. Design Evaluation of Hot Erosion Test Equipment (Job 12)

J. A. Ford
G. H. Reicks

A draft of the report summarizing the test data and the design evaluation, and giving recommended solutions is essentially completed and will be issued internally for review in the near future.

III. NEXT REPORT PERIOD ACTIVITIES

A. Rig 10 Tube Wastage (Task 20)

Four Rig-10 tests (Job 03) will be completed and preliminary test results published. Detailed analysis of existing wastage data (Job 04) will continue.

B. Rig 43 Tube Wastage (Task 21)

Effort will be initiated to conduct three tests (Job 03) to (1) confirm the correspondence in wastage rates between Rigs 10 and 43 at higher leak rates and (2) to determine the effect of reduced injection water pressure on wastage rates.

Fabrication and procurement of the steam injection system for Rig 43 (Job 06) will be initiated.

Preparation of the functional requirements for the modifications to Rig 43 (Job 11) needed to conduct gas space leaks will be initiated.

C. Leak Detection (Task 22)

A revised program plan will be written (Job 12) to (1) determine if high frequencies exist during a sodium-water reaction and (2) if they do exist, to establish the mounting techniques required to detect them.

Measurements will be obtained from the planned tests if approved (Job 13).

The data obtained from the planned tests, if approved, will be analyzed (Job 14).

Internal review of the leak detection criteria report (Job 20) will be completed and the report will be issued with limited distribution as an APDA technical memorandum.

D. Sodium-Water Reaction Data Correlation (Task 28)

APDA-227, the topical report entitled "Evaluation of Material Wastage Due to Reactions of Water in Sodium," will be issued (Job 02).

E. Hot Erosion Studies (Task 60)

The report summarizing the design evaluation of the hot erosion test equipment (Job 12) will be reviewed internally and incorporation of any comments will be initiated.

F. Leak Detection Topical Report (Task 71)

Work will be initiated on planning for the leak detection topical report (Job 01).

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FIG. 10.1 COMPARISON OF MATERIAL WASTAGE RESULTS IN RIG 10 TESTS

Test No.41

Test No.44

117469

FIG. 10.2 TUBE WASTAGE PATTERNS FROM RIG 10 TESTS

Shaded areas denote wastage

FIG. 10.3 SCHEMATIC OF TUBE BUNDLE SEGMENT SHOWING WASTED AREAS

117669

071569

FIG. 10.4 AMPLITUDE OF NOISE VERSUS LEAK RATE AROUND 2 KHZ

071769

FIG. 10.5 SPECTROGRAM OF TYPICAL BACKGROUND NOISE FROM FIG 10

071869

FIG. 10.6 SPECTROGRAM OF BACKGROUND NOISE FROM TEST NO. 43

071669

FIG. 10.7 SCHEMATIC DIAGRAM OF TEST NO. 40

111069

FIG. 10.8 SCHEMATIC DIAGRAM OF TESTS NO. 41 AND 42

FIG. 10.9 SCHEMATIC DIAGRAM OF TESTS NO. 43 AND 44

111869

FIG.10.10 SCHEMATIC OF JET TEST APPARATUS

111269

FIG. 10.11 JET OF WATER INTO WATER

111369

FIG. 10.12 JET OF BACKUP GAS INTO WATER

111769

FIG. 10.13 SIXTEEN-TRACE LOGARITHMIC AVERAGES OF GAS AND WATER JET TESTS

111469

FIG.10.14 CHEMICAL REACTION TEST APPARATUS SHOWN PHOTOGRAPHICALLY AND SCHEMATICALLY

All tests conducted with 500 μ sec
smoothing, ± 4 v attenuation
0 dc input - 0 dc output

**FIG.10.15 SIXTEEN TRACE LOGARITHMIC AVERAGES OF LOW AND HIGH
FREQUENCY CHEMICAL REACTION SPECTROGRAMS**

FIG. 10.16 RMS ACOUSTIC SIGNAL OF TESTS 40 THROUGH 44

SODIUM TECHNOLOGY PROJECT

PROJECT AGREEMENT NO. 11

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I. PREFACE

A. Scope

The present scope of the sodium technology project encompasses the following elements:

1. Impurity monitoring devices, including the development and evaluation of the Rhometer, oxygen meter, hydrogen meter, and plugging meter.
2. Sodium purification processes, including the evaluation and/or development of cold trapping, hot trapping, centrifuging, movable bed gettering, and thermal decomposition.
3. Sodium sampling and analysis, including the reliability of through-flow samplers, oxygen analysis, and hydrogen analysis.
4. Physical and chemical behavior of sodium and impurities, including the investigation of the solubility of hydrogen and sodium carbonate in sodium, the equilibrium distribution of hydrogen between sodium and the cover gas, and interaction of hydrogen and oxygen in sodium.

B. Relationship to Other Projects

This project is oriented entirely to the Commission's research and development program on the LMFBR.

II. TECHNICAL PROGRESS

Covered in this report is the progress in the development and evaluation of the Rhometer, centrifuge method of sodium purification, the solubility of hydrogen in sodium, hydrogen gettering, uranium gettering, and the results of the ASTM round robin on oxygen in sodium. In addition, results

are reported on a multitest program conducted in the sodium technology loop during the period September 1968 through February 1969 including

- A determination of the ambient temperature effect on the United Nuclear Corporation (UNC) oxygen meter at two different oxygen levels in 600 F sodium
- An evaluation of the UNC oxygen meters in high-temperature (900 F) sodium
- An evaluation of the hydrogen stability of loop sodium
- A determination of the sampling reliability of hydrogen
- A determination of the minimum flush time required for tube sampling of 900 F sodium
- A determination of the atmospheric purity requirements when sampling 900 F sodium, and a determination of whether end crimping is an effective means of sealing the ends of sample tubes
- An evaluation of the effect of loop sodium temperature on plugging temperatures.

The history of the test program, the test data, and chemistry changes are illustrated graphically in Figure 11.1.

A. Impurity Monitoring Devices

1. Rhometer (Task 20)

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The objective of this program is to develop an automatic temperature-compensated resistivity meter system capable of operating in the range of 400 F to 1000 F with a sensitivity equivalent to 1 ppm oxygen. The ideal Rhometer measures the change in the resistivity of sodium when changes in the impurity content occur, with the effects of temperature, supply voltage, and other incidental errors eliminated. The automatic compensator system combines a temperature measurement and a sodium resistivity measurement to derive an output in terms of incremental resistivity

$$\Delta\rho_{\text{Na}} = f\left[\rho_{\text{Na}(x)}\right] - f\left[\rho_{\text{Na}(t)}\right] \quad (11.1)$$

in which

$\Delta\rho_{\text{Na}}$ is incremental resistivity of sodium

$\rho_{\text{Na}}(x)$ is measured resistivity of sodium

$\rho_{\text{Na}}(t)$ is resistance temperature function of so-called pure sodium.

A platinum resistance temperature detector (RTD) is used to measure the sodium temperature. The resistance-temperature relationship of the RTD is then used to generate the resistance-temperature relation for sodium

$$f[\rho_{\text{Na}}(t)] = f[\rho_{\text{Pt}}(t)] + f[\rho_{\text{Na}}(t) - \rho_{\text{Pt}}(t)] \quad (11.2)$$

in which $\rho_{\text{Pt}}(t)$ is the resistance-temperature function for platinum.

A schematic diagram of the Rhometer automatic temperature-compensating system is shown in Figure 11.2. Sodium flows through a toroid pipe section. The toroid is a single shorted-turn secondary winding of a current transformer. Changes in the resistance of the sodium in the toroid cause changes in the current and output voltage of the transformer. The output of the toroid transformer, which is a function of sodium resistance, is demodulated by a phase detector, amplified as a d-c signal, and is one input to the automatic temperature-compensating system. The supply voltage for the toroid transformer is an independent 60-cycle a-c power supply which is isolated from the commercial power line and regulated with a high-gain feedback control.

A platinum RTD is mounted at the outlet of the sodium toroid to sense the temperature of the sodium. The RTD output serves as the input to the temperature-compensating system. The signal from the RTD is amplified and is the temperature input to a 20-segment slope generator. From the temperature input signal, the function generator computes the resistance of pure sodium at the toroid temperature. In the final amplifier, the output of the function generator is subtracted from the output of the toroid to obtain the change in sodium resistance due only to impurities.

A photograph of the air-cooled housing covering the toroid transformer is shown in position on the sodium technology loop in Figure 11.3. The transformer windings are thermally insulated from the sodium toroid, magnetically shielded, and the overall assembly is enclosed in an air-cooled housing to maintain a relatively constant ambient temperature.

The electronic components are shown in Figure 11.4.

a. Loop Evaluation, Prototypes (Job 06)

The prototype of the automatic temperature-compensated Rhometer was adjusted on line in the sodium technology loop during the current report period. The loop sodium was cold-trapped prior to and

throughout the entire procedure to maintain the level of impurities as low and as constant as practical. During the adjustment procedure, the sodium temperature was ramped in increments of 30 F from 400 F to 910 F. At each 30 F increment, the slope potentiometer corresponding to that increment was adjusted to produce zero output, thereby matching the sodium resistance value from the toroid.

Precision Power Supply - The 60-cycle power supply that supplies voltage to the toroid transformer completed 5000 hours of continuous operation requiring no maintenance. The output voltage was maintained within a precision of $\pm 0.04\%$ over the entire period and was within $\pm 0.01\%$ for any 24-hour period. The tolerance on the output voltage to meet the overall objective of the Rhometer program is $\pm 0.01\%$. The principal factor limiting the long-term precision of the output voltage is a low level cyclic disturbance generated as a beat frequency between stray induced current from the commercial 60-cycle lines and the 60-cycle local oscillator in the power supply. Methods of reducing this source of error are being studied.

Twenty-Segment Slope Generator - The slope generator produces an output proportional to the difference between the resistivity of platinum and of sodium at a given temperature. This output is the second term in Equation (2). There are 20 segments in the slope generator corresponding to 30 F increments of temperature which are adjusted to match the resistance-temperature curve for pure sodium.

During this quarter, three Rhometer adjustments of the slope generators were necessitated by RTD failures: two adjustment runs were 400 F to 910 F and a third was from 400 F to 730 F. The adjustments of the slope potentiometers at each temperature level were readily made to a precision within $<0.005\%$ of zero. Cold-trap temperatures were held within a band at 215 F to 220 F. Also, with each step increase in temperature, one hour was allowed for the temperatures in the loop to stabilize and to allow any dissolved impurities which may have resulted from the increased temperature to be cleaned up by the cold trap.

Figure 11.5 is a plot of a portion of the temperature ramp-versus-slope generator output, indicating the relative adjustments required to match the output of the toroid transformer to a sodium resistivity curve. The plot is of interest primarily in the temperature range from 700 F to 730 F. It will be observed that a much larger than normal adjustment would have been required if an adjustment had been made immediately after the temperature ramp. The apparent substantial change in sodium resistivity over the temperature range from 700 F to 730 F corresponds to sodium technology loop temperatures at which increases in plugging temperatures have been observed during earlier temperature ramps. Actually, the loop was cold-trapped 16 hours before the adjustment of the slope generator was made. At the time of the adjustment, the output of the toroid transformer

was normal and the adjustment that was required roughly corresponded in magnitude to the adjustments at the lower temperatures.

Resistance Temperature Detector - The RTD is an assembly consisting of a 1/8-inch-diameter, 1-inch-long element and a 1/4-inch-diameter, 12-inch-long support tube. The three platinum RTDs that were installed in thermowells in the loop to replace the defective units,¹ were used for adjusting the automatic compensator from 400 to 910 F. Operation above 800 F was intermittent because of electrical insulation breakdown between the resistance elements and ground.

The RTDs were removed and two of the three were returned to the manufacturer for study. The third, tested at APDA, was heated in short sections along its length, and a d-c potential was applied between the sensing element and the outer sheath. From this test, it appeared that the electrical breakdown was caused by electrolytic (ionic) conduction in the insulation for the lead wires in the support tube. This result confirmed an analysis of the design by the manufacturer of the RTD, wherein it was concluded that the low electrical resistance at elevated temperatures was caused by diffusion of the silver lead wires into the insulation. A modified unit, using a different material for the lead wires, was bench-tested to 730 F with no indication of insulation breakdown. After bench-testing, the modified RTD was installed in the thermowell at the Rhometer toroid in the sodium technology loop, and the automatic temperature compensator was adjusted.

During the period when the failure of the RTDs was being investigated, the automatic temperature compensator was converted to accept a signal from a thermocouple temperature sensor in place of the platinum RTD. Exploratory tests were performed over a limited temperature range to demonstrate that a system of this type could be used, but with reduced accuracy.

2. Oxygen Meter (Task 27)

a. Electrometer Evaluation (Job 03)

C. C. Scott

Due to the excessive zero drift incurred on the tube-type electrometers originally installed to continuously monitor the oxygen meters, a decision was made in April 1968 to obtain a more stable unit. An electrometer was obtained on consignment from Keithley Instruments with purchase contingent upon the unit's meeting specifications. The unit, Keithley Model 602, is a battery-operated solid-state amplifier utilizing a field effect transistor as the input stage. The input resistance is as high as the tube-type units ($>10^{14}$ ohms); the zero drift specification is approximately an order of magnitude better than the tube-type unit.

The unit was tested and found to operate well within its drift specification of 1 mv/day after a 30-minute warmup, after which it was installed to monitor oxygen meter No. 4 in August 1968. Since that time it has continued to operate as specified, i. e., within ± 0.3 mv of zero. It is concluded from the test performed with the Keithley Model 602 solid-state electrometer that it is entirely adequate for the purpose of continuously monitoring the oxygen meters, and no further testing for the electrometer is scheduled.

b. Behavior in High-Temperature
Loop Sodium (Job 05)

A. J. Friedland
 N. N. Mueller

Test Description - Oxygen meter 3 (UNC TPE-1/216) was exposed to 900 F sodium for 37 days. The six chemistry changes in the loop sodium comprising the test program (refer to Figure 11.1) are listed below:

<u>Day No.</u>	<u>Loop Sodium Chemistry Change</u>
8*	Cold-trapped to plugging temperature of 232 F (Prior to cold-trapping, the plugging temperature was 600 F)
15	12.8 grams of NaOH (equivalent to 8 ppm O ₂) injected
21	Hot-trapped at 1000 F
28	12.8 grams of NaOH injected
31	12.8 grams of NaOH injected
34	Cold-trapped to plugging temperature of 254 F.

Test Results and Discussions - The oxygen meter, which had a history of 18 months' exposure in sodium at 600 F prior to this 900 F test, was installed in a 1-inch side loop of the large sodium technology loop. The temperature was raised to 900 F in the 1-inch loop before sodium flow was initiated. The oxygen meter strip chart recorder was started concurrently with the 900 F sodium flow and the initial output was 1156 mv. There was a rapid increase in voltage for one day, but five additional days was required before the output of the oxygen meter reached steady-state conditions.

After 32 hours of steady output of the oxygen meter, the cold trap was started. Thirty-two hours of cold-trap operation reduced the oxygen level from 15 ppm to 6 ppm as measured by chemistry, and the corresponding oxygen meter output changed from 1189 ± 1 mv to 1217 ± 1 mv.

* Day No. 72 of the overall test program as shown in Fig. 11.1

After the cold trap was secured, there was an upward drift of the oxygen meter output voltage for seven days. However, using the measured chemistry at the time of steady output voltage of the oxygen meter before and after cold-trapping, a calibration constant was calculated as follows:

$$\ln \frac{C_2}{C_1} = k (E_2 - E_1) \quad (11.3)$$

where

C = concentration of oxygen in sodium
E = output voltage of the oxygen meter

$$\ln \frac{6}{15} = k (1217 - 1189)$$

$$k = -32.9 \text{ volts}^{-1}$$

which relates oxygen concentration C to voltage output E. The original calibration constant of OM-3 at 600 F sodium was $-22.7 \pm 10 \text{ volts}^{-1}$ (refer to Fig. 10 of Ref. 2); at 600 F, the meter was insensitive to oxygen above 25 ppm. The theoretical calibration of the UNC oxygen meters is -39.5 volts^{-1} (Reference 3) for 600 F operation; for the 900 F, the constant is -30.8 volts^{-1} .

On day 15, 12.8 g of NaOH, equivalent to 16 ppm oxygen and 1 ppm hydride hydrogen, was injected into the loop sodium. From measured chemistry the oxygen concentration changed from 6 to 21 ppm, and the oxygen meter output voltage changed 27 mv. Using the calibration factor of -32.9 volts^{-1} , the oxygen meter output change of 0.027 volts, and Equation (11.3)

$$\ln \frac{6}{C_2} = -32.9 (0.027)$$

$$C_2 = 15 \text{ ppm}$$

This discrepancy between measured chemistry and oxygen meter output may be explained in two ways: First, two days after the NaOH injection the meter failed and a steady-state condition may not have been reached; or second, the injected hydrogen reduced the oxygen activity and, therefore, was not detected by the oxygen meter.⁴ The meter failure is indicated by a rapid increase in output voltage without a corresponding change in sodium chemistry (see Fig. 11.6). If an assumption is made that the oxygen meter is correct, then at the time the hot trap was initiated, the oxygen concentration would have been 3 ppm. From the sodium sample taken the day before, measured chemistry showed an oxygen level of 24 ppm.

Conclusions - During the life of the oxygen meter in the 900 F

sodium, steady state was achieved twice for a total time of 60 hr in which the range of the output voltage was ± 1 mv.

The oxygen meter did respond to the first two changes in the oxygen level in 900 F sodium. The response was, within experimental error, as predicted in theory.³

The life of the oxygen meter in 900 F sodium was 18 days following the 18-month exposure in 600 F sodium.

3. Hydrogen Detector (Task 21)

a. Evaluate Loop Hydrogen Stability (Job 06)

A. J. Friedland
N. N. Mueller

The scope of this job is to establish the hydrogen stability of the sodium technology loop as an initial step in the evaluation program for the hydrogen detector. Testing is conducted in the 600 F to 900 F temperature range and in the 1.5 ppm to 3 ppm hydrogen concentration range.

Chemical analyses of some of the loop sodium samples taken for this evaluation have indicated a higher than expected level in total hydrogen content of the loop sodium. Additional analyses of sample tubes taken in the multitest program are analyzed and discussed in the following paragraphs (see Figure 11.1 for the measured chemistry of the samples analyzed throughout the test period).

Test Results and Discussion - During the first 36 days of the multitest program, the loop sodium was cold-trapped four times and the loop temperature was raised from 600 F to 900 F in 100 F increments. These cold-trapping operations did not reduce the hydrogen level to the anticipated total hydrogen value of 1.5 to 2 ppm. On day 37 with the loop at 900 F, the cold trap was operated for 20 hours. This was the beginning of the 32-day steady-state 900 F test period to determine loop hydrogen stability. Tables 11.1 through 11.4, statistical analyses of measured chemistry of all cold-trapped 900 F loop operation including oxygen values, show that the standard deviation of the hydrogen measurements was of the order of 30% of the mean value.

The 48-hour hot-trapping operation, started on day 85, was followed by two 12.8-g NaOH injections (1 ppm hydrogen and 16 ppm oxygen) and then cold-trapping on day 99. Following the last cold-trapping, the loop hydrogen level was steady for the next 53 days. The loop temperature was reduced to 600 F in 100 F decrements and on day 140, it was raised in a single increment to 900 F. The statistical analyses of these data are shown in Tables 11.5 through 11.8. During this 55-day period of stable hydrogen,

TABLE 11.1 - OXYGEN CONCENTRATION IN
LOOP SODIUM, PPM - FIRST TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
39	10/17/68	9	6	9	8.00	1.41
43	10/21/68	10	9	9	9.33	0.47
50	10/28/68	15	11	12	12.67	1.70
57	11/4/68	24	16	16	18.67	3.77
65	11/12/68	28	19	21	22.67	3.86
71	11/18/68	18	13	13	14.67	2.36
Averages		17.33	12.33	13.33	14.33	Overall Std. Dev.
					Std. Dev. of Avg.	5.72
Within-Site Std. Dev.		6.92	4.31	4.19	5.10	

TABLE 11.2 - HYDRIDE HYDROGEN CONCENTRATION
IN LOOP SODIUM, PPM - FIRST TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
39	10/17/68	0.7	0.6	0.6	0.63	0.047
43	10/21/68	1.2	0.6	1.4	1.07	0.34
44	10/22/68	1.2	0.6	0.85	0.88	0.25
46	10/24/68	1.25	0.75	1.0	1.00	0.20
50	10/28/68	2.0	1.0	2.1	1.70	0.50
57	11/4/68	1.5	1.0	1.0	1.17	0.24
65	11/12/68	0.9	1.1	1.4	1.13	0.21
71	11/18/68	2.2	0.9	1.4	1.50	0.54
Averages		1.37	0.82	1.22	1.14	Overall Std. Dev.
					Std. Dev. of Avg.	0.45
Within-Site Std. Dev.		0.48	0.19	0.43	0.31	

TABLE 11.3 - HYDROXIDE HYDROGEN CONCENTRATION
IN LOOP SODIUM, PPM - FIRST TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
39	10/17/68	1.9	1.7	2.4	2.00	0.29
43	10/21/68	1.0	0.9	0.9	0.93	0.047
44	10/22/68	1.8	1.55	1.25	1.53	0.22
46	10/24/68	2.35	2.8	2.3	2.48	0.22
50	10/28/68	2.80	2.9	2.6	2.77	0.12
57	11/4/68	1.1	1.8	1.1	1.33	0.33
65	11/12/68	2.1	1.2	1.2	1.50	0.42
71	11/18/68	2.7	2.6	2.5	2.60	0.082
Averages		1.97	1.93	1.78	1.89	Overall Std. Dev.
					Std. Dev. of Avg.	0.67
Within-Site Std. Dev.		0.62	0.70	0.68	0.63	

TABLE 11.4 - TOTAL HYDROGEN IN LOOP
SODIUM, PPM - FIRST TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
39	10/17/68	2.6	2.3	3.0	2.63	0.29
43	10/21/68	2.2	1.5	2.3	2.00	0.36
44	10/22/68	3.0	2.15	2.6	2.58	0.35
46	10/24/68	3.6	3.55	3.3	3.48	0.13
50	10/28/68	4.8	3.9	4.7	4.47	0.40
57	11/4/68	2.6	2.8	2.1	2.50	0.29
65	11/12/68	3.0	2.3	2.6	2.63	0.29
71	11/18/68	4.9	3.5	3.9	4.10	0.59
Averages		3.34	2.75	3.06	3.05	Overall Std. Dev.
					Std. Dev. of Avg.	0.89
Within-Site Std. Dev.		0.95	0.78	0.82	0.81	

TABLE 11.5 - OXYGEN CONCENTRATION IN
LOOP SODIUM, PPM - SECOND TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
100	12/17/68	9	9	8	8.67	0.47
106	12/23/68	8	5	6	6.33	1.25
109	12/26/68	7	7	7	7.00	0
120	1/6/69	5	5	5	5.00	0
123	1/9/69	7	5	6	6.00	0.82
142	1/28/69	4	5	6	5.00	0.82
147	2/3/69	3	3	4	3.33	0.47
151	2/7/69	7	7	8	7.33	0.47
154	2/10/69	7	6	7	6.67	0.47
Averages		6.33	5.78	6.33	6.15	Overall Std. Dev.
					Std. Dev. of Av.	1.60
Within-Site Std. Dev.		1.83	1.62	1.25	1.47	

TABLE 11.6 - HYDRIDE HYDROGEN CONCENTRATION
IN LOOP SODIUM, PPM - SECOND TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
100	12/17/68	0.4	0.5	0.7	0.53	0.12
106	12/23/68	0.6	0.5	0.6	0.57	0.047
109	12/26/68	0.8	0.6	0.4	0.60	0.16
120	1/6/69	0.8	0.3	0.4	0.50	0.22
123	1/9/69	0.4	0.4	0.4	0.40	0
142	1/28/69	0.3	0.3	0.4	0.33	0.047
147	2/3/69	0.5	0.3	0.4	0.40	0.082
151	2/7/69	0.5	0.5	0.4	0.47	0.047
154	2/10/69	0.6	0.4	0.4	0.47	0.094
Averages		0.54	0.42	0.46	0.47	Overall Std. Dev.
					Std. Dev. of Avg.	0.14
Within-Site Std. Dev.		0.16	0.10	0.11	0.081	

TABLE 11.7 - HYDROXIDE HYDROGEN CONCENTRATION
IN LOOP SODIUM,PPM - SECOND TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
100	12/17/68	1.8	0.9	1.0	1.23	0.40
106	12/23/68	1.5	1.3	1.3	1.37	0.094
109	12/26/68	1.2	1.3	1.1	1.20	0.082
120	1/6/69	1.4	0.8	1.4	1.20	0.28
123	1/9/69	2.1	1.8	1.6	1.83	0.21
142	1/28/69	1.1	1.2	1.3	1.20	0.082
147	2/3/69	1.5	1.5	1.5	1.50	0
151	2/7/69	1.2	0.9	1.0	1.03	0.12
154	2/10/69	1.2	1.2	1.5	1.30	0.14
Averages		1.44	1.21	1.30	1.32	
					Std. Dev. of Avg.	Overall Std. Dev.
Within-Site Std. Dev.		0.31	0.30	0.21	0.22	0.29

TABLE 11.8 - TOTAL HYDROGEN CONCENTRATION
IN LOOP SODIUM, PPM - SECOND TEST

Day No.	Date	Sample Site			Sample Average	Within-Sample Std. Dev.
		1	2	3		
100	12/17/68	2.2	1.4	1.7	1.77	0.33
106	12/23/68	2.1	1.8	1.9	1.93	0.12
109	12/26/68	2.0	1.9	1.5	1.80	0.22
120	1/6/69	2.2	1.1	1.8	1.70	0.45
123	1/9/69	2.5	2.2	2.0	2.23	0.21
142	1/28/69	1.4	1.5	1.7	1.53	0.12
147	2/3/69	2.0	1.8	1.9	1.90	0.082
151	2/7/69	1.7	1.4	1.4	1.50	0.14
154	2/10/69	1.8	1.6	1.9	1.77	0.12
Averages		1.99	1.63	1.76	1.79	
					Std. Dev. of Avg.	Overall Std. Dev.
Within-Site Std. Dev.		0.30	0.31	0.19	0.21	0.31

the loop was at 900 F for 17 days, 800 F for 4 days, 700 F for 4 days, and 600 F for 30 days.

The variation of the measured hydrogen chemistry is not readily explainable. On October 28, day 54 of loop operation, two sodium samples taken simultaneously were analyzed for hydrogen; the measured chemistry was as follows:

<u>Hydrogen Concentration, ppm</u>	<u>Sample Site</u>					<u>Tube</u>
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>Average</u>
Station 1						
Hydride hydrogen	0.4	0.5	0.4	0.3	0.6	0.44
Hydroxide hydrogen	<u>1.6</u>	<u>1.7</u>	<u>0.9</u>	<u>1.1</u>	<u>1.5</u>	<u>1.36</u>
Total Hydrogen	2.0	2.2	1.3	1.4	2.1	1.80
Station 2						
Hydride Hydrogen	2.0	1.0	2.1			1.7
Hydroxide Hydrogen	<u>2.8</u>	<u>2.9</u>	<u>2.6</u>			<u>2.77</u>
Total Hydrogen	4.8	3.9	4.7			4.47

The variation of these data is a sample of the data that were reported by measured chemistry prior to hot-trapping of the loop sodium. The reported measured oxygen chemistry also shows the same unexplained wide variations as shown in Table 11.1.

The combination of the hot-trapping and the removal of the injected NaOH by cold-trapping stabilized the hydrogen and oxygen levels in the loop sodium.

In addition to the relative standard deviations, there are other indications of the stability of the system. One indication of non-random chemistry measurement would be if the data showed an unusually low number of long runs (a run consists of a number of data points, of which all are above or all are below the median value). However, a test of the data on total hydrogen during the first period of operation according to the method of Chapter 12 of Reference 5 shows that this is not the case, and that the number of runs is consistent with the data behaving in a random manner.

Another indication of random measurements is data showing an overall trend with time. Using the method of Chapter 13 of Reference 5, it is found that the 95% confidence limits on the rate of variation of total hydrogen with time during the first test period is 0.0275 ± 0.0687 ppm/day. Since the range of slope brackets the value zero, the data do not show any trend with time other than would be expected on a random basis.

Conclusions - Hydrogen stability was improved during the last 55 days of loop operation which followed the hot-trapping operation. The improvement in hydrogen stability is indicated by the decrease in relative standard deviation of hydrogen by a factor of 2. The loop was at 900 F for 49 days before hot-trapping, and steady-state loop operating conditions may not have been reached. Any drift of the hydrogen concentration in the loop sodium is obscured by the data scatter, but there is a possibility of increasing hydrogen. Even though the oxygen meter was destroyed in the 900 F sodium (refer to Sec. II. A. 2. b), the response of the meter was 0.25 volts in the direction of decreasing oxygen, indicating that the hot trap had a definite effect on the loop sodium.

The number of runs of high or low hydrogen concentrations and the overall trend of the data with time in each of the two operating periods indicate that the data are random and that the hydrogen concentration did not change significantly with time.

b. Final Design and Installation (Job 08)

J. H. Coleman
J. A. Ford

The scope of this job is the final design and installation of the hydrogen detector into the sodium technology loop. Installation of the hydrogen detector was completed during this quarter. Sodium flow was established through the system at approximately 700 F and 1/2 gpm. This condition will be maintained for the initial clean-up of the system.

During preliminary checkout of the system, the two hygrometer probes which were in the hydrogen detector system were found to be defective. The probes were returned to the manufacturer and replacements are being sent. The cause of the probe failure has not been determined.

c. Data Evaluation (Job 09)

J. H. Coleman
J. A. Ford

The scope of this job is to prepare a mathematical model of the hydrogen detector test stand now installed on the sodium technology loop and to program the model for the IBM 1130 computer. The scope also includes preparation of a program to perform data reduction services for the test programs to be run involving the hydrogen detector. The first set of programs, those for system models, is complete and the programs are running. The second set, data reduction, is 90% complete.

The models predict the performance of the system based upon the theoretical diffusion rate of hydrogen through nickel and the relationship between hydrogen pressure and hydrogen concentration in a sodium system. This ideal model will be a reference system for the data analysis.

Simulation of the calibration tests scheduled for the meter indicated several changes which would be beneficial to the test program.

It is expected that the detection-injection system will not perform exactly as predicted by the theory; therefore, a short-duration test run has been planned which will allow an estimate to be made of the deviation from the model. The model will then be adjusted to more closely conform to the loop installation and the proposed test program will again be simulated incorporating the changes indicated earlier. The output from this simulation will be used to check the data reduction program.

4. Plugging Meter (Task 23)

a. Behavior at High Loop Temperatures (Job 06)

A. J. Friedland
N. N. Mueller

In a previous operation of the APDA sodium technology loop at 900 F, high plugging temperatures were obtained which were neither anticipated nor explainable on the basis of measured chemistry results.² The scope of this job is to collect more data on this phenomenon and to determine what effects conventional cleanup devices such as the cold trap and hot trap have on reducing and stabilizing the plugging temperature and to correlate, if possible, the high plugging temperatures with sodium chemistry.

Table 11.9 is a record of data obtained from these earlier plugging temperature runs.

Hydrogen and oxygen are the two contaminants under study, and the level of hydrogen and oxygen in the sodium is determined by the mercury amalgamation technique. Hot-trapping (zirconium-filled) and cold trapping were the only cleanup devices used in the 120-gallon isothermal sodium technology loop.

Test Conditions - The instrument used in this study is a variable-orifice, air-cooled plugging meter installed in a side loop of the 4-inch loop. The unit consists of a special 1/2-inch bellows-seal globe valve in which the seat is ground to a 15-degree angle for close control of clearance. A special fine thread on the stem and position indicator permits clearance settings to a tolerance of 1 mil. The temperature ramp is controlled by controlling forced air flow past the 1/2-inch finned sodium line by a hand operated slide valve.

The initial sodium flow rate is adjusted to 0.25 gpm, and the flow recorder is adjusted to read between 70 and 80% of the chart at the beginning of a test. Figures 11.7 and 11.8 show data from typical plugging runs that were recorded in this test series.

TABLE 11.9 - SODIUM TECHNOLOGY LOOP HIGH PLUGGING
TEMPERATURES AT LOW IMPURITY

<u>Date 1966</u>	<u>Loop Temp, F</u>	<u>Plugging Temp, F</u>	<u>Impurity Concentrations, ppm</u>			
			<u>Oxygen</u>	<u>Hydride Hydrogen</u>	<u>Hydroxide Hydrogen</u>	<u>Total Hydrogen</u>
12-9	898	573, 577	35	2.4	1.7	4.1
12-12	897	577	27	3.1	1.5	4.6
12-13	898	Cold-Trapping Operation				
12-14	898	450, 490	-	-	-	-
12-15	883	510	10	1.0	1.5	2.5
12-16	895	529	-	-	-	-
12-16	895	Cold-Trapping Operation				
12-16	895	325, 335	5	0.7	1.4	2.1
12-19	879	518, 516	-	-	-	-
12-19	879	Cold-Trapping Operation				
12-19	879	310	5	0.4	1.2	1.6
12-20	875	492, 490	7	0.2	1.3	1.5
12-21	825	508	6	0.3	1.2	1.5
12-22	-	520	-	-	-	-

Test Results - The plugging temperatures during the multi-test program are shown in Fig. 11.9 (a and b), which includes all activities designed to change loop sodium chemistry. There were eight cold-trappings, one hot-trapping, and three 12.8-gram injections of NaOH, each of which is equivalent to a 1 ppm hydrogen injection and an 8 ppm oxygen injection. The temperature of the loop sodium is also shown.

The sodium technology loop, which had some exposure to the atmosphere during maintenance work, was filled with sodium from the 600 F sodium of the storage tank. This sodium was used unaltered during the first ten days of testing, and the contamination level was 42 ppm oxygen and 7 ppm hydrogen with a corresponding plugging temperature of 430 F. The loop was then cold-trapped and the loop sodium contamination reduced to 10 ppm oxygen and 2.5 ppm total hydrogen with a plugging temperature of 227 F. Under these conditions with the cold trap isolated, the loop temperature was raised to 700 F, and in five days the plugging temperature had increased to 520 F. The following four cold-trap operations at 800 F and 900 F loop temperature did not significantly alter the loop sodium chemistry, as determined by the measured oxygen, hydroxide hydrogen, and nonhydroxide hydrogen.

Until the NaOH injection on day 78, as shown on Figure 11.1, the sodium chemistry was not purposefully changed. During 900 F loop sodium temperature operation, the maximum plugging temperature was 620 F.

Hot-trapping at 1000 F also reduced the plugging temperature, but its effect was much slower and the minimum plugging temperature obtained was 265 F after 48 hours of operation. The rise in the plugging temperature was very rapid after the hot trap was secured, with the plugging temperature increasing to 490 F in six hours.

The three injections of NaOH at a loop temperature of 900 F were not detectable by using plugging temperature techniques.

The loop temperature was lowered from 700 F to 600 F on day 109. The 520 F plugging temperature decreased to about 240 F after 70 hours of operation at 600 F and stayed at about 240 F during the next eight days of 600 F operation. The cold trap was initiated on day 121 and was continued for about 24 hours. After isolating the cold trap, the plugging temperature was about 233 F (not significantly different from 240 F) and did not change significantly until the loop temperature was again raised to 900 F.

After 141 days, the loop was again raised to 900 F, and the cold trap was operated continuously for seven days. Two days after the cold trap was secured, the plugging temperature at a loop temperature of 900 F rose from 213 F to 500 F. The tenth day after cold-trapping the plugging temperature was observed to be 520 F, and the test was terminated.

Discussion - From an initial plugging temperature condition of 600 F (cold trap 225 F), when the sodium loop temperature was at 700 F or above, the plugging temperature increased without a corresponding change in total hydrogen and oxygen. This phenomenon was observed after each cold-trap or hot-trap operation. At 900-F loop temperature, the plugging temperature increased to a maximum of 620 F 26 days after the sodium was cold-trapped and stayed at 620 F until the loop was again cold-trapped seven days later. Almost identical conditions were observed during previous 900 F operation.² The plugging temperature was lowered by cold-trapping to 233 F; and 16 hours after the cold trap was secured, the plugging temperature was 510 F.

On day 109 the loop was lowered from 700 to 600 F, and the plugging temperature decreased in 70 hours from 500 F to 240 F. The total hydrogen was 1.8 ppm; the oxygen was 5 ppm.

These test results suggest that the high plugging temperatures are caused by chemical species other than hydrogen and oxygen. Attempts to identify the species including forms of hydrogen and oxygen are being attempted by other investigators.^{6, 7, 8} The solubility of hydrogen and oxygen in 600 F sodium is approximately 15 ppm and 100 ppm,⁹ respectively, eliminating the possibility that hydrogen and oxygen by themselves could cause this plugging temperature effect. The measured chemistry shows that the highest concentrations of hydrogen and oxygen present in cold-trapped sodium were 4.1 ppm and 25 ppm, and from the solubility data of APDA and Reference 9, those concentrations alone could not cause the high plugging temperatures experienced.

Whatever species caused the high plugging temperature, it was also nonstable in 600 F sodium and disappeared in six days at that temperature. An hypothesis is that there is an impurity present in the loop as a solid which is generally not soluble in sodium, but with an increase in temperature above 600 F, the impurity slowly goes into solution. The cold or hot trap can remove the impurity faster than it can go into solution, thus temporarily reducing the high plugging temperature. Another possibility is to consider chemical changes. Because the species disappears in 600 F sodium, it may not survive during cooling of the sample tubes. Its survival time is greater than ten minutes; otherwise the plugging meter would not form a complete plug, but would instantaneously change or break up at the lower temperatures of the plugging meter. Trapping this species for chemical analysis may be very difficult, or impossible.

Conclusions - Under the conditions of relatively clean sodium, i. e., <10 ppm oxygen and <4 ppm total hydrogen, and with the sodium temperatures between 700 F and 900 F, the plugging temperature increased without a corresponding change in the measured hydrogen, hydroxide, and nonhydroxide or oxygen. The maximum conditions observed were 620 F plugging temperature at 900 F loop temperature.

Efforts to reduce the plugging temperature by cold-trapping and hot-trapping were only successful during the cold- or hot-trapping operation. Within 24 hours after the cold trap or hot trap was secured, the plugging temperature had increased to over 500 F. By lowering the loop temperature to 600 F the plugging temperature decreased, and in six days had reduced to 240 F, which is comparable to the plugging temperature before the loop was raised to 700 F 93 days before.

This work confirms the high plugging temperature phenomena previously experienced in December 1966 when the sodium technology loop was operated at 900 F.² This work is also in agreement with the work done at General Electric^{6,7} and at Los Alamos Scientific Laboratory.⁸

B. Sodium Purification Processes

1. Centrifuging (Task 12)

a. Topical Report (Job 15)

J. Matte

A topical report is being prepared on the design, construction, and initial testing of the sodium centrifuge.

The first draft of the report was completed. Several sections of the report were corrected or rewritten, and the corrected draft was distributed for review. The artwork was reviewed and some corrections were made.

b. Examination of Bowl Surface for Impurities (Job 16)

W. E. McHugh

A. A. Shoudy

The final report on the centrifuge bowl analysis work at BMI was received late in the quarter. At present, an evaluation of the data has not been made; it is estimated that it will be completed early next quarter.

c. Test Planning and Evaluation, Run No. 2 (Job 17)

J. Matte

The 100-hour test to demonstrate the capability of the centrifuge to operate over an extended period of time, begun on June 16, was successfully completed in the latter part of June. Plugging of the centrifuge discharge line caused a delay in stabilizing the sodium flow until the afternoon of June 17. Thereafter the centrifuge operated continuously at 50,000 rpm with a sodium flow rate of 1/4 gallon per minute at a sodium temperature of 480 F. The sodium flow was shut off on June 21, after an uninterrupted sodium processing run of 93 hours. The centrifuge was then slowed to 4000 rpm and the heat turned off in order to freeze the sodium remaining in the bowl. The total period of uninterrupted rotation was 127 hours.

The initial run in June, 1968, had been accompanied by a substantial leakage of sodium out of the bowl housing into the glove box, requiring the run to be terminated after 20 hours. Therefore, before starting this run, the leak source at the heater penetration of the housing was plugged and the gas vents adjusted to minimize leakage. One further adjustment to one of the vents was made during the first day's run without stopping the centrifuge or decreasing the sodium temperature (then at 350 F).

Following the long-term run, the centrifuge was disassembled to inspect the drag bushing, the floating seal ring assembly, and the drive spindle. The Oilite drag bushing, which had been impregnated with Dow Corning Silicone Fluid No. 550, appeared dry and tarnished from reaction with sodium. The bushing was covered with sodium when removed from the lower centrifuge housing; however, no noticeable wear was evident in the bore of the drag bushing. The bushing bore was measured to be 0.5037 inch. The bushing diameter before operation was 0.503 ± 0.000 inch.

Upon dissassembly, one of the seal rings was found to be broken, and the spindle was badly scored in the area of the broken seal ring. The spindle was also burned by heat generation from friction of the broken seal ring against the spindle surface. Other than the scoring, however, the spindle appeared to be normal.

d. Test Preparation, Run No. 2 (Job 18)

J. Matte
J. Wooton

This job includes all preparations and modifications necessary to properly prepare the centrifuge and its related equipment for the 100-hour mechanical performance evaluation, including changes to reduce drippage from the bowl housing, procurement of replacement or spare parts, preparation of the test specification, and a brief dry run at the beginning of the 100-hour sodium test.

All preparations were completed.

C. Physical and Chemical Behavior of Sodium Impurities

1. Hydrogen Solubility (Task 31)

A. A. Gordus*
E. F. Hill
S. A. Meacham

a. Planning, Design, and Construction
of Test Apparatus (Job 02)

The scope of this job is to design and construct the apparatus required to achieve the following objectives:

* University of Michigan

- Determine the equilibrium pressure of hydrogen in sodium between the concentrations of 0.1 ppm and 1.0 ppm at two temperature levels in the range of 240 to 1000 F
- Determine the effect of oxygen on hydrogen equilibrium in sodium at two oxygen concentrations.

During this report period, this task was completed.

b. Experimentation (Job 03)

The scope of this job is the execution of an experimental program to achieve the objectives listed above.

The experimental program consists of determining the equilibrium pressure of hydrogen over sodium between the concentrations of 0.1 and 1.0 ppm hydrogen in the sodium. Two sodium-filled nickel capsules, No. 994 and No. 998, were used in the program. The oxygen concentration was the variable between the two capsules. Thus, two isotherms were determined for each capsule.

The program was completed and the results will be presented in a topical report scheduled for publication in FY-70.

c. Data Analysis (Job 05)

The scope of this job includes a reduction of the experimental data to obtain (1) equilibrium isotherms; (2) saturation pressures, temperatures, and hydrogen content; and (3) other thermodynamic data such as heat of solution, heat of reaction, etc.

Evaluation of the experimental data is nearing completion and will be presented in a forthcoming topical report.

2. Cover Gas Impurities (Task 33)

J. H. Coleman
J. A. Ford

a. Hydrogen Gettering in Gas
Sample Lines (Job 02)

The specific objectives of the job are to determine the quantity and composition of deposits collected in the cover gas sampling lines and estimate the error in the cover gas analysis. The sample line which was removed from the system, as reported in the previous quarterly report, has been analyzed for sodium and hydrogen. The total amount of hydrogen in the sampling system was equivalent to not more than 22 ppm hydrogen in the sample stream when averaged over the 82 days the system was in operation. Over the same period, the time-averaged hydrogen

concentration of the gas reaching the chromatograph was approximately 29 ppm. The maximum error involved in the sampling system averaged over this period was 43%. The source of the error is in the final filter section where the sample gas cools to room temperature; 95% of the hydrogen is estimated to be in this section.

In an effort to achieve better temperature control of the sample line, two time-proportioning controllers were installed. Initial checks of these controllers showed they performed satisfactorily and eliminated the effects of the changing room ambient temperature without a wide temperature cycle.

b. Compilation of Chromatograph Experience (Job 03)

The objectives of this job are to compile the data obtained from the chromatograph during FY-69 and evaluate it in terms of loop operation and loop chemistry. Specific data may be chosen from earlier periods for comparative purposes. A preliminary scan of the data compiled thus far shows a pronounced effect of small variations of the sample line temperature on the chromatograph reading for hydrogen. An improved temperature control system has now been installed which it is hoped will eliminate these variations.

In general, the chromatograph does reflect changes which occur in the loop hydrogen concentration when intentional changes are made such as NaOH injections, hot-trapping, and cold-trapping; but it does not accurately reflect the hydrogen content of the sodium as determined by chemical analysis.

In the previous section, it was reported that an amount of hydrogen equivalent to 22 ppm H_2 in the sample gas was trapped in the sample line and that 29 ppm H_2 was recorded on the chromatograph when averaged over the period of operation. The total, 51 ppm H_2 , is the amount leaving the surge tank and presumably is in equilibrium with the sodium. By calculating the hydrogen pressure in equilibrium with the sodium from the chemical analysis of the sodium, it is found that the expected value of hydrogen leaving the surge tank is 425 ppm averaged over the test period. The discrepancy is believed to be due to the gettering of the hydrogen on the cooler surfaces of the surge tank, which are unheated. Heaters have now been installed along the top of the surge tank and the surfaces will be maintained at or slightly above the sodium temperature.

D. Sodium Sampling and Analysis

1. Sampling Reliability (Task 70)

a. Topical Report - Oxygen (Job 03)

M. K. Deora

A. J. Friedland

The first draft of this report has been completed and is being reviewed internally.

b. Hydrogen Sampling Reliability (Job 05)

A. J. Friedland

N. N. Mueller

The objective of this test program is to establish the precision of a combined sampling and analysis procedure when sampling 600 F to 900 F cold-trapped sodium for hydrogen. The following paragraphs describe that portion of the multitest program designed to meet the objectives of loop hydrogen sampling reliability evaluation.

Sampling Procedure - The present test program involves taking samples of sodium from the sodium technology loop and subsequently analyzing the samples in the chemistry laboratory. The samples are taken in two one-half inch stainless steel tubes installed in series, one before (Station 2) and one after (Station 1) the Rhometer, which is located in the one-inch side loop. The Station 2 tube is 36 inches long and the Station 1 tube is 30 inches long; they are located in a glove box having a controlled oxygen-free argon atmosphere.

Sample Tube Preparation - The sample tubes used during the combined test program were prepared with the procedure outlined as follows:

- Rinse the tubes with trichloroethylene to remove grease and other organic residue
- Rinse with 5% caustic solution at 70 °C to complete degreasing
- Rinse with distilled water and dry with acetone
- Vacuum dry overnight inside the laboratory dry box airlock
- With the tube inside the glove box, cap the ends with Gyroloc end cap fittings and seal inside a plastic bag.

The sealed sample tubes are then transferred to the glove-box-enclosed sampling station on the technology loop. Inside the glove box

the sample tubes are removed from the plastic bag, the end caps are removed, and the tubes are inserted in a horizontal position into the two sampling stations. They are then flushed with loop sodium at a flow rate of 0.5 gpm with a flush time of 4 hours.

Sources of Variation - The variation in the reported data results from variations in loop chemistry, in sampling, and in the chemical analyses. The overall variance is the sum of the individual variances from the several independent sources of error. Variations in loop chemistry were minimized by operating the loop with no deliberate changes in chemistry during the test period (refer to Sec. II. A. 3. a). The standard deviation of the test data is a measure of the precision of the combined sampling and analysis procedure when sampling the stable cold-trapped loop for hydrogen.

Test Results and Discussion - The hydrogen sampling test data are divided into two separate test periods of the combined test program. The data used for hydrogen sampling analysis for the first testing period was taken between the first and second cold-trapping of 900-F loop sodium, starting on day 38 of loop operation and running until day 72 (refer to Figure 11.1). The second testing period starts on day 100 and continues until program completion on day 155. During this 55-day period the loop was at 800 F for 4 days, 700 F for 4 days, 600 F for 30 days, and 900 F for 17 days. The two time periods were chosen because in both time periods (refer to Table 11.10 and Figure 11.10) the sodium was cold-trapped to less than 225 F. The sodium chemistry is significantly different in the two periods with the only operational difference being that the sodium was zirconium hot-trapped at 1000 F for 48 hours between the two testing periods. After hot-trapping and subsequent cold-trapping, the total hydrogen decreased from 3.05 ppm to 1.79 ppm, and the standard deviation of the tube average for total hydrogen decreased from 0.266 to 0.116.

Figure 11.1 presents all of the test data from the combined test program; all of the sodium chemistry analyses from cold- and hot-trapped sodium are listed in Table 11.11. The data are divided into two groups, with the hot-trapping on day 85 of the test being the division between the two test groups. The average of total hydrogen during the second period is 1 ppm below that in the first period, a significant difference for the condition of the test. Table 11.12 is a listing of all of the reported chemistry from Station 2 sample tubes, and Table 11.13 is a similar listing of Station 1 chemical analyses.

The sodium samples used for chemical analysis were removed from the sample tube following the site and location identification shown in Figure 11.11. When more than three sites per tube were analyzed for hydrogen, no oxygen analyses were made and the sites were uniformly distributed along the length of the sample tube, with site 1 at the sodium inlet end.

TABLE 11.10 - HYDROGEN SAMPLING STUDY - SAMPLE STATION 2

Sodium Flush Time - 4 hrs
 Loop Temp - 900 F
 Cold Trap Temp - 225 F
1/2" SS Tubes

	<u>Test Period</u>	<u>No. of Sample Tubes</u>	<u>Total Individual Analyses</u>	<u>$\bar{x}$</u>	<u>Tubes</u>		<u>Individual Analyses</u>	
					<u>S</u>	<u>$S/\bar{x}, \%$</u>	<u>S</u>	<u>$S/\bar{x}, \%$</u>
Hydride Hydrogen	1	8	30	1.135	0.315	27.8	0.453	39.9
	2	9	27	0.474	0.082	17.3	0.137	28.9
Hydroxide Hydrogen	1	8	30	1.894	0.627	33.1	0.675	35.6
	2	9	27	1.318	0.219	16.6	0.293	22.2
Total Hydrogen	1	8	30	3.05	0.811	26.6	0.887	29.1
	2	9	27	1.792	0.208	11.6	0.310	17.3
Oxygen	1	6	18	14.3	5.10	35.7	5.72	40.0
	2	9	27	6.15	1.47	23.4	1.60	26.0

TABLE 11.11 - SAMPLE TUBE AVERAGES - COLD- AND HOT-TRAPPED SODIUM AT 900 F

First Test Period								Second Test Period							
Test Day	Date 1968	Station 2			Station 1			Test Day	Date 1968	Station 2			Station 1		
		Concentrations, ppm			Concentrations, ppm					Concentrations, ppm			Concentrations, ppm		
		Hydride Hydrogen	Hydroxide Hydrogen	Total Hydrogen	Hydride Hydrogen	Hydroxide Hydrogen	Total Hydrogen			Hydride Hydrogen	Hydroxide Hydrogen	Total Hydrogen	Hydride Hydrogen	Hydroxide Hydrogen	Total Hydrogen
16	9-24	0.43	2.03	2.46				88	12-5	0.67	1.40	2.07			
22	9-30	0.8	2.17	2.97				92	12-9	0.83	1.87	2.70			
24	10-2	0.5	1.8	2.30				100	12-17	0.53	1.23	1.76			
29	10-7	1.3	1.7	3.00				106	12-23	0.57	1.37	1.94			
31	10-9	0.67	2.2	2.87				109	12-26	0.60	1.20	1.80			
33	10-11	2.13	1.3	3.43					1969						
36	10-14	0.43	1.43	1.86				120	1-6	0.50	1.20	1.70			
38	10-16	0.57	2.73	3.30				123	1-9	0.40	1.83	2.23			
39	10-17	0.63	2.00	2.63				142	1-28	0.33	1.20	1.53			
43	10-21	1.07	0.93	2.00				147	2-3	0.40	1.50	1.90			
44	10-22	0.883	1.7	2.583	0.867	1.65	2.517	151	2-7	0.47	1.03	1.50	0.567	1.367	1.934
46	10-24	1.00	2.48	3.48	1.29	1.96	3.25	154	2-10	0.47	1.30	1.77			
50	10-28	1.70	2.83	4.53	0.44	1.36	1.80								
57	11-4	1.17	1.33	2.50											
65	11-12	1.13	1.50	2.63											
66	11-13				1.133	1.533	2.666								
71	11-18	1.50	2.60	4.10				Average, $\bar{x}$		0.525	1.375	1.899			
73	11-20	0.80	2.50	3.30				Standard Dev. S		0.134	0.253	0.325			
78	11-25	0.47	2.27	2.74											
Average, $\bar{x}$		0.954	1.97	2.93	0.932	1.63	2.56								
Standard Dev., S		0.461	0.531	0.663	0.32	0.22	0.52								

TABLE 11.12 - HYDROGEN CHEMICAL ANALYSIS DATA-STATION 2

Date\Site	←—HYDRIDE HYDROGEN—→						←—HYDROXIDE HYDROGEN—→						←—TOTAL HYDROGEN—→					
	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
9-18	6.5	1.7	4.4				2.4	2.8	2.9				8.9	4.5	7.3			
9-24	0.5	0.4	0.4				2.0	2.8	2.1				2.5	2.4	2.5			
9-30	0.6	0.9	0.9				2.1	2.2	2.2				2.7	3.1	3.1			
10-2	0.6	0.4	0.5				1.8	1.4	2.2				2.4	1.8	2.7			
10-7	1.9	0.8	1.3				1.3	1.5	2.3				3.2	2.3	3.6			
10-9	0.7	1.0	0.3				2.0	2.1	2.5				2.7	3.1	2.8			
10-11	2.9	1.0	2.5				1.9	0.9	1.1				4.8	1.9	3.6			
10-14	0.5	0.5	0.3				1.3	1.5	1.5				1.8	2.0	1.8			
10-16	0.3	0.6	0.8				3.2	2.8	2.2				3.5	3.4	3.0			
10-17	0.7	0.6	0.6				1.9	1.7	2.4				2.6	2.3	3.0			
10-21	1.2	0.6	1.4				1.0	0.9	0.9				2.2	1.5	2.3			
10-22	1.3	1.1	0.6	0.6	0.8	0.9	2.5	1.1	1.5	1.6	1.3	2.2	3.8	2.2	2.1	2.2	2.1	3.1
10-24	1.0	1.5	0.5	1.0	0.9	1.1	2.3	2.4	2.6	3.0	1.9	2.7	3.3	3.9	3.1	4.0	2.8	3.8
10-28	2.0	1.0	2.1				2.8	2.9	2.6				4.8	3.9	4.7			
11-4	1.5	1.0	1.0				1.1	1.8	1.1				2.6	2.8	2.1			
11-12	0.9	1.1	1.4				2.1	1.2	1.2				3.0	2.3	2.6			
11-18	2.2	0.9	1.4				2.7	2.6	2.5				4.9	3.5	3.9			
11-20	1.1	0.6	0.7				2.7	2.6	2.2				3.8	3.2	2.9			
11-25	0.7	0.4	0.3				2.5	2.0	2.3				3.2	2.4	2.6			
11-29	2.2	1.1	2.6				1.7	1.5	3.1				3.9	2.6	5.7			
12-2	1.6	1.0	2.2				1.8	1.5	2.3				3.4	2.5	4.5			
12-5	0.9	0.5	0.6				1.6	1.2	1.4				2.5	1.7	2.0			
12-9	1.1	0.7	0.7				2.3	1.5	1.8				3.4	2.2	2.5			
12-12	2.2	1.8	2.0				1.9	1.5	1.6				4.1	3.3	3.6			
12-17	0.4	0.5	0.7				1.8	0.9	1.0				2.2	1.4	1.7			
12-23	0.6	0.5	0.6				1.5	1.3	1.3				2.1	1.8	1.9			
12-26	0.8	0.6	0.4				1.2	1.3	1.1				2.0	1.9	1.5			
1-6	0.8	0.3	0.4				1.4	0.8	1.4				2.2	1.1	1.8			
1-9	0.4	0.4	0.4				2.1	1.8	1.6				2.5	2.2	2.0			
1-28	0.3	0.3	0.4				1.1	1.2	1.3				1.4	1.5	1.7			
2-3	0.5	0.3	0.4				1.5	1.5	1.5				2.0	1.8	1.9			
2-7	0.5	0.5	0.4				1.2	0.9	1.0				1.7	1.4	1.4			
2-10	0.6	0.4	0.4				1.2	1.2	1.5				1.8	1.6	1.9			

TABLE 11.13 - HYDROGEN CHEMICAL ANALYSIS DATA -
STATION 1

Date \ Site	Hydride Hydrogen						
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
10-22	1.0	0.9	0.6	0.9	0.9	0.9	
10-24	1.3	2.4	0.8	0.9	1.0	0.6	2.0
10-28	0.4	0.5	0.4	0.3	0.6		
11-12	1.6	0.8	1.0				
2-7	0.7	0.4	0.6				
Date \ Site	Hydroxide Hydrogen						
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
10-22	1.9	1.4	1.8	0.9	1.9	2.0	
10-24	1.8	2.8	1.2	2.1	2.1	1.2	2.5
10-28	1.6	1.7	0.9	1.1	1.5		
11-12	1.8	1.4	1.4				
2-7	1.8	1.1	1.2				
Date \ Site	Total Hydrogen						
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
10-22	2.9	2.3	2.4	1.8	2.8	2.9	
10-24	3.1	5.2	2.0	3.0	3.1	1.8	4.5
10-28	2.0	2.2	1.3	1.4	2.1		
11-12	3.4	2.2	2.4				
2-7	2.5	1.5	1.8				

The Station 2 chemical analyses data are shown in Figures 11.12 through 11.17 with Table 11.10 presenting the compilation of the statistical data. The Station 1 chemical analyses data are shown on Figures 11.18, 11.19, and 11.20.

In analyzing the data, it is necessary to make an assumption concerning the validity of the test data using a 3-site test specimen for chemical analysis. The assumption is that the average of the 3 site analyses is the true average of the hydrogen level in the sample tube. The comparisons that are to be made are tube-to-tube and site-to-site. The tube-to-tube evaluation is to establish sodium chemistry stability, and the site-to-site evaluation is to establish the segregation of hydrogen in the sample during cooling.

For the statistical analysis for tube-to-tube and site-to-site variations, the F-test will be used, i.e., to test the null hypothesis that the K population means (K = sample tube means) are all equal, two estimates of S^2 will be compared, one based on the variation among the sample means and one based on the variation within the samples. Let S_W^2 and S_B^2 be the independent estimate of S^2 for within-the-sample and between-the-sample variations; then it follows that

$$F = \frac{\hat{S}_B^2}{\hat{S}_W^2}$$

where $\hat{S}_B^2$ is the estimate of S_B^2 and $\hat{S}_W^2$ is the estimate of S_W^2 , and is a value of a random variable having the F distribution with K - 1 and K(n - 1) degrees of freedom, where K is the number of sodium samples and n is the number of individual chemical analyses.

Since the between-sample variance S_B^2 can be expected to exceed the within-sample variance S_W^2 when the null hypothesis is false, the null hypothesis will be rejected if F exceeds F_0 , where F_0 is obtained from standard tables.

Tube-to-Tube Comparisons - Figures 11.12 through 11.20 show all of the test data from both sample stations and both test groups. Each line on the figures represents a sodium tube analysis, and these figures show the data scatter, particularly in Figure 11.13, the hydroxide hydrogen data from Station 2 in the first test period.

For the first test period hydroxide hydrogen data (Table 11.10), relative standard deviations of 35.6% for the individual chemical analyses and 33.1% for the tube average indicate that the data are random and that there is no improvement by averaging the data for each tube. However, the hydride hydrogen is better expressed by tube average than by the average of the individual chemical analyses, as shown by the lower

relative standard deviation of the former (27.8%) than the latter (39.9%). The four tubes analyzed from Station 1 (Figures 11.18, 11.19, and 11.20) reflect a similar pattern but with a lower relative standard deviation ($S/\bar{x}$).

The second grouping of test data is shown on Figures 11.15, 11.16, and 11.17. These data indicate that after the hot trap operation the loop hydroxide and hydride hydrogen concentrations and the relative standard deviations ($S/\bar{x}$) were lower (refer to Table 11.10). The single tube analysis from Station 1 is shown as a dotted line on the graphs and the tube average is within 1S of Station 2 data.

Site-to-Site Comparisons - Most of the chemistry data are in the form of a 3-site analysis. On a few tubes, sites 5, 6, and 7 were analyzed for hydrogen. All statistical analyses used the 3-site format, so that for all tube data of more than 3 sites, the sites were grouped to give a 3-site format, e.g.,

10-28-68	1	2	3	4	5	6
NaH, ppm	1.0	1.5	0.5	1.0	0.9	1.1

Data Used for

Analysis	<u>1.25</u>	<u>0.75</u>	<u>1.0</u>
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The data for hydride hydrogen is shown on Figures 11.12, 11.15, and 11.18. The statistical F test indicates that segregation does exist in the sodium sample tubes shown in Figures 11.12 and 11.18; i.e., that the site-to-site variation is significantly different than would be expected from the scatter of the individual analyses. For the second test period data at low hydride hydrogen levels (shown in Figure 11.15) the F test does not confirm that the site-to-site variation is more than would be expected from the random variation of the individual analyses. However, the variation of the site-to-site averages in Figure 11.15 shows the same general trend as the data in Figure 11.12 and 11.18, although more data would be needed to determine whether this trend is real. It is, therefore, concluded that hydride hydrogen does segregate in the sample tube during cooling at high hydride-hydrogen levels, and also appears to segregate at low hydride hydrogen levels, although the latter has not been confirmed at the 95% confidence level.

The data for hydroxide hydrogen (Figures 11.13, 11.16, and 11.19) do not show a definite pattern of site-to-site variations. No definite trend is detectable in the data shown in Figure 11.13, and only a limited trend is detectable in Figures 11.16 and 11.19. A possible reason may be that the segregation has to take place above the melting temperature of NaOH (608 F), and the 30 minutes required to lower the sodium temperature from 900 to 608 F may not be long enough for a reproducible

pattern of segregation to take place. Additional studies should be made, since the data scatter makes this study inconclusive.

If the two groups of data in Table 11.11 are combined, as the test was originally designed, then the combined test data are as follows:

	<u>H, ppm</u>	<u>OH, ppm</u>	<u>Total H, ppm</u>
Overall Average, $\bar{x}$	0.793	1.742	2.535
Overall Standard Deviation, S	0.434	0.529	0.759

Conclusions - There is segregation of hydride hydrogen concentrations (about 1 ppm). There may be some segregation at low hydrogen levels (about 0.5 ppm hydride hydrogen), but this requires additional confirmation.

No definite trend on the segregation effect of hydroxide hydrogen was detectable above that amount which could be attributed to the data scatter.

The zirconium hot-trapping of the sodium at 1000 F had an effect on the sodium so that cold-trapping could lower the overall hydrogen level from about 3 ppm to below 2 ppm and stabilize the sodium chemistry.

Removing sodium from the sodium technology loop by the sample tube method is a reliable sampling method for hydrogen at the hydrogen levels studied. It appears that both sample stations are reliable, but more analyses should be made from the samples taken from Station 1 to confirm this. Table 11.14 shows the data reproducibility and includes any effects of variations in loop chemistry, sampling, and analysis.

c. Flush Time and Atmospheric Purity Study (Job 06)

A. J. Friedland
N. N. Mueller

(1) Flush Time Study

The sample sodium from the technology loop bulk sodium for chemical analysis initially enters a bypass freeze tube. There are two horizontal bypass freeze tubes about 9 inches apart enclosed within a glove box provided with continuously purified argon. These two tubes are referred to as Station 1 (outlet) and Station 2 (inlet). The sample tube used on Station 1 is about 27.75 inches long and the one used at Station 2 is about 35.5 inches long. The procedures for taking the samples from these two stations have been established when sampling 600 F loop sodium. The results of this study, reported in Reference 10, indicate that

TABLE 11.14 - DATA FROM HYDROGEN
SAMPLING RELIABILITY STUDIES

	<u>Average, ppm</u>	<u>Standard Deviation, ppm</u>
<u>First Test Period</u>		
Station 1 (18 Tests)		
Hydride Hydrogen	0.95	0.46
Hydroxide Hydrogen	1.97	0.53
Total	2.93	0.66
Station 2 (4 Tests)		
Hydride Hydrogen	0.93	0.32
Hydroxide Hydrogen	1.63	0.22
Total	2.56	0.52
<u>Second Test Period</u>		
Station 2 (11 Tests)		
Hydride Hydrogen	0.53	0.13
Hydroxide Hydrogen	1.38	0.25
Total	1.90	0.33

NOTE: No overall variation of test data with time was observed during each test period.

No variation of test data was observed with the loop temperature changes which took place during the second test period.

- There is no difference in daily results (no sample-to-sample variation) for the sample tube averages taken after the 4-hour flush time. At the test conditions (about 11 ppm oxygen level) there is no difference in the results when sampling from Station 1 and Station 2.
- At both stations, samples taken at 1-hour flush times showed higher oxygen concentration than samples at either the 4-hour or 2-1/2-hour flush times.
- At Station 1 there is no difference in sample tube averages when using a 4-hour or 2-1/2-hour flush time; however, the Station 2 sample taken at 2-1/2-hour flush time showed higher oxygen concentration than the 4-hour flush time sample.

- Therefore, a 4-hour flush time is required at both sample stations to obtain consistent results.

Thus the results of the previous study indicate the significance of flush time in obtaining consistent sodium samples from the technology loop. In order to gain similar experience regarding the significance of flush time when sampling 900 F cold-trapped sodium for oxygen, a test program has been conducted with the objective of studying the flush time required for sample tubes when sampling 900 F cold-trapped sodium (refer to Figure 11.1).

Description of the Test - That portion of the combined test program designed to meet the objective of the study of minimum flush time required for sampling 900 F cold-trapped sodium for oxygen is shown in Figure 11.21. On day 37 of loop operation a cold-trap operation was initiated and continued overnight. The cold trap was isolated after about 20 hours of operation and then a period of 10 days was allowed for the 900 F loop sodium to reach steady state.

To meet the objective of the test program, the following test was performed: Flush time was varied from 15 minutes to three hours; five sets of sample tubes were taken for 1/4-hour, 3-hour, 1/2-hour, 2-hour, and 1-hour flush time on days 50, 51, 52, 53, and 58 of operation, respectively, with 0.5 gpm flow of loop sodium at 900 F.

Test Results and Discussions - During the present test program, an overall cut in the total number of analyses originally scheduled to meet the test objective was necessary because of very limited manpower available for chemical analyses. Therefore, from the sets of samples taken from Station 1 and Station 2 for the flush time study, only the samples taken from Station 2 (inlet) were analyzed. The selection of the samples from Station 2 for the flush time study for 900 F over Station 1 was based on past experience, where the results of the flush time study for 600 F loop sodium indicated that the effects of flush time were more pronounced at Station 2 than at Station 1. At Station 1 there was no difference in sample tube averages when using a 4-hour or 2-1/2-hour flush time; while for Station 2, a sample taken at 2-1/2-hour flush time showed higher oxygen concentration.

Each of the sodium samples taken at Station 1 for the flush time study was divided into six equal segments in the chemistry laboratory, and each segment was analyzed for oxygen by the mercury amalgamation method. These analyses are reported in Table 11.15

The chemical analyses for the samples taken at 1/2-hour and 2-hour flush times showed significantly higher oxygen concentrations (21 ppm and 22.3 ppm, respectively) than for the sample taken at 4-hour flush time (12.7 ppm). The samples taken for 1/4-hour, 1-hour, and 3-hour flush times showed oxygen levels (10.3 ppm, 9.2 ppm, and 12.8 ppm,

TABLE 11.15 - RESULTS OF SAMPLING RELIABILITY FOR OXYGEN AT 900 F LOOP SODIUM

Sample Tube Number	Date of Sample	Sample Tube Flush Time, hr	Plugging Meter Temp, F	Oxygen Concentration, ppm							Nonhydroxide Hydrogen Concentration, ppm						Hydroxide Hydrogen Concentration, ppm						Total Hydrogen
				Site							Site						Site						
				1	2	3	4	5	6	Average	1	2	3	4	5	Average	1	2	3	4	5	Average	
1250 (x)	10-28-68	4	594								0.4	0.5	0.4	0.3	0.6	0.44	1.6	1.7	0.9	1.1	1.5	1.36	1.80
1251 (y)	10-28-68	4	594	15(a)	11(a)	12(a)				12.67	2.0	1.0	2.1			1.70	2.8	2.9	2.6			2.43	4.13
1253 (y)	10-29-68	1/4	600	13	9	6	10	10	14	10.33													
1257 (y)	10-31-68	1/2	600	32	23	19	17	16	19	21.00													
1263 (y)	11-6-68	1	604	9	10	9	8	7	12	9.17													
1259 (y)	11-1-68	2	604	44	21	17	13	16	23	22.33													
1255 (y)	10-30-68	3	600	15	13	13	12	12	12	12.83													
1261 (y)	11-4-68	4	607	24(a)	16(a)	16(a)				18.67	1.5	1.0	1.0			1.17	1.1	1.8	1.1			1.33	2.50

(a) Only the three sites were analyzed for oxygen

(x) Outlet - Sample Station 1

(y) Inlet - Sample Station 2

respectively) similar to those for the 4-hour flush time. Therefore, it is felt that the 1/2-hour and 2-hour flush time effects alone are not responsible for the significant difference in the oxygen level as seen by the chemical analyses, and some other effect may have occurred. Investigations conducted to check for a possible leakage in the analytical glove box during the analysis of these samples with high oxygen value showed no indications of any such contamination inside the glove box.

Similar high levels in oxygen concentration were again measured by chemistry during a sample tube temperature profile study. These spikes in the oxygen level are unexplained, since there were no deliberate changes made in the loop chemistry in terms of monoxide injections or cold traps, hot traps, etc., during this period of operation (see Figure 11.21).

Conclusion - A complete evaluation of the test data to study the minimum flush time required when sampling at 900 F is not possible because of the unexplained observations of significantly high oxygen levels in sodium when sampled for 1/2- and 2-hour flush times. The oxygen concentrations measured for the 1/4- and 1-hour flush times were somewhat lower than those for the 3- and 4-hour flush times, but more data would be needed to confirm this trend.

(2) Atmospheric Purity Study

M. K. Deora

Sodium sample stations initially were not enclosed in a glove box with a high purity inert atmosphere. The chemical analyses of sodium samples during this period were very erratic, and it was believed that this was the result of sample contamination due to air inleakage during the sampling operation. To prevent this problem, the sampling stations were completely enclosed within a glove box that was provided with continuously purified argon and the entire operation of sampling was conducted inside the glove box. Preliminary results showed that the incorporation of the glove box considerably reduced the scatter in the chemical analyses; therefore, the current sampling procedure uses the high-purity glove box atmosphere during the entire operation of sampling at APDA, for consistent results.

Although the present technique of sampling ¹⁰ which incorporates the glove box is adequate for obtaining consistent chemistry results, the actual operating procedure is difficult and time-consuming. For sampling radioactive sodium from such facilities as the FFTF, EBR-II, and LMFBR reactors, it was decided to investigate an alternative to the high-purity glove box for consistent sodium samples.

The objective of this test program is to study whether or not end-crimping of sample tubes isolated at 900 F is an effective means of eliminating the necessity for a high-purity environment when sampling for oxygen.

Description of the Test - The portion of the multitest program to meet the objective of the study of end-crimping of sample tubes isolated at 900 F as an effective means of eliminating the necessity for a high purity glove box environment when sampling for oxygen is shown in Figure 11.21.

To meet the objective of this test program, the following test was performed: On day 60 and 63 of loop operation (refer to Figure 11.1), two sets of nickel sample tubes were prepared for this study because it is easier and safer to crimp a nickel tube than a stainless steel tube. The tubes were end-crimped, with two crimps at each end of the sample tube.

It would have been desirable to eliminate the high-purity glove box during the entire operation of sampling; however, because of past experience with sample tubes leaking at the fittings during sampling, which could endanger the safety of an operator, an argon atmosphere was maintained in the sample glove box during installation of the sample tube, four-hour flush time, and end-crimping. After the end-crimping was completed, the glove box was opened and filled with air. The sample tubes were then allowed to cool down in air. After cool-down (3 to 4 hours) the sample tubes were removed from the sample station and exposed to air overnight with no end caps. The tubes were then capped and transferred to the chemistry glove box for oxygen analysis. Chemical analyses for oxygen were performed at six sites on each one of the two tubes at Station 2. The results of the chemical analyses are reported in Table 11.16.

Test Results and Discussion - Because of the small number of samples taken, no statistical analysis of the results was performed; however, from the data (refer to Table 11.16) it can be observed that there is no significant site-to-site variation and that the two sample averages are not significantly different. Furthermore, the end-crimped sample tube averages are not significantly different from that for a sodium sample taken in an argon atmosphere, for this level of oxygen (about 11 ppm). The results indicate that there was no contamination, because if there were contamination, the sodium site near the end would be higher in oxygen concentration.

Conclusions and Recommendations - The results of this preliminary study indicate that hot crimping of the sample tubes was effective in preventing any contamination due to oxygen when the sample tubes were exposed to air during the cool-down period, removal operation, and overnight exposure with no end caps, before they were transferred to the chemistry laboratory glove box for analysis.

From these preliminary results, there is a strong possibility of eliminating the high-purity glove box completely for the

TABLE 11.16 - RESULTS OF CHEMICAL ANALYSIS FOR THE
ATMOSPHERIC PURITY STUDY AT 900 F LOOP SODIUM

Sample Tube No. (b)	Date	Plugging Temp, F	Oxygen Concentration, (a) ppm						
			Sample Station 2						
			Site 1	Site 2	Site 3	Site 4	Site 5	Site 6	Average
1251	10-28-68	594	15(c)	11(c)	12(c)				12.67(d)
1269	11-8-68	604	11	(e)	10	11	11	11	10.80
1271	11-11-68	606	12	12	12	11	11	12	11.67

(a) Total alkalinity calculated as oxygen in sodium monoxide

(b) Nickel sample tube

(c) Only three site analyses were performed

(d) With argon atmosphere maintained in glove box

(e) Sample was lost

entire sampling operation; therefore, it is recommended that more effort be directed toward this study.

(3) Temperature Profile Study

A sodium sampling and analysis reliability study for oxygen using the mercury amalgamation procedure has been completed for the 11 ppm, 47 ppm, and 76 ppm oxygen levels. During this study the samples were analyzed in the chemistry laboratory by dividing each sample into five or six equal parts along its length. These segments are designated as sites, and each individual chemical analysis for a site gives the oxygen concentration at that particular site. The results of this study indicate that a statistically significant site-to-site variation in oxygen concentration existed at all three oxygen levels at the outlet (Station 1) and that at the inlet (Station 2) a site-to-site variation occurred only at the 76 ppm oxygen level. This site-to-site variation along the length of the sample tube at the two sample stations is thought to be a segregation effect brought on by nonuniform cooling of the sample tubes. Therefore, a study was conducted in order to better understand the significance of the segregation effect along the sample tubes and to determine whether this effect is the result of non-uniform cooling along the length of the sample tube.

The objective of this test program is to study the mechanism of segregation by determining the temperature profile of a Station 1 and a Station 2 sample tube during cooling following flow isolation at 900 F.

Description of the Test - That portion of the multitest program dealing with the mechanism of segregation by determining the temperature profile of an outlet and inlet sample station tube during cooling following flow isolation at 900 F is shown in Figure 11.21.

To meet the objective of this test program the following test was performed: The Station 1 sample tube of the sample set taken on day 64 and the Station 2 sample tube of the sample set taken on day 65 were employed to make the desired temperature measurements (refer to Figure 11.1). Eight thermocouples were placed on the sampling lines, six thermocouples equally spaced on the sample tube, and one on each of the elbows adjacent to the sample tube (as shown in Figure 11.22). The temperature of the sample tube was monitored after isolating the sodium flow at 900 F.

Test Results and Discussions - The results of the temperature profile study of Station 1 and Station 2 during the cooling period following sodium flow isolation at 900 F are shown in Figures 11.23 and 11.24, respectively. There is a remarkable difference between the temperature profiles of the tubes at the two stations, and the temperature differences are more pronounced at Station 2 than at Station 1. At Station 1 thermocouple 5, near the center of the sample tube, recorded a higher

temperature than the other thermocouples during the first 30 minutes. The maximum difference in temperature, about 60 F, occurs between the center (thermocouple 5) and the two ends of the sample tube (thermocouples 2 and 7), with the center at the higher temperature. This is followed by a rapid drop in temperature near the center of the tube, and the thermocouple near the center records a lower temperature than any of the other thermocouples along the sample tube. The maximum difference in temperature between the two elbows (thermocouples 1 and 8) and the center (thermocouple 5) of the sample tube is about 75 F, with the elbows at the higher temperature.

The sodium started to freeze near the center in the sample tube about 3-1/2 hours after the sodium flow through the tube was isolated, and froze completely within five hours.

The two thermocouples (1 and 8) on the elbows at Station 2 record higher temperatures than the remaining thermocouples along the length of the sample tube and stay this way during the entire period of cooling. The maximum difference in the temperature occurring between the two elbows (thermocouples 1 and 8) and near the center (thermocouples 5 and 6) of the sample tube is about 225 F, with two elbows at the higher temperature. The sodium near the center would start to freeze in about two hours after the sodium flow through the tube had been isolated and would freeze completely within four hours.

This preliminary study thus indicated that the rate of cooling between the two sample tubes at Station 1 and Station 2 is significantly different. In order to further understand the phenomenon of cooling, particularly at Station 2, another sodium sample was taken at Station 2. The natural circulation of sodium was promoted by raising the elevation of the sample tube section relative to the elbows by 3/4 inch to 1 inch. The sodium technology loop was operating at 600 F during this portion of the test; the results of the recorded temperature during the cooling period are shown in Figure 11.25. The new temperature profile along the sample line is significantly different from the previous one shown in Figure 11.24, and a maximum temperature difference between the thermocouples at the two elbows (1 and 8) and near the center of sample tube (5 and 6) is about 100 F, with the two elbows at the higher temperature. The sodium near the center of the tube started to freeze about two hours after isolating the sodium flow through the tube, and froze completely in three hours. Furthermore, the pattern of the temperature profile is very similar to the one previously recorded for Station 1 (see Figure 11.23).

The results of the chemical analyses of the sample tubes used during the temperature profile study are shown in Figure 11.26. These results indicate a significant variation in oxygen concentration from site to site for the first two samples obtained at 900 F. The variation in oxygen concentration is more pronounced at Station 2 than at Station 1, with

tube averages of 25.0 and 24.4 ppm oxygen, respectively. Results of the chemical analyses for 600 F sodium show a uniform oxygen level of about 5 ppm at the three sites of the sample tube analyzed.

Interpretation of Test Results - The results of the temperature study at both stations indicate that the rate of cooling along the length of the sample tube is not uniform. The corresponding results of the chemical analyses for oxygen concentration along the length of the two sample tubes used during the temperature profile study have a significant site-to-site variation in oxygen concentration (see Figure 11.26). Furthermore, the effect of nonuniform cooling is more pronounced at Station 2 than at Station 1, suggesting that if the site-to-site variation effect is due to a nonuniform cooling rate, then there should be a more pronounced site-to-site variation effect at Station 2 than at Station 1. However, this is not supported by the past experience at APDA, because all the results of the past studies^{10,11,12} indicate that the site-to-site variation in oxygen concentration is more pronounced at Station 1 than at Station 2. Although the results of the chemical analyses of the two sample tubes used during this study do indicate a site-to-site variation effect which is more pronounced at Station 2 than at Station 1, the oxygen concentration measured by chemistry for these two sodium samples is unusually high (about 24 ppm) and is unexplainable because the normal operating oxygen level during this period was about 12 ppm (indicated by other samples taken prior to and after these samples during steady operation, as shown in Figure 11.21).

Another sample taken at Station 2 during the temperature profile study at 600 F failed to provide any additional information because the level of oxygen in the technology loop sodium was low and the chemical analyses indicated 5 ppm oxygen at the three sites of the sample tube analysed for oxygen. Also, the temperature profile was significantly different from the previous one at 900 F at the same station.

Conclusions and Recommendations - The preliminary study seems to support the opinion that the site-to-site variation in oxygen concentration is a segregation effect caused by nonuniform cooling rate along the length of the sample tube. It is concluded that more studies should be conducted to confirm these preliminary results and to further understand why the segregation effect has been more pronounced at Station 1 than at Station 2.

2. ASTM Round Robin on Oxygen
in Sodium (Task 73)

E. F. Hill
S. A. Meacham

The objectives of this task are

- To prepare samples of sodium containing known quantities of oxygen as specified by the ASTM subcommittee

- To distribute these samples to five participating laboratories
- To collect and evaluate the results obtained by the five laboratories
- To report the results and the committee's conclusions.

The first two objectives were completed in FY-68.

a. Reduction of Data and Report (Job 05)

The scope of this job is defined as the reduction, evaluation, and reporting of the data submitted by the five participating laboratories.

The final draft has been sent to the committee members and the AEC-RDT office for final comments and approval before distribution.

E. Loop Operations

1. Supporting Chemistry Services (Task 51)

- a. Uranium Gettering Technique (Job 04) E. F. Hill
 S. A. Meacham

The objectives of this program are

- To briefly review the most pertinent technical literature on the gettering technique for oxygen in sodium.
- To evaluate the proposed Brookhaven National Laboratory (BNL) technique for oxygen in sodium using uranium as a getter metal in the APDA sodium technology loop.

Twelve uranium specimens which had been exposed to static sodium for 1 hour at 500°C were obtained from BNL. The specimens, designated A₁, B₂, C₃, E₂, F₃, and G₁, were cleaned by electropolishing and weighed prior to experimentation. All the specimens were 0.508 cm thick by 1 cm wide by 12.5 cm long with a net surface area* of 26.3098 cm², with the exception of Specimen C₃, which was 0.508 cm thick by 1 cm wide by 12.35 cm long and had a net surface area of 25.9946 cm².

Two experiments were conducted at two oxygen levels by exposing the specimens three at a time to the sodium of the sodium technology

* Each specimen had two 2 mm diameter holes drilled through, and a correction was made for these in calculating the surface area of the specimens.

loop using a specimen holder of 3/4-inch stainless steel tubing. The experimental conditions and test results are shown in Table 11.17.

TABLE 11.17 - TEST CONDITIONS FOR URANIUM
GETTERING EXPERIMENTS

	<u>Test 1</u>	<u>Test 2</u>
Sodium Conditions		
Flow through Sample Holder, gpm	1/2	1/2
Temperature, °C (F)	485 (905)	485 (905)
Oxygen Level, ppm	6.7	9.5
Exposure Time of Specimens, hr	2	2
Pretest Weight of Specimens, g	A ₁ 11.3897 B ₂ 11.3588 C ₃ 11.4429	E ₂ 11.7364 F ₃ 11.0974 G ₁ 11.1063
Weight Gain of Specimens, g	A ₁ 0.0050 B ₂ 0.0058 C ₃ 0.0045	E ₂ 0.0112 F ₃ 0.0119 G ₁ 0.0128
Average Weight Gain, g/hr	97.3	194.93

* Calculated value as Na₂O, as determined by the mercury amalgamation method.

The average results from the loop tests fall well within the range of the BNL static pot tests.¹³

III. NEXT REPORT PERIOD ACTIVITIES

A. Impurity Monitoring Devices

1. Rhometer (Task 20)

The Rhometer automatic temperature compensator is scheduled to operate on line in the sodium technology loop during the next report period and is expected to indicate change in impurities that occur in the sodium due to chemistry changes.

2. Oxygen Meter (Task 27)

a. Behavior in High Temperature Loop Sodium (Job 05)

No further effort is planned for this job.

3. Hydrogen Detector (Task 21)

The temperature and flow controls will be tested and adjusted for minimum variability from the setpoint. The injection-removal system will be tested for operability and as a preliminary fix on system parameters. The calibration run is scheduled to begin near the end of the next quarter (Job 08).

The data reduction (Job 09) will be completed.

4. Plugging Meter (Task 23)

No further effort is planned for this task.

B. Sodium Purification Processes

1. Centrifuging (Task 12)

The draft topical report for this task will be completed and distributed to RDT for review and comment.

C. Physical and Chemical Behavior of Sodium Impurities

1. Hydrogen Solubility (Task 31)

Preparation of the topical report will begin and thermodynamic studies will be incorporated into the topical report.

2. Cover Gas Impurities (Task 33)

A further refinement of the optimum temperature will be attempted and verification of the residual error involved will be accomplished by passing a stream of precalibrated gas through the sampling system. The gas is argon containing only hydrogen as an impurity in the range 100 and 700 ppm. It is planned to fit these tests into the already scheduled test programs (Job 02).

The scope of job (03) will be expanded to cover FY-70. Data evaluation will be continued.

D. Sodium Sampling and Analysis

1. Sampling Reliability (Task 70)

The final draft of the topical report on oxygen sampling reliability will be submitted to the Commission for comment.

2. ASTM Round Robin on Oxygen in Sodium (Task 73)

Completion of the final report, subject to AEC-RDT and Committee approval.

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2. Meyers, J. E., and Hunter, R. A., "Impurity Monitoring in a Large Sodium Test Loop," presented at the International Conference on Sodium Technology and Large Fast Reactor Design, Argonne National Laboratory, November 7-9, 1968.
3. Minushkin, B., "Electrochemical Oxygen Meter Operating Manual," UNC-5113, August 31, 1965.
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5. Wilks, S. S., Elementary Statistical Analysis, Princeton University Press, 1951.
6. "Sodium Components Development Program Mass Transfer Investigation in Liquid Metal System"- Quarterly Progress Report No. 7, September-November, 1968, GEAP-5719, General Electric Company, Sunnyvale, California.
7. Simpson, J. L., Dutinon, D., and Pohel, L. E., "Some Effects of Hydrogen in Sodium Loop," AEC-ANL International Conference on Sodium Technology and Large Fast Reactor Design, Argonne National Laboratory, November 7-9, 1968.
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9. Eichelberger, R. L., "Solubility of Oxygen in Sodium: A Recommended Expression," AI-AEC-12685, November 1, 1968.
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12. "Quarterly Technical Progress Report on AEC-Sponsored Activities, April-June 1968," APDA-228, 1968.
13. "Quarterly Progress Report January-March, 1968," BNL-50111, pp. 41-46, 1968.

FIG. 11.1 CHRONOLOGY OF LOOP CHEMISTRY DURING MULTITEST PROGRAM

112969

FIG. 11.2 RHOMETER AUTOMATIC TEMPERATURE-COMPENSATING SYSTEM

112169

FIG. 11.3 RHOMETER AUTOMATIC TEMPERATURE-COMPENSATING SYSTEM—TOROID TRANSFORMER IN AIR-COOLED ENCLOSURE ON SODIUM TECHNOLOGY LOOP

Digital Voltmeter

Recorder

Function Generator

Isolated 60-cycle
Power Supply

Output Voltage
Controller

112269

**FIG. 11.4 ELECTRONIC COMPONENTS OF THE RHOMETER AUTOMATIC
TEMPERATURE COMPENSATING SYSTEM**

113069

FIG. 11.5 FUNCTION GENERATOR OUTPUT VERSUS SODIUM RESISTIVITY

FIG. 11.6 OXYGEN METER PERFORMANCE IN HIGH-TEMPERATURE LOOP SODIUM

112669

FIG. 11.7 PLUGGING METER RUN 10-1-68

112469

FIG. 11.8 PLUGGING METER RUN 12-6-68

FIG. 11.9a EFFECT OF LOOP SODIUM ON PLUGGING TEMPERATURE

LEGEND

- = Oxygen
- = Hydroxide Hydrogen
- △ = Nonhydroxide Hydrogen
- △ = Total Hydrogen

C.T = Cold Trap
H.T = Hot Trap

↕ = 12.8-Gram Injection of NaOH
(Equivalent to 1 ppm of Hydrogen and 16 ppm of Oxygen)

LOOP SODIUM IMPURITY CONCENTRATION

FIG. 11.9b EFFECT OF LOOP SODIUM ON PLUGGING TEMPERATURE

114569

FIG. 11.10 SAMPLE TUBE SITE AVERAGES — STATION 2

112569

FIG. 11.11 CHEMICAL ANALYSIS SCHEME FOR INLET SAMPLE TUBE

113269

FIG. 11.12 ANALYSIS FOR HYDRIDE HYDROGEN AT STATION 2—FIRST TEST PERIOD

113369

FIG. 11.13 ANALYSIS FOR HYDROXIDE HYDROGEN AT STATION 2—FIRST TEST PERIOD

114269

FIG. 11.14 ANALYSIS FOR TOTAL HYDROGEN AT STATION 2—FIRST TEST PERIOD

112669

FIG. 11.15 ANALYSIS FOR HYDRIDE HYDROGEN AT STATION 2 – SECOND TEST PERIOD

FIG. 11.16 ANALYSIS FOR HYDROXIDE HYDROGEN AT STATION 2 – SECOND TEST PERIOD

114169

113669

FIG. 11.17 ANALYSIS FOR TOTAL HYDROGEN AT STATION 2 – SECOND TEST PERIOD

113969

FIG. 11.18 ANALYSIS FOR HYDRIDE HYDROGEN AT STATION 1 – FIRST TEST PERIOD

113769

FIG. 11.19 ANALYSIS FOR HYDROXIDE HYDROGEN AT STATION 1-FIRST TEST PERIOD

112769

FIG. 11.20 ANALYSIS FOR TOTAL HYDROGEN AT STATION 1-FIRST TEST PERIOD

FIG. 11.21 TIME VARIATION IN OXYGEN CONCENTRATION DURING SAMPLING RELIABILITY STUDY

FIG. 11.22 LOCATION OF THERMOCOUPLES ON SAMPLE TUBE IN TEMPURTE PROFILE STUDY

114069

FIG. 11.23 TEMPERATURE AFTER FLOW ISOLATION AT 900 F — STATION 1

113169

FIG. 11.24 TEMPERATURE PROFILE AFTER FLOW ISOLATION AT 900 F—STATION 2

112069

FIG. 11.25 TEMPERATURE PROFILE AFTER FLOW ISOLATION AT 600 F — STATION 2

113869

FIG. 11.26 OXYGEN CONCENTRATION VERSUS SITE LOCATION

COMPILATION OF FERMI EXPERIENCE

PROJECT AGREEMENT NO. 15

PROJECT ENGINEER: E. C. Kovacic

I. PREFACE

A. Scope

The purpose of the project is to make available to the fast reactor development program the experience that has been gained from the Enrico Fermi Atomic Power Plant project. The scope of effort covers the compilation of noteworthy experience into reports suitable for general use, starting with the evolution of the design and progressing through fabrication, construction, testing, and operation of the Fermi plant. Important components and systems will be treated in individual reports which will include sufficient descriptive and background material to make each a reporting entity.

B. Relationship to Other Projects

This project is related generally to the Commission's program of the development of fast reactor technology.

II. TECHNICAL PROGRESS

The status of the reports that had not been formally published prior to the current quarter is as follows:

A. Compilation of Current EFAPP Experience (Task 19)

R. A. Costello

The reports for the months of November through March, APDA-CFE-27 through APDA-CFE-31, respectively, were issued. Reports for the months of April, May, and June are in various stages of preparation.

EFAPP Primary Sodium System Pumps, APDA-309 (Task 20)

T. P. Ross

This report was published in June.

III. NEXT REPORT PERIOD ACTIVITIES

A. Compilation of Current EFAPP Experience (Task 19)

Reports for the months of April, May, and June will be issued.
Due to termination of funding, no additional reports will be initiated.

FAST FLUX TEST FACILITY
PROJECT AGREEMENT NO. 16

PRINCIPAL INVESTIGATOR: J. B. Nims

I. PREFACE

A. Scope

The broad scope of this program is to provide engineering consulting assistance to Pacific Northwest Laboratory (PNL) in connection with the design of the Fast Flux Test Facility (FFTF). APDA provides this assistance by consulting with PNL personnel on fast reactor and sodium technology, including sodium components, fuels, cladding and structural materials, and instrumentation.

B. Relationship to Other Projects

The study is being conducted in direct support of the AEC program to develop a facility for irradiation of fast reactor fuels and materials.

II. TECHNICAL PROGRESS

J. B. Nims

During the quarter, several discussions were held between PNL and APDA personnel, at PNL request, in order to provide PNL with information concerning the Fermi reactor system. On June 12, P. G. Baumgartel visited APDA and discussed sodium, NaK, and inert cover gas systems with various APDA personnel; and June 13, C. R. Nash and H. D. Lenkersdorfer of PNL discussed systems interface management and the Fermi coordinator system with APDA personnel; and on May 15, E. C. Kovacic of APDA, while on a visit to Richland for other business, discussed sodium and inert gas systems with various PNL personnel.

III. EXPECTED RESULTS

The project agreement has not been renewed for FY-70, and no further work is expected.

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