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PROGRAM STATUS REPORT: SAMPLING OF
RADIOACTIVE SODIUM FROM FFTF

K. C. Knoll

F. A. Scott

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PROGRAM STATUS REPORT: SAMPLING OF
RADIOACTIVE SODIUM FROM FFTF

K. C. Knoll and F. A. Scott

ABSTRACT

A technique for sampling the coolant sodium of the Fast Flux Test Facility for impurity analysis is demonstrated. It overcomes the problems presented by the reactivity and radioactivity of the sodium.

The sample tubes are installed, checked for leaks, filled with sodium and sealed by crimping. The samples are cooled rapidly and uniformly to minimize impurity segregation and removed from the sampler. All these operations are performed remotely by using master-slave manipulators. Sodium flow was regulated and the temperature maintained at the desired levels.

Analysis of sodium taken from three sections along the sample tube gave no evidence of longitudinal segregation of oxygen. Sodium sealed in tubes by crimping showed no detectable leakage of air through the crimps after as much as 35 days of storage in air.

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PROGRAM STATUS REPORT: SAMPLING OF
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K. C. Knoll and F. A. Scott

INTRODUCTION

The primary objective of the FFTF is to test fast reactor fuels in prototypical environments. A part of this environment is the sodium coolant. To assure meaningful test results, the quality of the coolant must be continuously monitored and controlled. Information concerning trace impurity levels in the sodium will be obtained from in-line instruments and chemical analysis. Chemical analysis will be essential for instrument calibration, for the measurement of elements and chemical species not monitored by in-line instruments, and as a backup in case of instrument failure or malfunction. Sampling will also provide for archive samples which will permit "postmortem" studies of extraordinary conditions.

The acquisition of a valid representative sample is a prerequisite to chemical analysis. Obtaining such a sample of sodium from a large reactor inventory is quite difficult. At least three major problems are present:

- The sodium is extremely reactive chemically and must be rigidly protected from the atmosphere at all times.
- The sodium is highly radioactive due to the presence of sodium-24; thus, all sampling operations must be performed remotely with considerable biological shielding between the sodium and the operating personnel.
- The sample size is, of necessity, very small relative to the inventory so assurance that the small sample is representative of the whole is difficult.

This report describes a first-generation removable-section bypass sampler designed to overcome these problems. It discusses the examination of the samples obtained with the sampler and outlines an additional development effort based on the information gained.

SUMMARY AND CONCLUSIONS

Remotely performed sampling of sodium coolant using a removable section bypass sampler has been demonstrated to be entirely feasible. With the equipment described, sample tubes were installed, checked for leaks, filled with sodium, sealed by crimping, cooled by air quench, and removed from the sampler by use of master-slave manipulators. Samples were cooled rapidly and uniformly to minimize impurity segregation. Sodium flow through the samplers could be controlled and measured and the temperature of the sodium maintained at loop temperature.

The fittings used to connect the sample tube to the sodium line proved adequate for some 30 changes. However, only one type of fitting has been investigated; others might be found more suitable.

The samples taken were uniform in impurity concentration. Analysis for oxygen of sodium taken from three sections along the sample tube gave no evidence of longitudinal segregation. Sodium in tubes sealed by crimping at one end showed no noticeable leakage of oxygen through the crimps after as much as 35 days storage in air.

A program of future work is outlined.

DISCUSSION

ANALYTICAL SAMPLING CRITERIA

Certain criteria must be met by a sodium sampling system if the samples are to be valid for analytical purposes. These are:

- The sampler must provide a representative sample of the system. This should be done by sampling at system temperature and as close as possible to the point of interest.
- The sampling procedure must maintain the integrity of the sample and of the system by: 1) preventing contamination from the sampler and other sources, and 2) preventing segregation or by providing means for handling segregation if it occurs.
- The sampler or sampling procedure must not affect the system by introducing significant impurities into the system.
- The sampler must take samples of adequate size.
- The sampler should be simple.

REACTOR SAMPLING CRITERIA

A sampler for the FFTF primary sodium must meet the following additional criteria:

- The sampler must be remotely operable.
- Rapid sampling should be possible.
- The sampler should be useful for all analysis (both elemental and species) that might be necessary.
- Indefinite storage of sample container should be possible.

With these criteria in mind, a review of candidate sampling methods was completed in 1967.⁽¹⁾ The removable section bypass loop technique was found to be best suited because it closely meets all of the listed criteria.

DESCRIPTION OF SAMPLER

The sampler was built to take two sample tubes, each heated by a heater mounted in the hinged door of the furnace (Figure 1). Originally, both sample tubes were U-shaped but after a short use, the upper furnace was altered to take an L-shaped tube. The sample tubes were 1/2-inch OD stainless steel or nickel tubing with an 0.035-inch wall. The length of the "L" and of each arm of the "U" was about 30 inches. As-built drawings are included in the Appendix of this report.

DESCRIPTION OF SAMPLER INSTALLATION

Sodium was supplied by a sodium loop located just back of the sampler. A schematic diagram of the piping is given in Figure 2. The valve MV2 in the bypass between inlet and outlet lines was used to control the flow through the sampler. The sodium flow to each sampler passed through an electromagnetic flow meter and was controlled by air-operated bellows seal valves on the inlet and outlet line. All lines were trace heated and each heated section had a thermocouple on it.

The sampler was installed in a sheet metal enclosure 5 × 7 1/2 × 9 feet high with a recessed window to simulate a lead glass viewing window. Two extended-reach master-slave manipulators were mounted through the front wall.

Each sampler was provided with a control panel at the operators station with read-outs for flowmeters and thermocouples. Controllers for the trace heaters and for the air operated and solenoid valves were also located on the consoles.

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FIGURE 1. Removable Section By-Pass Sampler

SAMPLE COOLING

The sample tubes were cooled by an air quench after a sample was taken. In the U-tube this was provided for by a tube with holes in top and bottom running down the center of the "U". The L-shaped sampler had a perforated quench tube running along its length just back of the sample tube. These quench tubes could be fed with either nitrogen or compressed air, and flow was controlled with solenoid valves.

FIGURE 2. Flow Diagram of Removable Section By-Pass Sampler

Uniform cooling of the sample was necessary to reduce longitudinal segregation. A series of observations were made to determine the uniformity of the cooling of the "L" sample tube with the air quench. A sample tube was installed with 12 thermocouples spot welded to it (Figure 3). Output of the

FIGURE 3. Location of Thermocouples on Sample Tube

thermocouples was read out on a data acquisition system capable of reading all twelve every second. Sodium was passed through the sampler at 600, 800 and 1000°F. When the temperatures were in equilibrium, the oven door was opened and the quench air turned on immediately. The first cooling profiles showed rapid but nonuniform cooling, so the quench tube was altered to obtain a more uniform rate by lengthening the end nearest the outlet and by enlarging or drilling other holes near sections which showed too slow cooling. Figure 4 is a cooling profile of the hottest and coolest portions of the sample tube before alterations were made in the quench tube. Figure 5 is a similar plot made after the quench tube had been altered. In Figure 4, the sodium was all frozen in 42 seconds; Figure 5 in 54 seconds. The approximately 50°F spread between the lines was an apparent temperature difference rather than a real one. It arose from differences in thermocouple calibrations and attachment rather than from actual difference in temperature. This was indicated

FIGURE 4. High and Low Temperature Points on Sample Tube Before Quench Tube Alterations

FIGURE 5. High and Low Temperature Points on Sample Tube After Quench Tube Alterations

in that the maximum and minimum temperature readings did not occur at the ends of the tubing section. Also, the apparent temperature differential existed at time 0 when a relatively high sodium flow existed in the system. Because of the heat capacity of the sodium, little or no temperature differential was possible under those conditions.

A second calibrated thermocouple was sequentially attached to each of the thermocouple points while sodium was circulating and the temperature measured. All measured values were within the expected precision of the measurement system $\pm 5^{\circ}\text{F}$.

FITTINGS FOR REMOVABLE SECTION

Sample tubes were connected to the sodium lines with union-type connectors. The female fitting was welded to the sodium lines and the male to the sample tubes. The original U-shaped sampler had a clamp device to hold the connectors together but regular nuts were used on all sample tubes during later work. A new gasket was used each time a sample tube was installed and invariably formed a leak-tight seal the first time. However, on several occasions after continued use, leaks developed after the sampler was up to the loop temperature and sodium circulation had been in progress for some time. Figure 6 shows the oxide build up of a typical leak.

A combination of conditions could have caused these leaks. The gasket sealing area may have become corroded by the presence of hot liquid sodium and sodium oxide. As the connector was tightened down cold, the seating was enough to keep the joint from leaking during the leak check, but when the system was heated, the nut relaxed and allowed the gasket to lose its contact to parts of the corroded seating area. Thus, the system, although leak tight when cold, may permit a sodium leak when hot. From the limited experience gained in this work,

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FIGURE 6. Sodium Leak at Connector

it appears that the female fitting should be replaced after being used about 30 times. If a tubing section containing the fittings is attached to the system with similar fittings, it should be possible to take 900 samples before requiring the welding of a new fitting.

HELIUM LEAK CHECKING

Before permitting sodium to flow through the sample tube, it was necessary to assure that all connections were leak tight. In the U-shaped tube, this was done by evacuating the tube with a helium leak detector through a side arm welded into the tube and extending out of the bottom of the furnace. When helium was sprayed around the joints, any leaks present were found and corrected. After leak-checking, the side arm was sealed with a cap. The upper sampler had a device built in to eliminate the use of a side arm on the sample tube. As can be seen in Figure 1, a side arm left the sodium inlet line just before the sample tube and, passing through a stainless steel bellows valve (MV1), led to an evacuation pot which was evacuated with the rough pump of a helium leak detector. In operation, a sample tube was installed and the sodium lines leading from the sample tube to the evacuation pot were heated to melt the enclosed sodium. When valve MV1 was opened, the sodium was withdrawn from the tubes into the evacuation pot and the sample tube was evacuated. Helium was then sprayed on the joints and any leaks detected and corrected. Before sampling was started, MV1 was closed and the sodium in the side arm allowed to freeze.

CRIMP SEALING OF SAMPLE TUBES

One of the earliest problems in the sampling program was a method for sealing the sample tubes to keep air from contacting the sodium. The most logical way was to crimp the tube shut at each end while it was still on the sampler. To

do this, a hydraulically operated guillotine was purchased which takes replaceable jaws and can develop a 15 ton thrust. Several jaw configurations were tried; the best one is shown in Figure 7. With these jaws, it was possible to seal annealed stainless steel tubing but not consistently. When #200 nickel tubing was crimped, it would generally seal. However, when the tubes were full of sodium, neither nickel nor stainless steel would form a hermetic seal every time. Therefore, it was decided to use stainless steel tubing and double crimp each end; the sodium enclosed between the two crimps was depended upon to prevent the ingress of air to the sample. As a test of the validity of this method, two pieces of nickel and one of stainless steel tubing full of sodium were capped at one end and double crimped at the other. After storage in air, consecutive one-inch samples were cut off for the oxygen analysis, the first one being taken as close to the crimp as possible (about 3/4 inch). The results in ppm oxygen are given in Table 1 and demonstrates that no leakage has occurred.

FIGURE 7. Crimper Jaws

TABLE 1.

OXYGEN CONTENT OF SODIUM SAMPLES
TAKEN CONSECUTIVELY FROM CRIMPED END

<u>Tubing Material</u>	<u>Days Stored</u>	<u>Section Analyzed ppm Oxygen</u>			
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
SS	21	11	9	6	
Ni	21	30	18	19	
Ni	35	18	18	20	21

SAMPLING EXPERIENCEINITIAL OPERATION OF SAMPLER

Soon after start-up, difficulties were encountered with the sampler as built. The clamp used to compress the fittings did not permit accurate seating of the gaskets, and it allowed leaks. The presence of the quench tube between the legs of the U-shaped sample tube prevented crimping in place. Sealing the side arm tube with a cap after leak-checking proved difficult to do with manipulators. Also, we were unable to check its leak tightness. Only three U-tube samples, all with contact operation, were taken.

MODIFICATIONS

To correct these problems, the upper sampler was changed to use the L-shaped tube with the quench tube behind it and the evacuation pot connected to it. Nuts were put on the sample tube during fabrication and were used to fasten the flanges together instead of the original clamps. An open-end wrench was necessary to tighten these because an impact wrench could not be used. Roller ratchet wrenches proved impractical. A cutoff, open-end box wrench with special grips to fit the manipulator jaws proved to be the most satisfactory. Gaskets

were prefitted to the male flange on the sample tube and were held in place by slightly dimpling the outside top of the sealing diameter section with a center punch.

To permit handling the crimper with the manipulators, a commercial counter balance was mounted on a trolley inside the enclosure. This reduced the effective weight of the guillotine to about 5 pounds and permitted it to be positioned easily with the manipulators.

The extended-reach manipulators were capable of reaching any part of the sampler necessary and performing all necessary operations. Thus, completely remote operation of the sampler was made possible with no access to the enclosure required.

REMOTE OPERATION PROCEDURE

The sample tube with the gaskets in place is picked up with the manipulators and put in place on the sampler (Figure 8). While the tube is held in place with one manipulator, the nuts are tightened down with the modified wrench fitted to the manipulator jaws (Figure 9). The connections are checked for leaks using the system described under Helium Leak Checking on Page . If no leak is detected, the furnace door is closed and latched, the system is preheated up to 300°F, and sodium flow is started. The desired temperature and flow are established and maintained for the sample pre-conditioning time. At the end of the sampling run, the outlet valve is shut off; the oven door is opened; and the crimper is brought into place near the outlet end of the removable section (Figure 10). The sample tube is crimped next to the fitting and then again several inches away (Figure 11). The guillotine is then moved to the inlet end of the sample tube which is crimped about 3 inches from the end and then again next to the inlet fitting. The inlet valve is

closed and the quench is turned on. Less than 2 minutes are consumed in the crimping, thus, the temperature does not drop more than 100°F. When the sample tube and inlet and outlet lines have cooled to 200°F, the nuts are removed with the manipulator-operated wrench. The sample tube is removed with the manipulators by pushing down and pulling outward to break the solidified sodium column. The procedure has proved to be simple and a number of samples have been taken by using it. Figure 12 shows a crimped sample tube after removal.

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FIGURE 8. Placing Sample Tube in Position

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FIGURE 9. Nut Tightening

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FIGURE 10. First Crimp at Outlet End

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FIGURE 11. Second Crimp at Outlet End

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FIGURE 12. Crimped Sample Tube

SAMPLING RESULTS

A series of twenty-four samples were taken by using this procedure to study oxygen segregation. Sections of each sample at the inlet, center, and outlet were analyzed for oxygen; a fourth section was analyzed for carbon. The first 10 samples were taken during normal sodium loop operation with the cold trap about 350°F. The rest of the samples were taken with the cold trap at higher temperature. At the time each sample was

removed, a plugging run was made by the loop operator. Table 2 lists the analytical results obtained from these samples. Sample 1 results were omitted because of questionable validity.

The difference of oxygen content at the inlet, center, and outlet of the sample tube was not greater than the variation expected in the analysis. This indicated an absence of longitudinal segregation of oxygen at these levels.

The reproducibility of the samples was excellent when the cold trap temperature was closely controlled (Samples 11 to 24). The standard deviation of the results at each oxygen level was never greater than 3 ppm. The carbon analysis results were more scattered which was to be expected at those low levels of carbon.

The consistency of the sampling was further shown by comparison with the plugging meter results. Both showed a constant oxygen content at steady cold-trap temperatures and an increase in oxygen as the cold-trap temperatures were increased. The numerical oxygen values calculated from the plugging meter data were consistently lower than the analytical values, probably because of the particular curve used to calculate the plugging meter results.

Although the sample tubes were crimped while the sodium was at temperatures as high as 800°F; no crimps were pinched open; thus, no sodium escaped. Although in several cases, the gaskets remained in the female flange when the sample tube was removed, removing the gasket with the manipulators was not difficult.

The inlet, center, and outlet end of the three U-shaped samples taken were also analyzed (Table 3).

TABLE 2
ANALYTICAL RESULTS OF REMOVABLE
SECTION SAMPLE

Sample Number	Temp. Loop Sodium, °F	Cold Trap Temp., °F	Oxygen ppm			Carbon ppm	O ₂ ppm Plugging
			Inlet	Center	Outlet		
1	500	350	-	-	-	0.7	
2	600	350	10	8	13	(a)	
3	600	350	11	11	13	(a)	
4	600	350	7	9	9	0	
5	600	350	11	12	10	2	
6	600	350	10	10	11	18	
7	600	350	4	3	4	4	
8	600	350	8	8	14	2	
9	600	350	13	12	15	6	
10	600	350	(a)	(a)	(a)	(a)	
11	800	400	19	17	17	5	11
12	800	400	28	26	25	5	17
13	800	400	24	24	22	6	19
14	800	400	24	25	26	4	16
15	800	400	25	23	23	7	18
16	800	400	24	25	25	4	17
17	800	400	32	27	28	4	17
18	800	400	24	21	21	4	19
19	800	440	30	32	25	2	25
20	800	440	27	27	26	4	24
21	800	440	29	27	29	4	24
22	800	440	29	28	28	4	24
23	800	440	32	30	35	4	22
24	800	440	28	(a)	(a)	5	25

(a) Not Analyzed

TABLE 3
ANALYTICAL RESULTS OF U-SHAPED REMOVABLE
SECTION SAMPLES

<u>Sample</u>	<u>Sodium Temp., °F</u>	<u>Collection Time, hr</u>	<u>Inlet End</u>	<u>Center</u>	<u>Outlet End</u>
1	600	4	32	14	13
2	600	4	14	13	19
3	800	5	48	31	34

FUTURE WORK

Based on the experience gained with the sampler described in this report, design work has begun on a second, more compact, model. The following are descriptions of other tasks that need to be completed in the sampler development program:

Sample Tube Connectors

Sample tube connectors are necessarily subject to very severe conditions: repeated make-up, rapid and extreme thermal cycling, hot flowing sodium and the presence of sodium oxide. They must be able to seal consistently and be capable of convenient remote separation. Only union connectors have been evaluated. While they have worked satisfactorily, they have limitation and other types should be evaluated.

Leak Checking System

The present method of checking the sample tube connections for leaks is both time-consuming and cumbersome. Experience gained during this work indicated that generally the union sealed the first time. Possibly, as experience with the connectors increases, leak checking may be found unnecessary.

Integrity of Crimp-Seals

Present evidence indicates that the double crimp seals are sufficient to prevent atmospheric contamination of the sodium for a limited time at least. Their suitability for long-term storage of archive samples needs further study. If the use of the sodium hermetic seal proves satisfactory, the crimper jaws probably should be redesigned to reduce possible tube pinching from too much pressure. Also, more work on the possibility of a true cold weld is recommended.

Segregation of Impurities

Longitudinal segregation of oxygen does not appear to be a problem when the samples are taken under the conditions described. Verification that other impurities do not segregate longitudinally is necessary. If any radial segregation is found to occur, ways of minimizing it must be considered.

Sample Labeling

Some way must be devised to identify samples as they are taken. The sample tube will need to be labeled at several places along its length so cut sections can be identified. The method must provide a mark that is permanent, legible, unimpaired by handling, and not destroyed by heat. Presently, the best candidate seems to be some forms of electrochemical marking.

Decreasing Size of the Sampler

Because FFTF sampling requirements call for six bypass samplers in one enclosure of limited size, careful sizing and placement of the samplers will be required. The sampler sizes must be acceptable for both the analyses required and the enclosure; thus, the design needs to be a minimum-maximum compromise between these two requirements. Also, the samplers must be in such a position that they can all be operated with the master-slave manipulators.

Provision for Maintenance

Because of the high radiation fields that will be present in the FFTF sample enclosure, no contact maintenance will be possible. Remote maintenance will be very difficult because of the necessary compactness of the equipment to accommodate the size limitations. The best solution for maintenance appears to be "unitized" sampler construction so that the entire sampler can be disconnected from the sodium lines and replaced by another. After time is allowed for the radioactivity to decay, the disabled sampler can be repaired under contact conditions.

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ACKNOWLEDGMENT

The authors wish to express appreciation to E. L. Osborne for his help in the operation of the sampler.

APPENDIX

SODIUM SAMPLER DRAWINGS (AS BUILT)

Drawing No. H-3-28965

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