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Carbon in the Primary Sodium System  
of the Enrico Fermi Atomic Power Plant

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SUMMARY

When APDA conducted the 1000 F high temperature tests on the primary system of the Enrico Fermi Atomic Power Plant, carbonaceous gases and vapors were evolved from the graphite in the reactor plug. The purpose of this report is to summarize the efforts that have been expended to determine the amount of carbon evolved, the methods suitable for removing the carbon from the sodium, the possible problems that could be caused by the carbon, and methods of operational control of the carbon. Based upon the study conducted and reported in this summary report, there should be no further outgassing from the plug due to temperature. Some gas may be evolved each year due to radiation. However, these gases should be mostly hydrogen. Any carbonaceous gases that should be evolved can be readily dissolved into the sodium and removed in the carbon removal system thereby forming no crust. To date, the experiments on methods of removing carbon from sodium are not complete. However, sufficient data are in to indicate that of the possible problems that could be postulated as being caused by the off-gases none appear to be serious. Additional experimental work is under way to provide high-power operating criteria for Fermi. The plugging indicator orifice plate at Fermi has been redesigned to provide orifices of 0.030 inches in diameter to indicate particle sizes that would be of the order of the pore size of the screens in the reactor subassemblies.

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## I. INTRODUCTION

### A. Carbon in the Primary Sodium

During the high temperature test (1000 F), which AEDA conducted on the primary sodium system of the Enrico Fermi Atomic Power Plant, carbonaceous gases and vapors were evolved from the graphite in the reactor plug. Although the graphite is canned, it is vented to the argon cover gas system through small vent holes located in the top of each can. Some of these gases may have been purged out of the primary gas system prior to reacting; however, a sufficient amount of them reacted with the primary sodium to form a crust at the interface between the sodium and the argon cover gas.

Since the original evolution, considerable effort has been expended to determine the amounts of carbon evolved, the methods suitable for removing the carbon from the sodium, the possible problems that could be caused by the carbon, and methods of operational control of carbon. The purpose of this report is to summarize the results of these efforts.

### B. Plug Design

The rotating shield plug, shown in Figures 1, 2, and 3, serves as an integral part of the biological shield and as a part of the subassembly handling equipment. It consists of a 1-1/32-inch-thick stainless steel shell containing layers of shielding materials and insulation. The shielding materials consist of 18 inches of stainless steel, approximately 6 feet of graphite of which about 20% is borated to 1.5 w/o boron as boron carbide, and 2 feet of carbon steel. The plug has a 12-inch-thick carbon steel cover plate that carries the hold-down assembly and the offset handling equipment.

The 18 inches of stainless steel shielding consists of thin plates located at the bottom of the plug, above which are 7 layers of graphite blocks, 2-5/8 inches square by 10-7/16 inches high, canned in carbon steel. The upper layer of cans as well as those adjacent to the hold-down column penetration contains borated graphite. Each layer is supported by a 2-inch-thick carbon steel plate. Spaces between the cans and the penetrations of the plug are filled with cast boron (1 to 1-1/2%) steel; other voids are filled with plain carbon steel. The remaining plug shielding consists of six 2-inch-thick carbon steel plates located above the cans. A 12-inch-thick layer of canned stainless steel wool insulation packed to a density of 20 lb/cu ft is placed between the top carbon steel plate and the 12-inch-thick plug cover plate.

A 3-inch hole is provided through the center of the plug for alignment purposes, and three 5-inch holes are provided for removing the lower guide tubes. All 4 holes will be plugged with shielding material during plant operation.



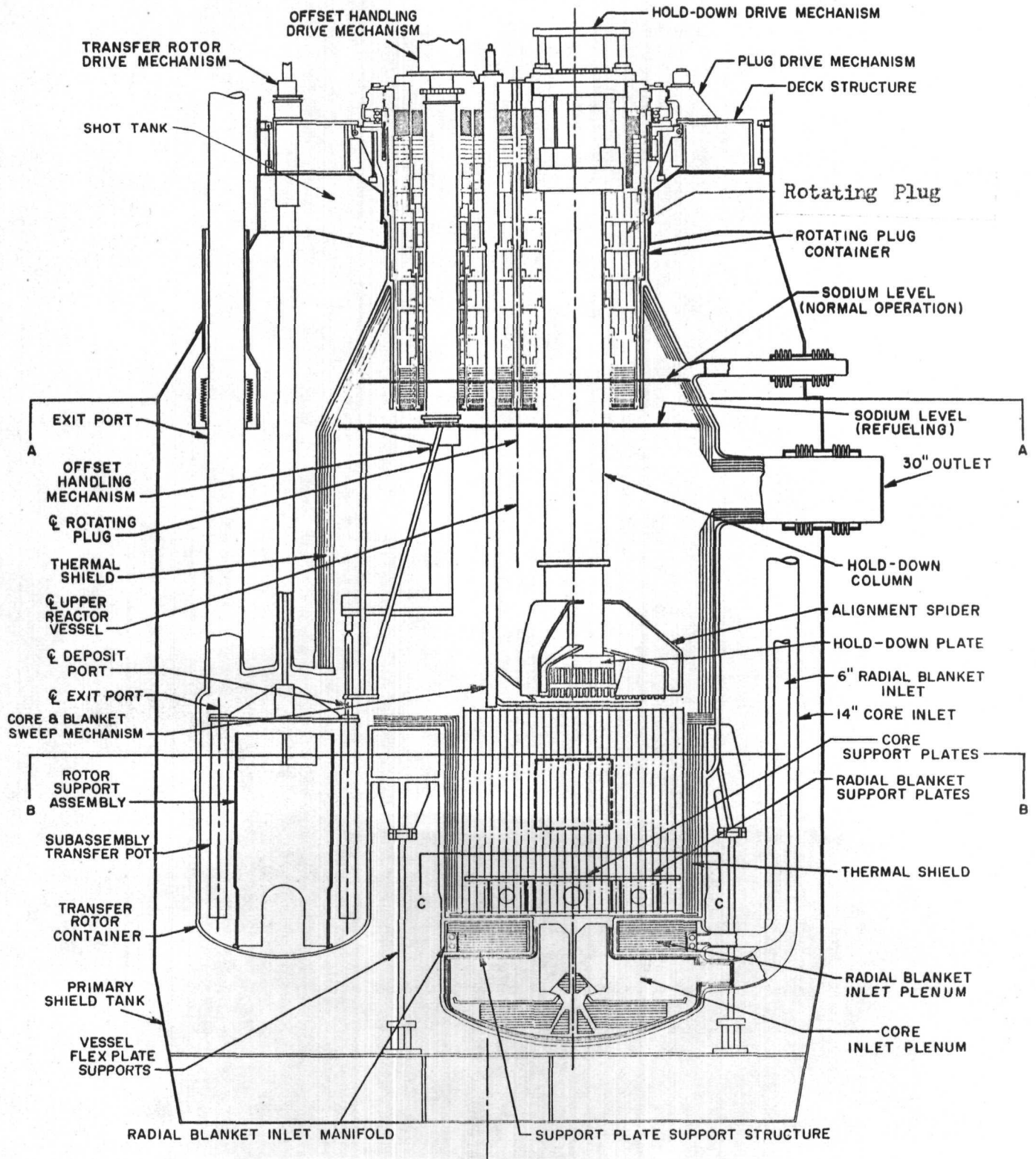


Fig. 1. Cross Section of Reactor Showing Shield Plug in Place

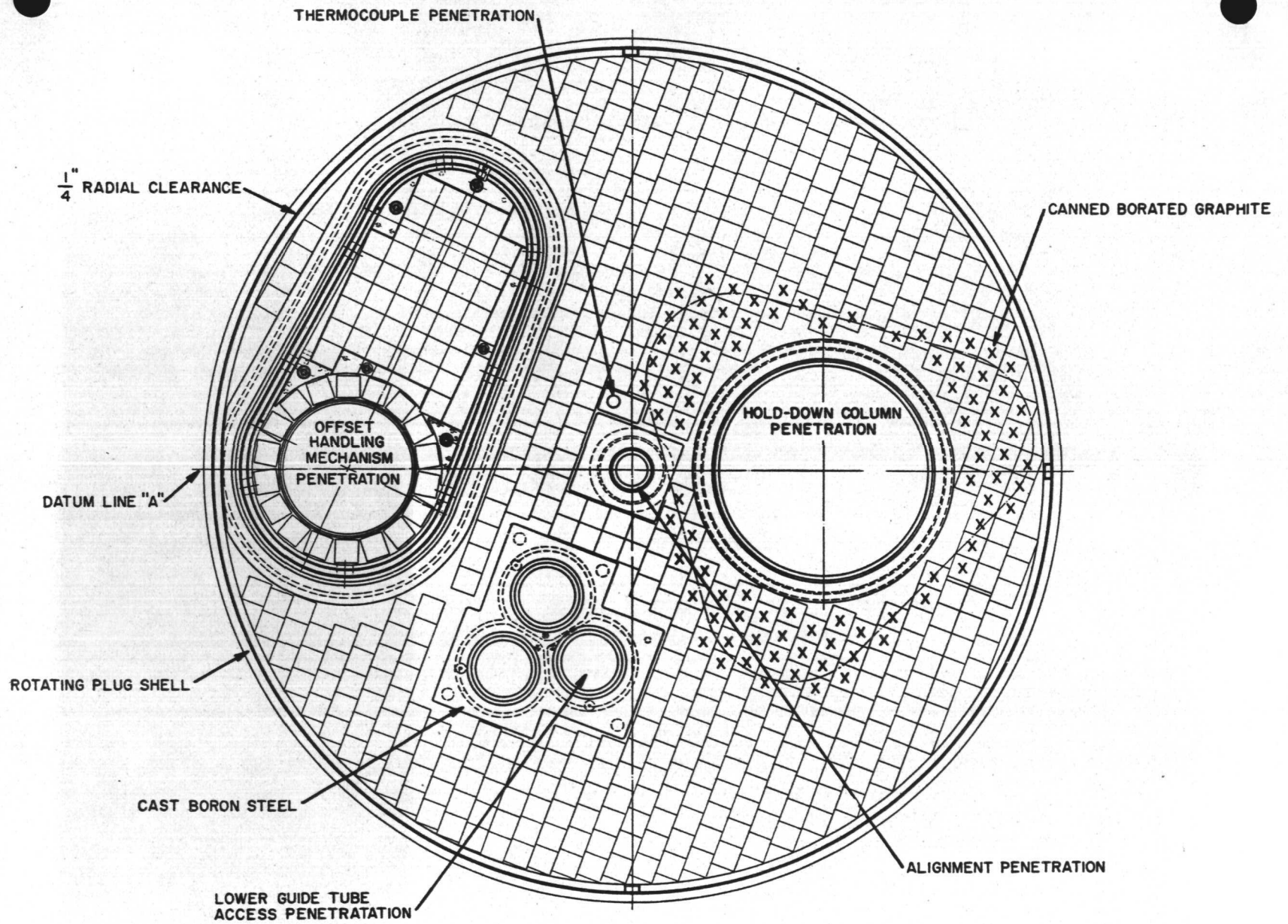


Fig. 2

ROTATING SHIELD PLUG (HORIZONTAL SECTION)

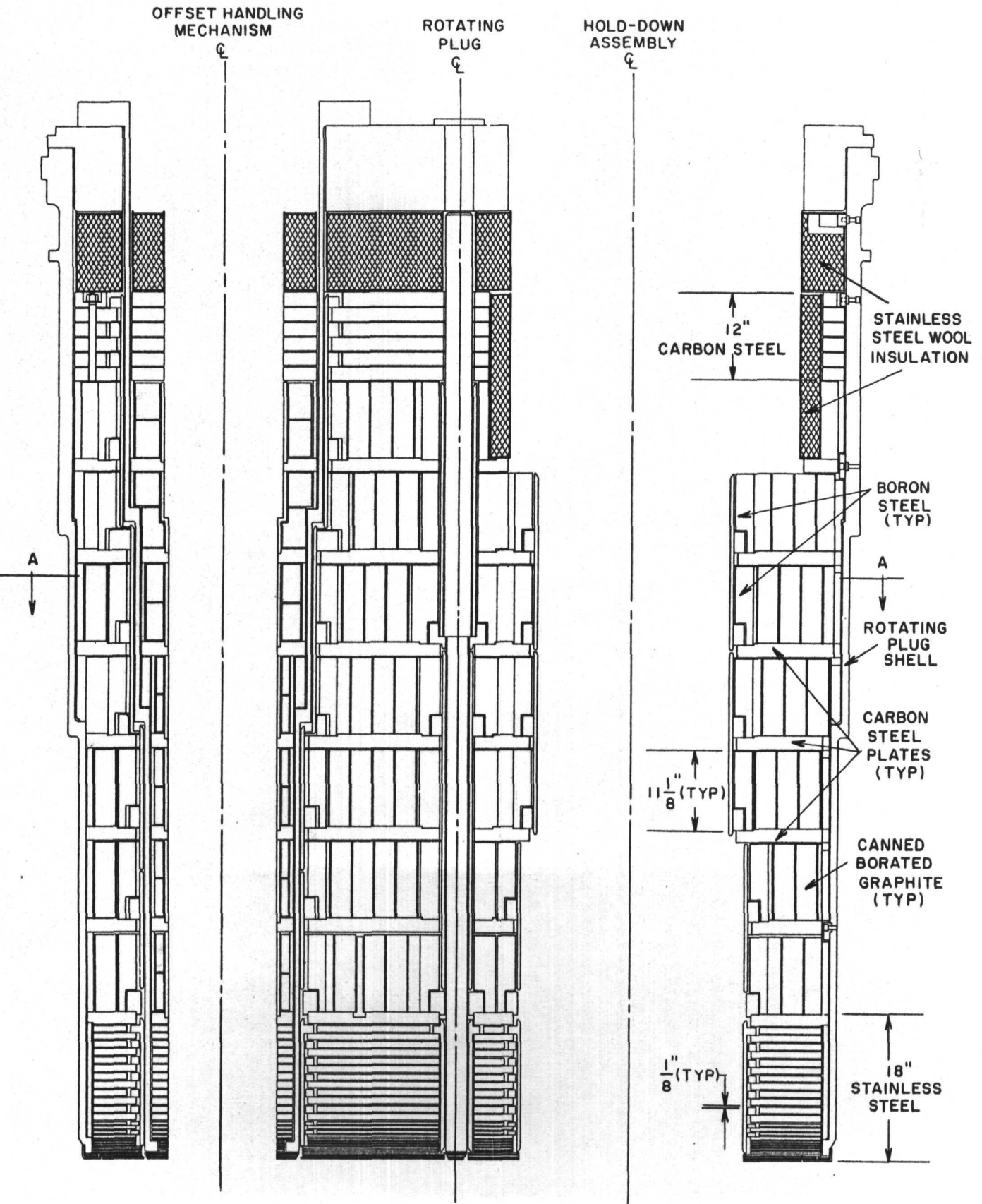
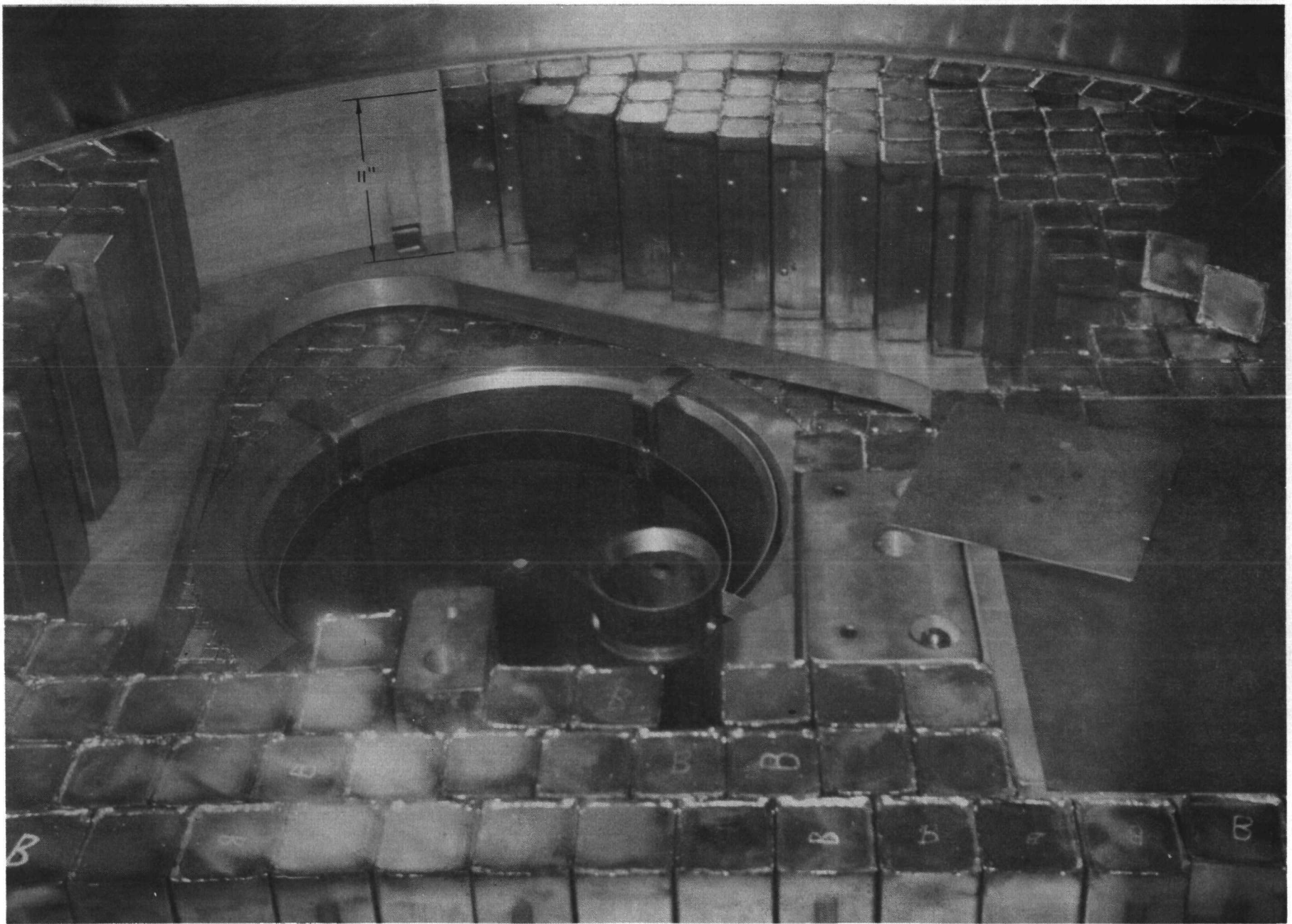


Fig. 3 ROTATING SHIELD PLUG (SECTIONAL ELEVATION)

FIGURE 4



INTERIOR OF ROTATING PLUG SHOWING INSTALLATION  
OF CANNED GRAPHITE SHIELDING



### C. Can Design

As mentioned above, the graphite in the plug is canned in carbon steel cans. The dimensions of these cans are 3" x 3" x 11", made of 16 gauge carbon steel. The graphite block inside each can is 2-5/8" x 2-5/8" x 10-7/16". The difference in dimensions allows space for any differential expansion between the graphite and the steel can.

In the top of each can are three holes about 50 mils in diameter. The purpose of these holes was to prevent pressure build-up due to temperature changes and, in the case of the borated graphite, to allow for helium release. It was through these holes the carbonaceous gases escaped and entered the primary sodium.

## II. SOURCES OF CARBON IN THE PRIMARY SODIUM.

### A. Gas Evolution

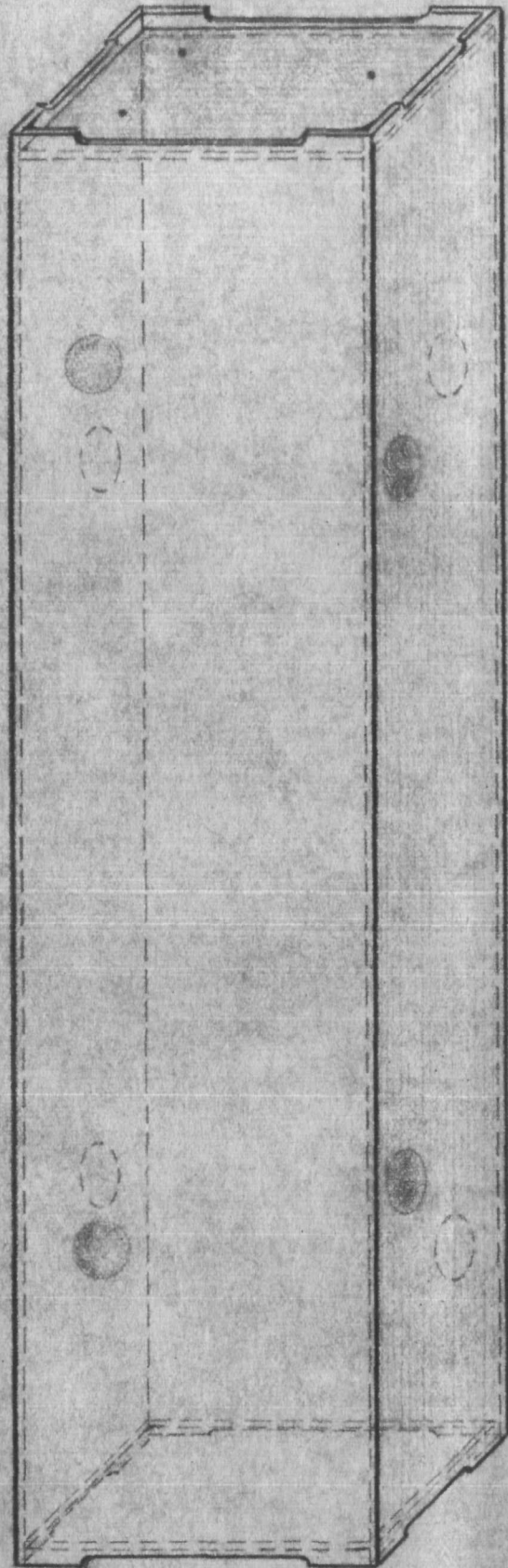
The graphite shielding material in the rotating plug actually is a semigraphitic material (Reference 1). The material in its initial step of the manufacturing process consisted of graphite particles bound together by molding powder in the weight ratio of 3 parts graphite to one part molding powder. The molding powder consisted of a phenol formaldehyde resin blended with 10 per cent by weight of hexamethylenetetramine which was employed as a hardener. The raw (or green) mixture was blended and was then press molded at 265 F into blocks. Next, the material was kiln dried for 10 hours at 1000 F. The end product of this process consisted of the graphite particles bound together by carbon particles and incompletely decomposed molding powder.

The gases and vapors which were released from the plug graphite during the 1000 F test are the products from the thermal decomposition of the remaining molding powder. These reaction products consist of CO, CO<sub>2</sub>, CH<sub>4</sub>, H<sub>2</sub>, H<sub>2</sub>O, N<sub>2</sub>, O<sub>2</sub>, a trace of C<sub>2</sub>H<sub>6</sub>, and a complex mixture of higher boiling organic compounds called pyrolyzates. These reaction products were released from the plug graphite shielding cans into the reactor vessel cover gas through which they could diffuse or be transported to the sodium.

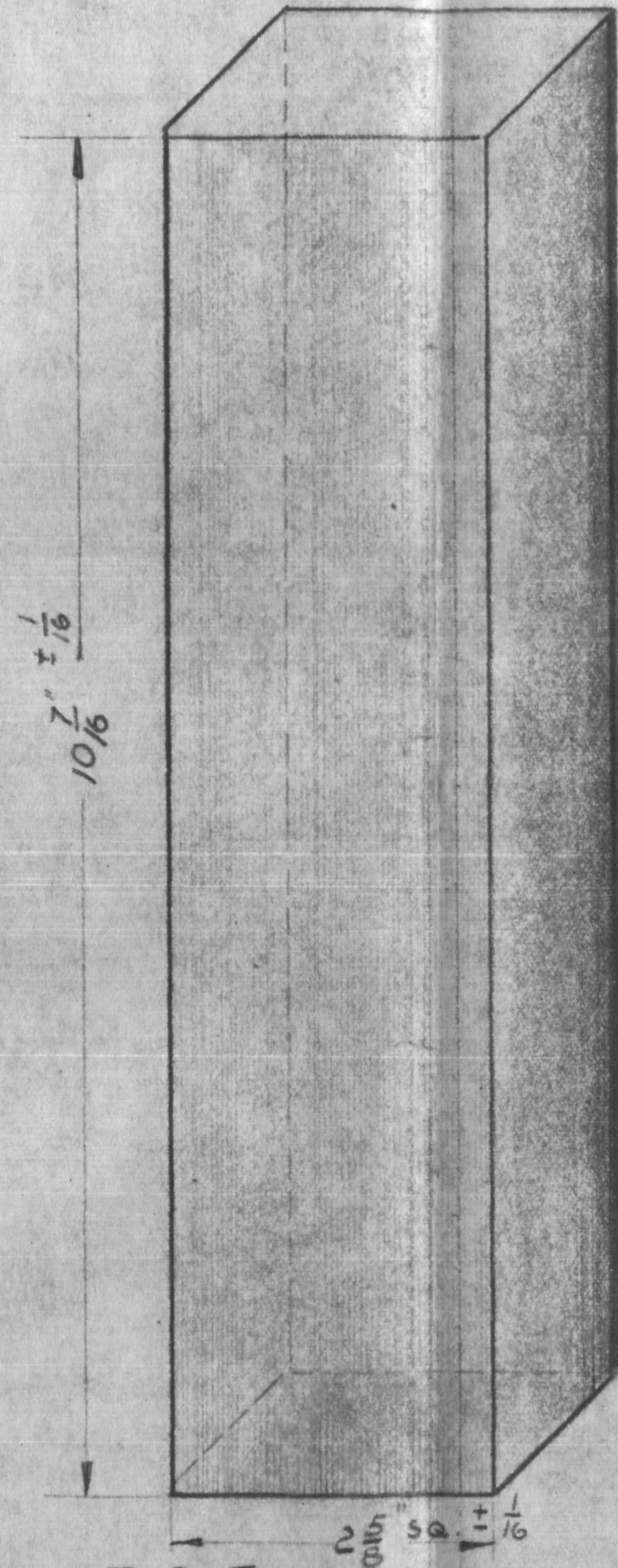
### B. Amount of Carbon Containing Gases Released by Plug Graphite During the 1000 F Tests

A series of tests has been run on samples of the plug graphite to determine the outgassing characteristics of the material (Reference 3). The samples were obtained from bricks of plug graphite which were being held in storage and which had not been installed in the plug. The test specimens included some full size bricks and numerous smaller pieces. There is considerable variability in the data, but the difference between full size bricks and the smaller pieces is not statistically significant. Using the results of the full brick tests (Reference 4) and the information on the temperature history of the plug (Reference 5), a calculation

CANISTER



GRAPHITE BLOCK



SECTION  
SHOWING CLEARANCES  
AROUND GRAPHITE IN CANISTER

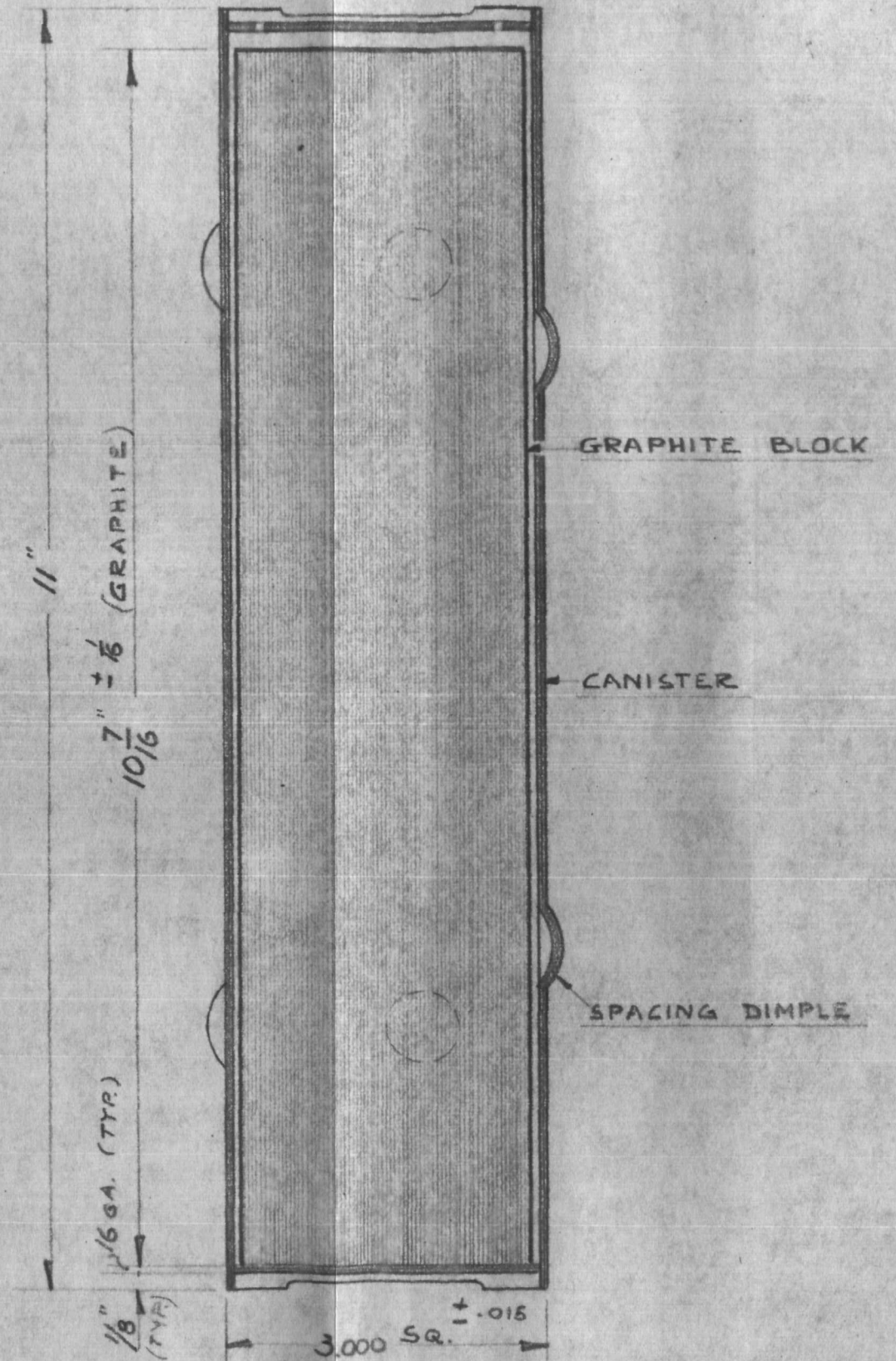


FIG 5.

GRAPHITE FOR ROTATING PLUG

has been made of the volume of carbon-containing gases evolved during the 1000 F isothermal. The results of the calculation are summarized as follows:

<u>Gas</u>	<u>Cubic Feet</u>
CO	25
CO <sub>2</sub>	78
CH <sub>4</sub>	197
C <sub>2</sub> H <sub>6</sub>	3

The tests showed that the quantity of pyrolyzate released during outgassing of the graphite was substantial (Reference 3). On the basis of the test results, it is estimated that the volume of pyrolyzate vapors released during the 1000 F test was equivalent to 3 liquid gallons.

C. Amount of Carbon Introduced into the Sodium

1. Carbon in Sodium

After the presence of the plug graphite offgases was detected, purging of the cover gas with argon was begun. Some fraction of the offgases undoubtedly was removed from the system by this process and did not react with the sodium. The pyrolyzate vapors have a high boiling range and some fraction may have diffused upward into the cooler regions of the plug where condensation would have occurred.

Assuming that all of the gases reacted with the sodium and that only reactions 2, 5, 14 and 15 occurred (see Section II-D), the quantity of gas given in the preceding tabulation would result in the introduction into the sodium of 10 pounds of carbon--of which 8 pounds would be elemental form and 2 pounds would be combined as the carbonate. Also assuming that all of the pyrolyzate vapors reacted with the sodium and were decomposed to the elements (Equation 17, Section II-D), this would have introduced into the sodium 23 pounds of carbon in elemental form.

Using the above quantities, the total carbon introduced into the sodium is 31 pounds as elemental carbon and 2 pounds as carbonate. Considering that there was probably some effective purging of the cover gas and some condensation of the pyrolyzate vapors, the assumption of 100 per cent reaction with the sodium undoubtedly biases the calculated carbon content on the high side. On the other hand, the calculations are based on data from outgassing tests run on only a few samples of the plug graphite and the test results showed considerable variability from one sample to another. Therefore,



the quantities of gases and vapors used in the calculation may not represent the true average for all the plug graphite. However, judging from the available data it is believed that the calculated gas release values are within a factor of 2 of the actual values and that the calculated pyrolyzate release values are within a factor of 3 or 4 of the actual values.

The computed values for elemental carbon and carbonate reported above are equivalent, respectively to 90 ppm and 6 ppm carbon in the primary sodium. The average elemental carbon content of the sodium as received was reported by the supplier as 30 ppm. The supplier did not report the carbonate content. After the plug graphite outgassing occurred, the sodium in the primary system was sampled 24 times for purposes of determining the carbon content. Multiple samples frequently were obtained during a single sampling operation. At least one sample from each sampling operation was utilized for an elemental carbon analysis and a total of 28 samples were thus analyzed. The average value of the 28 samples was 95 ppm. The standard deviation of the individual samples was 46 ppm. The range of values was 15 ppm to 192 ppm. The cause of the wide variation is not known, but it could be due to the presence of particulates in the sodium resulting in a nonhomogeneous sample; to sample contamination during handling; or, to analytical scatter. Nevertheless, the analytical values are not inconsistent with the computed values. Most of the samples also have been analyzed for carbonate. The average of all analyses is approximately 70 ppm equivalent carbon as carbonate. The scatter in the values initially was worse than for the carbon values, ranging from <10 >1000 ppm equivalent carbon.

After the primary system was drained, samples of the sodium were taken directly from the storage tanks. Three samples were taken soon after the transfer of the sodium to storage, and the carbon and carbonate analyses which were obtained appeared to be somewhat higher than the values previously obtained while the sodium was still in the primary system. Several months later four more samples were taken and the analyses showed substantially lower values of elemental carbon and carbonate and greatly reduced scatter. The average analysis obtained from the four samples was 33 ppm elemental carbon and 11 ppm equivalent carbon as carbonate. Substantially the same values were obtained from analysis of samples taken from storage just prior to return of the sodium to the primary system.

Since the sodium has been back in the primary system eight samples have been removed from the primary system. The average elemental carbon content is 19 ppm, and the average carbonate value is 10 ppm equivalent carbon.

During this time, improved sampling techniques and analytical techniques were developed and used. Therefore, the initial values may be very uncertain. The discrepancy between the initial



measured values could be attributed to this uncertainty and the improvement in measuring techniques.

The lower values obtained for the carbon content cannot be evaluated in terms of known amounts of carbon removal during the cleanup operations. Little carbon was removed by either filtering or hot trapping. However, the amounts removed by settling and by cold trapping are not known. However, there is some evidence that cold trapping will remove carbon.<sup>(15)</sup>

In summary, then, there is a discrepancy between the analytical results obtained for the carbon content of the sodium prior to cleanup and subsequent to cleanup. The final measured values could be attributed to an improvement in measuring techniques, possible settling out of the carbon while the sodium was in storage, and removal of carbon in the cold trap.

## 2. Solubility of Carbon in Sodium

The solubility of elemental carbon in sodium has been investigated at KAPL and the work is reported in KAPL-1807 (Reference 10). The data show a strong dependence of solubility on the oxygen content of the sodium. The data are reproduced on the graph in Figure 6. The 10 ppm oxygen curve shown on the graph has been derived by a linear cross-plot of the 40 ppm and 240 ppm curves, both of which were determined experimentally. Based on plugging temperature measurements, the oxygen content of the Fermi sodium has been  $\leq 10$  ppm. Taking the KAPL data at face value and assuming the data cross-plot is valid, the quantity of elemental carbon that could be maintained in solution has been calculated for the primary system.

500 F	35 ppm	11 lbs
600 F	39 ppm	12 lbs
700 F	43 ppm	13 lbs
800 F	46 ppm	14 lbs
900 F	49 ppm	15 lbs
1000 F	53 ppm	16 lbs

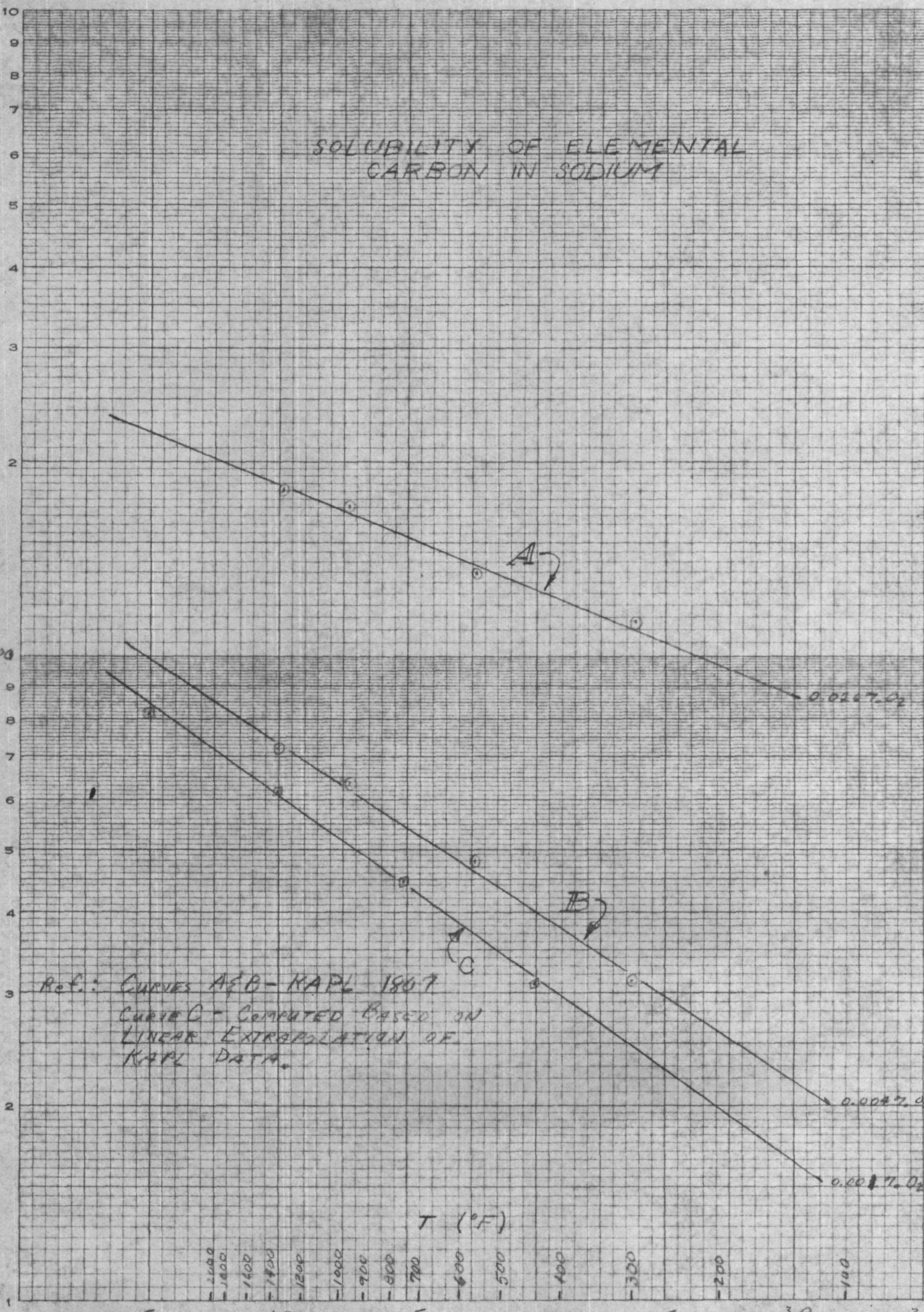
The solubility of sodium carbonate in sodium is reported in the literature (Reference 11). The data are reproduced on the graph in Figure 7. The quantity of sodium carbonate that could be maintained in solution in the primary system is tabulated as follows:

1000

# SOLUBILITY OF ELEMENTAL CARBON IN SODIUM

EUGENE DIETZGEN CO.  
MADE IN U. S. A.

Carbon (ppm)



NO. 340R-L210 DIETZGEN GRAPH PAPER  
SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH

FIG. 6

NO. 340R-L210 DIETZGEN GRAPH PAPER  
SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH

EUGENE DIETZGEN CO.  
MADE IN U.S.A.

Concentration  $\text{Na}_2\text{CO}_3$  (ppm)

Solubility of  $\text{Na}_2\text{CO}_3$  in Sodium

10

300

400

500

600

700

800

900

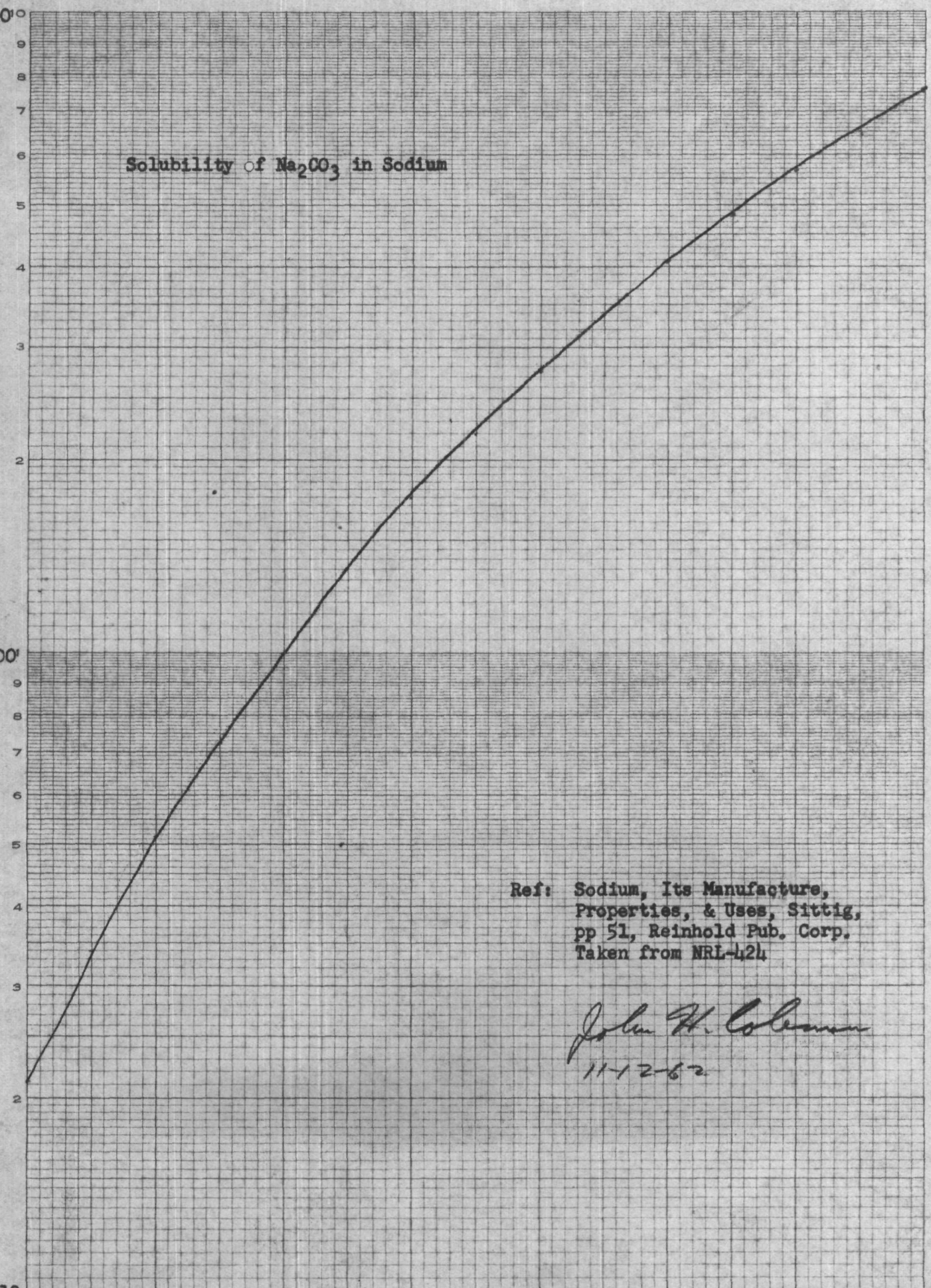
1000

Temp. °F

Ref: Sodium, Its Manufacture,  
Properties, & Uses, Sittig,  
pp 51, Reinhold Pub. Corp.  
Taken from NRL-424

*John H. Coleman*  
11-12-62

Fig. 7



300 F	20 ppm	6 lbs
500 F	100 ppm	30 lbs
600 F	175 ppm	53 lbs
700 F	280 ppm	84 lbs
800 F	400 ppm	120 lbs
900 F	550 ppm	165 lbs
1000 F	750 ppm	225 lbs

It is known that some elemental carbon in particulate form was present in the primary system following outgassing of the plug. About one pound of carbon particles have been recovered in the various cleanup operations (see Section V of this report). On the basis of the estimated carbon release to the sodium and based on the carbon solubility data, the presence of the particulate carbon is not surprising.

Although carbonate has been found in the filter residues, it is not certain that the carbonate was filtered from the sodium. The carbonate could have resulted from exposure of the residue to the air prior to analysis. On the basis of the estimates of input of carbonate into the system and based on the solubility of the carbonate, all of the carbonate should have been in solution.

### 3. Crust Formation

The possibility of crust formation due to plug graphite outgassing during operation will depend on the rate of gas release and the rate of reaction with the sodium. As stated previously, the rate of release is estimated to yield a maximum of 2 pounds of carbon per year. These quantities are equivalent to about 7 ppm carbon and 14 ppm carbonate per year in the primary system. Tests conducted by Atomics International (AI) on the reaction rates of carbon dioxide and carbon monoxide with sodium show that above 750 F these gases react almost instantaneously (Reference 16). Other observers have reported the reaction of sodium with carbon monoxide to proceed vigorously at much lower temperatures.

In light of the very small quantities of carbon and carbonate that will be released to the sodium and the very low rate of release, it seems reasonably certain that there will not be a crust formed during plant operation.



#### D. Chemistry Involved

The actual reactions into which the gases entered with the sodium are not known. Based on a review of previous work and thermodynamic considerations, numerous reactions could have occurred, (Reference 2). A list of these is shown in the following table:

TABLE I  
CHEMICAL FORMULAE

1.  $2\text{Na} + \text{CO} = \text{Na}_2\text{O} + \text{C}$
2.  $2\text{Na} + 3\text{CO} = \text{Na}_2\text{CO}_3 + 2\text{C}$
3.  $4\text{Na} + 3\text{CO} = \text{Na}_2\text{CO}_3 + \text{Na}_2\text{C}_2$
4.  $6\text{Na} + 6\text{CO} = (\text{NaCO})_6$
5.  $4\text{Na} + 3\text{CO}_2 = 2\text{Na}_2\text{CO}_3 + \text{C}$
6.  $4\text{Na} + \text{CO}_2 = 2\text{Na}_2\text{O} + \text{C}$
7.  $4\text{Na} + \text{O}_2 = 2\text{Na}_2\text{O}$
8.  $2\text{Na} + 2\text{H}_2\text{O} = 2\text{NaOH} + \text{H}_2$
9.  $4\text{Na} + \text{Na}_2\text{CO}_3 = 3\text{Na}_2\text{O} + \text{C}$
10.  $2\text{Na} + \text{NaOH} = \text{Na}_2\text{O} + \text{NaH}$
11.  $\text{Na}_2\text{O} + \text{CO}_2 = \text{Na}_2\text{CO}_3$
12.  $2\text{Na} + \text{H}_2 = 2\text{NaH}$
13.  $\text{NaOH} + \text{CO}_2 = \text{NaHCO}_3$
14.  $\text{CH}_4 = \text{C} + 2\text{H}_2$
15.  $\text{C}_2\text{H}_6 = 2\text{C} + 3\text{H}_2$
16.  $2\text{Na} + 2\text{C} = \text{Na}_2\text{C}_2$
17.  $\text{Pyrolyzate} + \text{heat} = n\text{C} + n\text{H}_2$

There is very little information available on the kinetics of these reactions. However, it is believed that reactions 2, 5, 7, 8, 10, 12, 14, 15, and 17 predominated, resulting in the formation of C,  $\text{Na}_2\text{CO}_3$ ,  $\text{Na}_2\text{O}$ , and NaH. There is data and some evidence that reactions 3 and 4 also may have occurred to some extent.

An increase in the plugging temperature confirms the formation

of sodium oxide. Chemical analysis of the sodium and of solids recovered from the primary system have confirmed the formation of carbon and sodium carbonate. Qualitative tests which were made for NaH, Na<sub>2</sub>C<sub>2</sub> and (NaCO)<sub>2</sub> showed evidence of the hydride and carbide, but there was no positive indication of the carbonyl.

#### E. Possible Outgassing of Plug Graphite During Reactor Operation

To evaluate the problem of plug graphite outgassing during reactor operation, a series of tests has been run to determine what additional quantity of gas will be released. Three possible causes of additional outgassing were recognized and were investigated; viz., temperature, thermal cycling, and radiation.

##### 1. Effect of Temperature and Thermal Cycling

Samples of plug graphite were heated from room temperature to 1000 F in 100 F increments (Reference 3). The rate of gas evolution was measured as a function of time at each temperature level until gas evolution ceased. Following cessation of outgassing at 1000 F, the samples were cooled to 500 F and the cycle was repeated twice. The results of these tests show that no further outgassing of the plug graphite will occur due to thermal effects (Reference 6 and 7). During the recent heat-up of the primary system to 750 F, there was no evidence of plug graphite outgassing.

##### 2. Effect of Irradiation

In-pile irradiation tests were run in the temperature range 700 F to 1000 F to determine if irradiation would cause gas to be released from the plug graphite (Reference 8). Based on the results of these tests, the upper limit for gas release from the plug graphite is computed to be 188 scf/year at 200 iW Core A operation (Reference 6). The irradiation effect is directly proportional to the absorbed dose, so at 430 iW Core A operation the outgassing rate would be approximately twice as much. These gases are reported to be mostly hydrogen (Reference 9). However, assuming the composition of the gases were similar to that in the thermal outgassing tests, the amount of carbon introduced into the sodium during 200 iW Core A reactor operation could amount to 2 pounds per year. This estimate of the carbon is probably very conservative because the composition of gases resulting from radiation exposure of organic substances is reported to be mainly hydrogen (Reference 9).

### III. METHODS OF CARBON REMOVAL

To date, three methods have been used to remove the carbon from sodium. These are mechanical separation, cold trapping, and hot trapping. Some removal of carbon has been achieved with these, but none has been completely successful. Other methods of removal such as distillation, using chemical additives, etc., have been considered but have not been tried. This section of this report is devoted to a discussion of the methods which have been attempted to date to remove the carbon.

#### A. Mechanical Removal

##### 1. Filters

Dummy subassemblies containing filter screens were present in the core section during preoperational testing. The screens were of two grades, the "coarse" type with approximately a 10-mil opening and the "fine" filters with approximately a 2-mil opening. About 50 of each type were installed during most of the test period.

Figure 8 shows the arrangement of the component parts of the filter subassemblies. The shroud around the screen is designed to prevent the trapped material from falling back into the sodium system when the pumps are stopped.

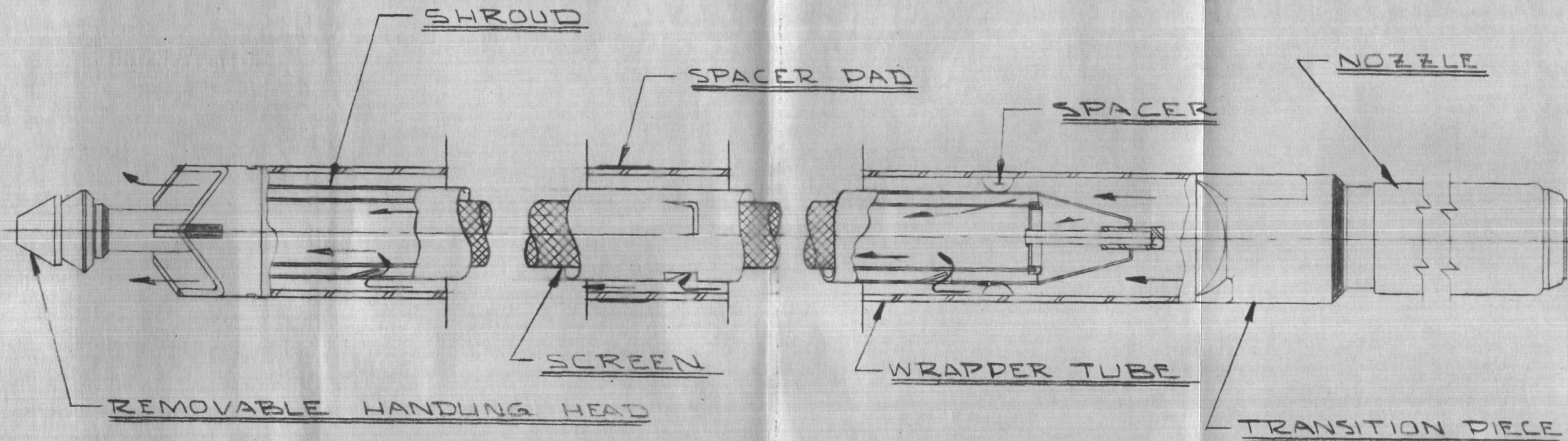
When the filters were removed from the reactor, the residue from some was weighed and analyzed. On the basis of these data, the amount of carbon removed from the primary system by the filters is estimated as follows:

Material on each of the fine screens	-	4-5 grams
Material on each of the coarse screens	-	0.2-0.4 grams
Total material on all the screens	-	235 grams

These results showed that some of the carbon in the sodium is filterable. Based upon this premise, the primary sodium in the storage tanks was filtered through a 6-mil filter and through a 1.5-mil filter. Only gram quantities of residue were collected on the filters, indicating the solids had settled out in the storage tanks.

Tests were conducted on a 400-gallon sample of the sodium to determine the efficacy of 5-micron filters to remove carbon from the sodium. Again, negligible quantities of residue were collected.

When the work on the hold-down mechanism was completed, clean filter subassemblies and filter trap subassemblies were installed in the reactor prior to filling with sodium. Since then the sodium has been circulated through these filters. The fine filter subassemblies can filter and hold all particles from about 2 mils to



FILTER SUBASSEMBLY

FIG. 8

R. CAREY  
10-2-62



1/8 inch. The filter trap subassemblies can trap all larger particles which can enter the nozzles of the subassemblies. These larger particles could have dropped back into the system from the original filter subassemblies.

Since the primary system was refilled, two fine screen filter subassemblies and two filter trap subassemblies have been removed specifically for purposes of recovery and analyses of residues. Both of the filter trap subassemblies have been substantially clean--that is, one or two flakes of residue weighing only a fraction of a gram have been found. The two fine screen filter subassemblies contained 12 gm and 15 gm of residue of which 23% and 7% respectively was elemental carbon. The fraction of carbonate is not available. The quantities of residue found on these filters are not significantly different from what was recovered from filters removed following the plug graphite outgassing. Apparently, during the draining of the primary system a small amount of undissolved material remained in the system. Removal and examination of additional filters is scheduled prior to nuclear operation. Subsequent to the time the above information was obtained, a clean fine screen filter subassembly was placed in the core and was left there for several weeks during which time the sodium was being circulated. This filter has been inspected and was found to be completely free of particulates.

## 2. Hydroclones

A Dorr hydroclone unit was installed in the primary sodium service system and 8,700 gallons of sodium were passed through it. Under the conditions of operation, the hydroclone should have removed 95 per cent of the carbon particles 5 microns or larger in diameter. Nevertheless, only one-half gram of solids was collected from the 8,700 gallons. These solids were analyzed for carbon only and were found to contain 51 per cent carbon.

## B. Cold Trapping

The cold trap can remove materials from sodium which have a temperature-dependent solubility in sodium. Some reports indicate that both carbon and sodium carbonate have a temperature-dependent solubility in sodium (see Figures 6 and 7). For this reason, the cold trap was run extensively in an effort to reduce the carbon content of the sodium. However, based upon the results of chemical analysis of the sodium for carbon and carbonate, it is not known whether this cold trapping had much effect on the concentration of these materials in sodium or not. The cold trap has been removed and its contents are being analyzed to determine, among other things, whether any carbon or carbonate is contained in the residue.

The curves of Figure 9 show the data obtained during plug outgassing and also show that the cold trap did reduce the plugging temperature to acceptable limits subsequent to the outgassing.

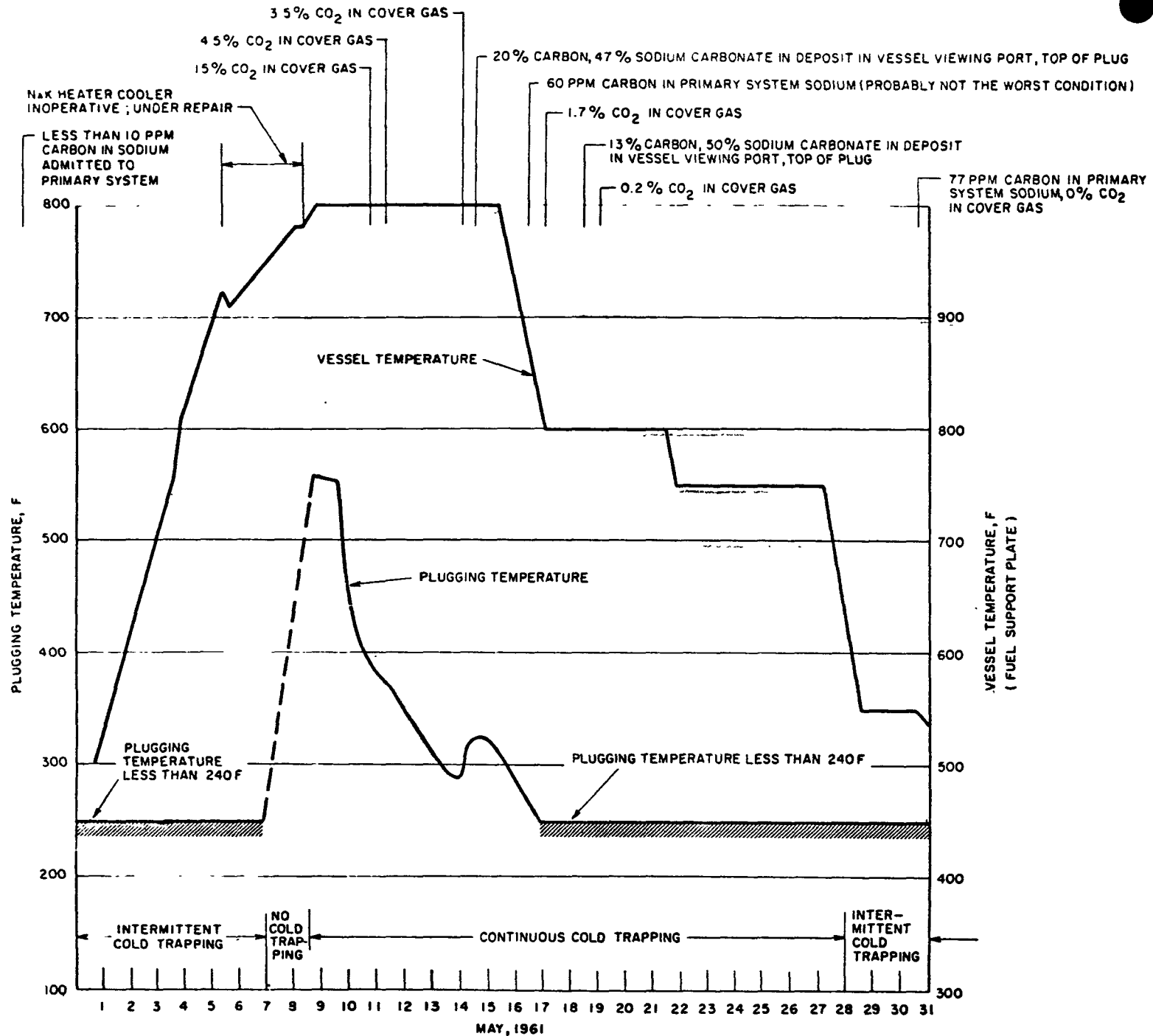


Fig. 9. Data Taken During 1000 F Test

### C. Hot Trapping

A hot trap utilizing stainless steel foil was installed in the primary sodium service system. The temperature of the hot trap and the sodium flowing through it was maintained at 1200-1300 F.

The amount of stainless steel foil in the trap was 1080 lb having a surface area of 1830 ft<sup>2</sup>. This area was divided about equally between 10-mil corrugated foil and 20-mil flat foil. The foil in the trap was in the form of coils built up of alternate layers of flat and corrugated foil.

After about three hundred hours of operation of the hot trap at 1200 F with a system temperature of about 600 F, the trap was cut open and the foil analyzed for carbon content. The results of these analyses are shown in Table II.

Since no statistically significant carbon increase was found in the foil, considerable experimental work is being conducted to account for this and to develop means of removing carbon from sodium. These experiments are discussed elsewhere in this report.

It is pointed out that during operation of the primary system at 1000 F, the stainless steel components were carburized to varying degrees. For the most part, the depth of carburization was about 0.001" or less and represented a carbon increase in this case of about 0.10%. Since this slight degree of carburization did occur, we know that at least some of the carbon in the system at that time was in a form capable of carburizing stainless steel. On this basis, it is assumed that the form of the carbon in the sodium was changed on lowering the sodium temperature to 500 F or, more likely, that on reheating the sodium to 650 F for the hot trap operation, an insufficient amount of carbon was redissolved to cause carburization of the hot trap materials or that the carbon was reduced by the cold trapping which preceded hot trapping.

TABLE II  
HOT TRAP CARBON PICK-UP SUMMARY

Original carbon content of foil

By mill report	.07 (10 & 20 mil foil)
By lab	.08 (20 mil foil)
	.10 (10 mil foil)

Final carbon content of foil

A. 10 mil samples from top section

<u>Tag</u>	<u>Carbon</u>	<u>Location</u>
01-TO-A	.12	At circumference
01-TO-B	.08	" "
01-TO-C	.09	" "

B. 20 mil samples from top section

<u>Tag</u>	<u>Carbon</u>	<u>Location</u>
02-TO-A	.09	At Circumference
02-TO-B	.09	" "
02-TO-C	.09	" "
02-TI	.10	At core

C. 20 mil samples from center section

<u>Tag</u>	<u>Carbon</u>	<u>Location</u>
02-CO-A	.11	At circumference
02-CO-B	.11	" "
02-CO-C	.09	" "
02-CI	.10	At Core

D. 20 mil sample from bottom section

<u>Tag</u>	<u>Carbon</u>	<u>Location</u>
02-BO-A	.10	At circumference
02-BO-B	.10	" "
02-BO-C	.09	" "
02-BI	.09	At core

#### IV. DISCUSSION OF POSSIBLE PROBLEMS

##### A. Possible Problems Caused by Plug Graphite Off-Gases

The off-gases from the plug graphite possibly could cause several problems; namely,

1. Formation of a crust on the sodium surface that could interfere with operation of mechanical components.
2. Carburization of stainless steel and other structural materials exposed to carbon in the sodium.
3. Formation of particulate material in the sodium.

##### B. Crust Formation

During and/or immediately following the high temperature test conducted by AIDA, a crust formed on the surface of the primary sodium in the reactor. This crust, upon cooling, was such that it "froze" the rotating plug in position so that it could not be turned. This was freed later by "washing" the crust out of the annulus around the plug with sodium.

Several questions arose concerning the crust material. Some of the most important questions were:

1. What is the crust?
2. What is its shear strength?
3. Will it disperse or dissolve in sodium?

To answer these questions, several analyses and tests were conducted. An analysis of some of the crust material showed the composition to be:

Sodium (Na)	13.1%
Sodium Oxide (Na <sub>2</sub> O)	13.7%
Sodium Carbonate (Na <sub>2</sub> CO <sub>3</sub> )	66.2%
Carbon (C)	2.9%
Residue	0.1%

In tests at various temperatures, the shear strength was found to be:

100 psi	at 70 F
24 psi	at 190 F
0 psi	at 500 F

An experiment was conducted to determine qualitatively whether the crust material would disperse or dissolve in liquid sodium. It was found that at 500 F the material did not disperse at all. At 600 F, it dispersed in about a week; while at 750 F, it dispersed in about one day.

As indicated in Section II of this report, something less than 200 cubic feet of gases per year may be evolved from the plug graphite during operation. Assuming uniform evolution, this is only about 0.55 cubic feet per day. This quantity is low enough that it can be handled by dissolution in the sodium followed by subsequent removal in the purification system. In other words, no crust should form. However, should a crust tend to form, it would present no safety problem since it would be dispersed and/or dissolved when the reactor sodium surface is above 750 F during operation. Also, there would be no shear strength to restrict operation of mechanical components at this temperature. The control and safety rod latches are below the sodium level so they are unaffected.

## C. Carburization

### 1. Carburization of Stainless Steel in Sodium

Carburization of stainless steel in sodium is a two-step phenomena. First, there is a reaction between the carbon in the sodium and the steel which results in the formation of a carbide on or at the steel surface, undoubtedly in the form of a chromium carbide. Secondly, the carbon diffuses into the base metal to form the carburized case consisting of carbon in solid solution and precipitated carbides. The rate at which carburization proceeds depends on the following factors:

#### a. Carbon Content in Sodium

The single most important factor in carburization is the carbon content of the sodium. The carbon content will vary with temperature, and at each temperature there is undoubtedly an equilibrium concentration which will not result in carburization. Reference 12 suggests that this is about 20 ppm at 1000 F. At increasingly higher levels, carburization will proceed at increasingly faster rates due to the formation of higher steel surface carbon content. At lower levels, carburization will not occur.

#### b. System Temperature

The system temperature is important since it affects carbon concentration in the sodium, the rate of surface carbide formation and the rate of carbon diffusion into the steel. When sodium is saturated with a contaminant and the temperature of the sodium is lowered, the contaminant crystallizes out of solution quite readily. This is shown by the plugging indicator and cold trap operation. In fact, this is the principle upon

which the plugging indicator works. However, once the contaminant has precipitated out of solution, raising the temperature of the system does not immediately redissolve the contaminant. Dissolution is a very slow process and is quite temperature dependent. This appears to be true for carbonaceous materials as it is for other contaminants. Once the carbon has precipitated out of solution, it will require some time to redissolve it. Reference 12 indicates that at temperatures less than about 850 F, the diffusion coefficient is so small that even if all other conditions are right for carburization, there would be no concern, since the carburized case would be extremely thin, even after many years of operation.

c. Condition of Metal

The condition of the metal subjected to carbon contaminated sodium also appears to be a significant factor in carburization. The main effect here is associated with the diffusion coefficient of the carbon in the metallic lattice. As in most diffusion processes, diffusion is enhanced by stress conditions or by cold working. This effect has been shown in Reference 13.

d. Other Impurities in Sodium

At the present time, there is very limited data on the effect of other impurities in the sodium. The fact that carburization of stainless steel components in the system during the 1000 F isothermal test proceeded at a slower rate than predicted in Reference 12, suggests that the carbon was tied up in some form such that it would not react with the steel or the carbon content was not as high as predicted. In Figure 6 it was shown that the oxygen content had a gross effect on the carbon solubility. It would appear that a carbonate complex is formed that is stable in the presence of stainless steel.

It is also proposed that the presence of impurities such as oxygen, hydrogen or nitrogen may form a protective film on the metallic surfaces which is impervious to carbon.

2. What Materials Are Affected

The discussion thus far has been devoted to stainless steel. For the most part, the other metals in the system are not significantly affected by carbon.

The nickel and cobalt base alloys, such as Colmonoy, Inconel, and Stellite do not have any carbon solubility and, therefore, are not carburized. The active metals such as tantalum and zirconium apparently form a tight adherent carbide layer which prevents further carburization.

### 3. Effect of Carburization on Stainless Steel.

The effect of carburization on some mechanical properties of stainless steel is reported in Reference 14. In general, carbon tends to strengthen stainless steel and reduce ductility. However, the reduction in ductility is small for carbon contents up to 0.5%. For 1.3 w/o carbon, elongation of 12% is observed.

A carburized case tends to increase the fatigue strength for strains up to about 1%. From the standpoint of reactor vessel integrity, this effect would appear to be beneficial.

### D. Plugging of Core and Blanket Subassemblies by Carbon Particles

The effect of approximately spherical particles of various sizes on the operation of the subassemblies is considered here for each type of subassembly.

The stated particle sizes are based upon geometry only. It should be reiterated that the sodium will have been filtered thoroughly prior to installing the core. Also, the clear space for flow of primary sodium in the IHX between the tubes is 0.1875 inch (3/16"). Therefore, the likelihood of plugging a subassembly is very remote.

#### 1. Core Subassembly

Considerable effort has been expended to remove all particulate matter from the primary sodium system prior to loading fuel subassemblies. As mentioned in Section III, the sodium has been thoroughly filtered during the preoperational testing period. Also, filter trap subassemblies were used during this period to remove large particles which could possibly be entrained by the sodium.

It is now inconceivable that particles are remaining in the system large enough to completely plug the subassembly. This would require a particle 1.408 inches, the ID of the lower end of the subassembly. Each subassembly has a strainer in the lower portion. The ID of this strainer is 1.2 inches. Although it is unlikely, should a particle of this size enter a subassembly, it would greatly reduce the flow through the subassembly. However, there would still be a flow path through the conical portion of the strainer.

Particles of about 0.040 inches or larger will be retained by the strainer in the subassemblies. These strainers have holes of 0.040" + 0.002" in diameter. It would require a large number of particles to reduce flow in this area appreciably.

A cell of the birdcage assembly holding a fuel pin is shown in Figure 10. The maximum allowable diameter of a fresh fuel pin is 0.159 inches. Under irradiation the pin is expected to increase in diameter to 0.172 inches at 0.8 per cent burnup. The minimum internal dimension of the cell is 0.182 inches.



As shown in the upper portion of the sketch, Figure 10, particles smaller than 0.041 inches would pass through the birdcages of a fresh subassembly. Particles of this size and larger should be stopped by the strainers. Even after irradiation of the fuel to 0.8 per cent burnup, particles 0.035 inches and smaller would still pass through the subassembly.

## 2. Inner Radial Blanket Subassembly

The inner radial blanket (IRB) subassemblies have orifices in the entrances of 0.5 inches. Based upon the filtering which preceded loading these and the improbability of entraining particles of this size or larger in the sodium it is unlikely that flow restrictions will occur here. Particles of 0.2 inches or smaller can pass completely through the inner radial blankets. •

## 3. Outer Radial Blanket Subassembly

For flow control, the lower support plate for the outer radial blanket is orificed. These orifices vary in size between 0.250 inches and 1.2 inches. The sodium flow rate in the outer radial blanket area is very low and follows a very tortuous path prior to entering the plenum below the support plate. This precludes carrying particles large enough to plug these orifices. As in the inner radial blanket subassemblies, particles of 0.2 inches or smaller can pass through the outer radial blanket subassemblies.

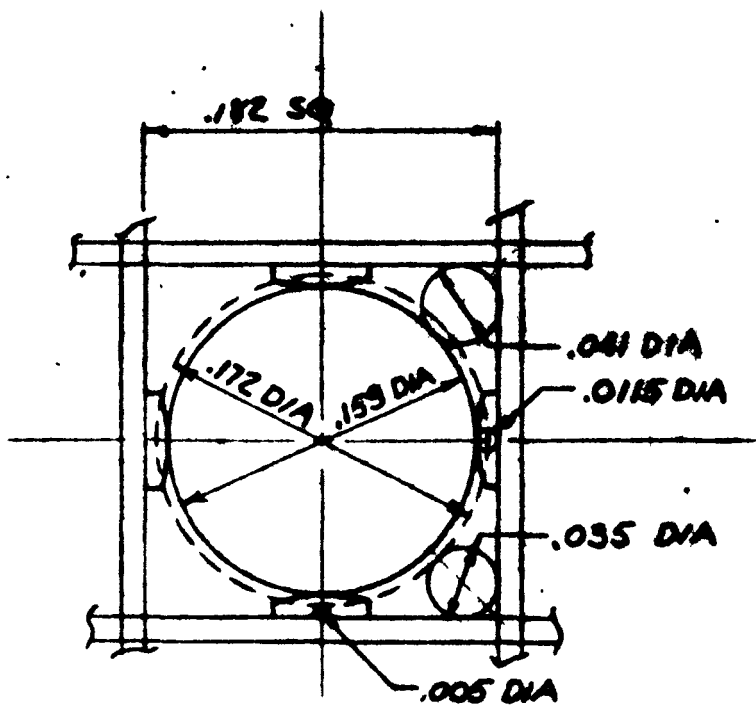
# V. OPERATIONAL CONTROL OF CARBON

## A. Experimental Program

### 1. Short Range Tests

FEA is conducting carburization tests on the primary sodium. These tests will establish some of the parameters of carburization such as:

- a. Is the carbon now in the primary sodium in a form capable of carburizing stainless steel 304? Based on preliminary results, it appears that there is a carburizing potential in the Fermi sodium.
- b. What effect does the bulk sodium temperature have on the carburization of stainless steel? This is still being studied.
- c. Does the oxygen concentration in the carbon-contaminated sodium have an effect on the composition of carbon and as a result on carburization of the stainless steel? Based on preliminary results, it does not appear that the oxygen content of the sodium exerts much influence on carburization.



Scale 10X

Fig. 10. Cross Section of Fuel Pin in a "Cell" of the Birdcage

Final answers to these questions will indicate whether it is feasible to clean carbon from the sodium with a hot trap and will define the operating parameters to be followed for efficient hot trapping and to prevent carburization of the primary system.

In conducting the tests, sodium from the storage tanks at Fermi (primary sodium) was put into two pots. One of these pots had a known amount of oxygen (about 50 ppm Na<sub>2</sub>O) added. The bulk sodium temperature was held constant at three different temperature levels: about 500, 800, and 1000 F. At each temperature level, a tab of 304 stainless steel was maintained at about 1200 F while immersed in the sodium for about 100 hours. After each run, the tabs were removed, new tabs were inserted, and the bulk temperature adjusted to the next level. The stainless steel tabs were analyzed metallurgically for carbon content. The corrected results are shown in Table III.

Refinements have been made both to the equipment and the operating procedures. New hot finger tests will be conducted using only two temperature parameters -- 500 F and 800 F. These tests should provide more meaningful data than the first tests.

## 2. Longer Range Carbon-in-Sodium Program

The tests described above are directed toward the immediate problem at hand; that is, removal of the carbon contaminants that were introduced into the sodium during the 1000 F preoperational testing. In addition to these tests, APDA is undertaking another testing program to study the carbon-in-sodium problem in relation to nuclear operation of the Fermi plant. The objective of this program is to provide an understanding of the problem that is sufficient for establishing adequate operating procedures of monitoring and corrective actions with respect to carbon contamination.

### B. Hot Trap

A permanent hot trap has been installed in the cold trap room. After some of the experimental work now going on is completed, decisions will be made concerning the optimum operating conditions for this hot trap.

The first temporary hot trap was cut apart and analyzed for carbon content after about 300 hours of operation, on the assumption that some carbon had been absorbed in the stainless steel foil. To improve the permanent hot trap, surveillance tabs have been installed which will show the carbon content of the trap without the need for cutting up the trap.

### C. Plugging Indicator

The plugging indicator originally installed in the primary sodium service system had orifices 0.052 inch in diameter. As indicated in Section IV-D, this is larger than the flow channels of the fuel, which are 0.040 inch in diameter. Therefore, to preclude any possibility of a particle passing the plugging indicator and later lodging in the flow

TABLE III

## SUMMARY OF EXPERIMENTAL RESULTS OF HOT FINGER TESTS

(Corrected Values)<sup>(4)</sup>

Run <sup>(1)</sup> No.	SODIUM		HOT FINGER	304 STAINLESS FOIL							
	Relative Oxide	Bulk Temp (F)	Temp (F)	Exposure (hrs)	Carbon Content (w/o)				Carbon Pickup (w/o change)		
					Original	Top	Middle	Bottom	Top	Middle	Bottom
6	L	600	1100	95	0.049 <sup>(2)</sup> 0.050 <sup>(2)</sup>	0.055 0.046	0.066 0.046	0.070 0.029	12 -8	35 -8	43 -42
3	L	800	1200	97	0.073 <sup>(3)</sup> 0.077 <sup>(3)</sup>	0.109 0.162	-- 0.133	0.104 0.156	49 110	-- 73	42 103
8	L	800	1130	100.3	0.052 0.063	0.050 0.068	0.052 0.055	0.052 0.064	-4 8	0 -13	0 1
4	L	1000	1200	99.8	0.073 <sup>(3)</sup> 0.077 <sup>(3)</sup>	0.073 0.098	0.094 0.073	0.088 0.109	0 27	29 -5	20 42
5	H	678	1124	95	0.049 0.050	0.061 0.052	0.058 0.046	0.052 0.058	24 4	18 -8	6 16
1	H	815	1200	92	0.073 0.077	0.097 0.127	0.091 0.104	0.085 0.075	33 65	25 35	16 -3
7	H	800	1200	100.3	0.052 0.063	0.054 0.045	0.049 0.054	0.055 0.064	4 -29	-6 -14	6 2
2	H	1000	1114	139.5	0.073 0.077	0.116 0.127	-- 0.100	0.103 0.109	59 65	-- 30	41 -42

(1) Tests were run in this order: Pot No. 1 - 1, 2, 5, 7; Pot No. 2 - 3, 4, 6, 8.

(2) Top values are Detroit Edison's results, bottom values are Detroit Testing Laboratory's results.

(3) Assumed.

(4) Raw data correction: DE's results divided by 1.545; DTL's results multiplied by 1.155.

channels of the fuel, the orifice plate of the plugging indicator has been redesigned. The orifices in the new plate are 0.030 inch in diameter, instead of 0.052 inch. Also, some consideration is being given to the possible installation of a variable orifice plugging indicator now undergoing tests at the Quarry.

#### D. Monitoring Techniques

##### 1. Analysis

The Fermi plant has a permanent sampling station located on the primary sodium service system. Samples of radioactive sodium can be taken from the service system into a hold-up tank and be isolated from the system to permit decay of the radioactive sodium. The sodium sample can then be transferred to a sampler located in an area that is accessible to operating personnel. The sampler can be removed and transferred to an analytical laboratory.

The methods that are currently available for determining the quantity of carbon in sodium of the purity required for reactor applications give the total carbon, elemental carbon, and carbonate content. The available carbon analysis methods are all chemical in nature. There are no physical methods developed such as emission or X-ray spectroscopy. The chemical methods require stringent sample handling techniques, ultrapure reagents, high-purity inert cover gas, ultraclean apparatus, and extra careful procedure.

It is axiomatic that an analysis can be no better than the sample. The chemical analyses of carbon in sodium are carried out on samples of sodium that have been extracted from the sodium system. Due to inherent factors in the analytical methods, the quantity of sodium sample actually analyzed is limited to several grams or less. Samples of this size can be biased to significant proportions by microgram quantities of carbon contaminants introduced by the sample container, by the cover gas, or by handling. If large samples are taken to reduce the effect of trace contaminants, there is the problem of nonhomogeneity due to segregation. Finally, the presence of a solid carbon phase in the sodium being sampled can cause random variation in the actual carbon content in the sample. During the recent high temperature clean-up operation at the Fermi plant, 29 samples of sodium were extracted from the primary system and were analyzed. The elemental carbon values ranged from 10 to 750 ppm. The experience at SRE has been similar. Atomic International (AI) reports that in 20 samples of sodium taken from the SRE for elemental carbon determination, the analytical values ranged from 5 to 236 ppm. In a recent round-robin program sponsored by the AEC on analysis of carbon in sodium, the carbon values reported by the six participating laboratories ranged from 3 to 91 ppm at the 15-20 ppm level; from 7 to 60 ppm at the 60-70 ppm level; and from 16 to 140 ppm at the 90 ppm level. The participating laboratories included GE-APED, AI, NL, duPont, NDA, and Ethyl.

ANDA will perform tests to establish operating procedures for sample preparation, sample handling and transfer, and sample analysis--keeping in mind that there is a permanent plant sampling station already installed. The tests will be directed toward a procedure that is adaptable to sampling and analysis when the system is radioactive. In this regard, tests will be run to evaluate the feasibility of distilling the sodium out of the sample container before the container is removed from the sampling station so as to greatly reduce sodium activity and permit early handling of the sample.

## 2. Stainless Steel Tabs

At the present time, it is planned to have at least one and possibly two surveillance subassemblies in the reactor core. The prime purpose for these subassemblies will be to monitor the effect of fast neutron irradiation on various structural materials used in the reactor. They will also be used to obtain information on the effect of sodium impurities on these materials. It is planned that after a given exposure period, specimens will be removed from the reactor and tested for the effects of irradiation on mechanical properties of the materials. The materials will include all the grades of stainless steel now incorporated in the reactor as well as the nonferritic materials that have been used. These specimens will also be examined to determine the effect of sodium impurities on the material. This could include such things as metallographic examination for carburization, hydriding, et cetera. These specimens will be at about the same temperature as reactor components and would indicate whether any carburization of these materials is occurring.

## VI. CONCLUSIONS

Some of the important conclusions of this report are that:

1. There should be no further outgassing from the plug due to temperature. However, some gas (up to a maximum of 200 cu ft) may be evolved each year due to radiation while operating at 200 megawatts. These gases are expected to be largely hydrogen with lesser amounts of CO and CO<sub>2</sub>.
2. The primary sodium can dissolve about 20 pounds of carbon. Since the expected maximum gas evolution rate will produce only 2 pounds of carbon per year, there should be no crust formed on the sodium.
3. The experiments on methods of removing carbon from sodium are not complete.
4. None of the possible problems that could be postulated as being caused by the offgases appear to be serious.

5. Experiments are under way to provide information needed to establish high-temperature, long-term operating criteria for Fermi.
6. The plugging indicator orifice plate has been redesigned to provide orifices of 0.030 inch in diameter instead of the present 0.052 inch. The 0.030-inch hole is smaller than the 0.040-inch openings in the fuel subassembly strainer.

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