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EXAMINATION OF THE
ENRICO FERMI SODIUM COLD TRAP

Report of Work Performed Under

Contract No. AT(30-1)-3186

With the New York Operations Office, USAEC,

For the Division of Reactor Development and Technology

U. S. Atomic Energy Commission

November, 1965

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ABSTRACT

Under AEC contract AT(30-1)-3186, Westinghouse Atomic Power Division sampled and analyzed the contents, and evaluated the performance, of a sodium cold trap removed from service at the Enrico Fermi Atomic Power Plant. The trap, a 500 gallon vessel packed with stainless steel wire mesh, operated for 3700 hours in the primary sodium system. It was removed from service prematurely, so that a new one could be installed before nuclear operation of the plant.

An effective method was developed, and employed, to remove fifty-five representative sodium-mesh samples from selected regions of the non-radioactive cold trap. Methods for determining the impurities of interest in the samples from the trap were adapted from procedures described in the technical literature. Based on the chemical analyses of the trap contents, less than 20% of its designed oxygen removal capability was realized. Most of the contaminants were concentrated in the lower mesh region. The trap contained an estimated 50 pounds of oxygen, 0.9 of a pound of carbon, and smaller amounts of carbonate, hydrogen, and nitrogen. Spectrographic analyses for metallic impurities revealed high concentrations of copper (up to 360 ppm) and iron (up to 165 ppm).

Although a section of the innermost cylindrical wall had partially collapsed and a portion of the lower mesh had been displaced, the cold trap operated satisfactorily. There was no evidence of flow maldistribution.

1. SUMMARY

1.1 Introduction

Cold traps are employed to remove oxygen from sodium, and they have performed satisfactorily in many sodium systems. None of the cold traps removed from service before the Fermi trap were examined in detail. The availability of the Fermi primary sodium system cold trap offered an opportunity to develop sampling and analytical procedures for the detailed examination and evaluation of a trap that had been employed to purify a large sodium system.

1.2 Program

In March, 1963 Westinghouse Atomic Power Division contracted with the Atomic Energy Commission to examine the Enrico Fermi cold trap. The general objectives of the contract, listed below, were achieved.

1. Develop sampling and analytical techniques suitable for cold trap examination.
2. Determine the chemical and physical composition and spatial distribution of the trap contents.

3. Evaluate the operational performance of the trap, and correlate the experimental results for the development of sodium cold trap design criteria.

The cold trap was transferred to the Westinghouse Waltz Mill site. Sampling and analytical techniques were surveyed. Two methods of examining the trap were considered: (1) cored samples from the trap, and (2) melting and draining the contents of the trap and disassembling the trap. The second technique would have disturbed the oxide deposits on the wire mesh, redistributed some of the oxide during remelting, and prevented physical examination of the sample material in place. Therefore, only the coring method was investigated thoroughly. After many trials the following suitable sampling technique was developed. First, access holes were drilled in the shell. Then, a steel tube with a sharpened tip was driven into the cold trap to take representative sample cores. Fifty-five samples, one inch in diameter and up to twenty-seven inches long, were removed from the trap.

The trap, the sample cores, and the mesh in the core samples were physically examined. This examination revealed a gross correspondence between the appearance of the sample cores and impurity level, a partially collapsed inner wall, and partially displaced mesh in the trap.

Appropriate methods were selected, modified, and used for the chemical and spectrographic analyses of the samples removed from the

trap. A literature survey of chemical analyses resulted in an annotated bibliography described in report WCAP-2540.

Westinghouse and three other laboratories performed round-robin analyses to determine the variations in results that could be expected from current state-of-the-art analyses of sodium samples containing a large amount of oxygen.

Samples from selected elevations were analyzed for total carbon, carbonate carbon, oxygen, nitrogen, hydrogen, metallic impurities, and mesh content. Based on the results of these analyses, the trap was estimated to contain about 50 pounds of oxygen, 0.9 of a pound of carbon, and smaller amounts of nitrogen, hydrogen and metallic impurities.

To evaluate the performance of the cold trap, its operating history was reviewed, and the amount of accumulated oxygen was estimated on the basis of plugging temperatures. Samples of Fermi primary sodium, taken before and after cold trapping, were analyzed chemically and spectrographically to reveal the purification achieved by the cold trap. Chemical and spectrographic analyses of the cold trap contents revealed the actual impurities removed from the system.

The analysis of the vertical distribution of impurities showed that most of them were concentrated in the lower third of the mesh region. The horizontal distribution of impurities and the mesh con-

centrations were relatively uniform at each trap elevation that was sampled, indicating that there was no flow maldistribution.

In March, 1965 Westinghouse completed the experimental work and disposed of the cold trap and its contents.

1.3 Conclusions and Recommendations

The Fermi cold trap removed oxygen and other contaminants from the primary sodium. These contaminants were concentrated in the lower mesh region of the trap. The oxygen concentration in the bottom of this mesh region approached the maximum theoretical value of 25.8 w/o, and the rest of the trap contained very little oxygen. The trap realized only 17% of its design capacity for oxygen removal before it was removed prematurely from service in the Fermi system. If the trap had remained in service it probably would have achieved its design capacity based on the relief mechanism described below.

The following theory was postulated to explain the partially collapsed inner walls and the displaced mesh. The trap was plugged during one period of service, and the resulting pressure buildup collapsed the inner wall of the trap. Displacement of the caked mesh relieved the flow resistance, and allowed the sodium to flow through the trap again.

Based on an engineering analysis of the performance of the Fermi cold trap, some design improvements are recommended. An annular cold

trap with inward radial flow is specifically recommended because of the following advantages: (1) upon entering a trap of this design, sodium is distributed on a large frontal area of mesh for initial oxide deposition and purification; (2) a trap of this design would have better structural integrity than the Fermi trap, eliminating the problem of wall deformation.

A mechanism was postulated for sodium oxide precipitation based on the results from the examination of the Fermi cold trap.

To learn more about the performance of cold traps, the following programs should be performed:

1. A comprehensive program of chemical analyses to identify the specific compounds involved in the precipitation and concentration processes that occur in cold traps.
2. A study to obtain accurate solubility data for oxygen, hydrogen, nitrogen, and metallic impurities in sodium.
3. A physical chemistry program to determine the mechanisms of oxide precipitation and accumulation on a metal surface.
4. An investigation of the effectiveness of cold traps for removing fission products from sodium.
5. Examination of other sodium cold traps, already removed from service, including some traps that contain fission products.

2.0 BACKGROUND AND OBJECTIVES

The use of sodium as a high temperature heat transfer medium is increasing, particularly in advanced fast nuclear reactors. To prevent corrosion of containment materials and fuel cladding, the sodium purity must be kept at a high level. Oxygen is the most important impurity to be controlled, and cold traps are normally used to remove oxide from sodium. A cold trap cools the ~~sodium~~ below the saturation temperature for sodium oxide in sodium, causing the sodium oxide to precipitate and deposit in the trap. Cold traps are usually packed with stainless steel mesh that acts as a nucleating medium for the precipitate. Most cold trap designs have been based on empirical data for flow path, geometry, oxygen buildup, capacity, mesh size and density, and removal of impurities other than oxygen. Cold traps have been removed from service at several locations including APDA, EBR-II, ANL, SRE and Hallam.* However, none of these traps were examined for identification and distribution of impurities before examination of

* APDA - Atomic Power Development Associates
EBR-II - Experimental Breeder Reactor II
ANL - Argonne National Laboratory
SRE - Sodium Reactor Experiment
Hallam - Hallam Nuclear Power Facility

the Fermi cold trap was started. Methods existed for analyzing impurities in sodium, and, with some changes, these methods were applicable to the cold trap samples. The cold trap in the Enrico Fermi primary sodium system was removed from service so that a new unit could be installed for radioactive operation. The availability of this partially expended cold trap offered an opportunity for a detailed examination and evaluation of a cold trap which had been used to purify a large sodium system.

In March, 1963 Westinghouse Atomic Power Division contracted with the Atomic Energy Commission to perform an experimental and analytical study of the Fermi cold trap with the following objectives:

1. Survey, develop, and select sampling and analytical techniques suitable for cold trap examination.
2. Prepare a program for sampling, examination, and analysis of the cold trap contents.
3. Determine the chemical and physical composition, and spatial distribution of the trap contents.
4. Evaluate the operational performance of the trap with particular regard to flow maldistribution.
5. Correlate the experimental results for the development of sodium cold trap design criteria.
6. Dispose of the sodium cold trap and its contents.

3. DESCRIPTION AND OPERATION OF THE COLD TRAP

The general characteristics and operating history of the Enrico Fermi primary sodium cold trap are described below.

3.1 Description of the Cold Trap⁽²⁾

The cold trap, shown in Figure 1, was a jacketed, 500-gallon, stainless steel vessel, approximately 4 feet in diameter and 7.5 feet long, filled with 11-mil diameter stainless steel mesh packed to a density of 12 lbs. per cu. ft. The mesh region consisted of loosely woven wire cloth rolled into a tight bundle. The mesh bundle and the cold trap had the same vertical axis. The vessel was rated for an internal pressure of 60 psi, and an external pressure of 15 psi, at 1000°F. It carried the ASME seal for an unfired pressure vessel. The maximum sodium flow rate was 100 gpm, and the usual operating flow rate was 50 gpm.

The flow of sodium was as follows. Before entering the cold trap, the sodium passed through the shell side of an economizer (upstream of the trap) where its temperature was lowered, but not enough to initiate precipitation. Upon entering the trap, the sodium was cooled further by NaK (a sodium-potassium alloy) in the jacket. The nominal NaK flow rate was 31,600 lbs. per hr., and the

ENRICO FERMI PRIMARY COLD TRAP

Figure 1

corresponding inlet and outlet NaK temperatures were 200°F and 300°F. During the operation of the trap, the NaK flow rate and/or inlet temperature were adjusted to achieve the desired sodium temperatures. Because the sodium velocity in the 3/4-inch annulus was relatively high (0.15 fps), the precipitated oxide was carried along to the bottom of the trap. The velocity of the sodium decreased to 0.01 fps as it flowed up through the mesh region, and the oxide was deposited on the mesh wires. On leaving the cold trap, the sodium passed through the tube side of the economizer, where it was heated by the incoming sodium. The operating temperature of the cold trap could be varied between 250°F and 900°F by altering either the flow rate, or the temperature, of the NaK in the cooling jacket.

A 5 cu. ft. space was provided at the top of the trap for expansion of the sodium in the event the trap was isolated during heating to operating temperature. An 80 psig rupture disc was located at the top of the tank for relieving overpressure. Design data from the Fermi cold trap operating manual is given in Appendix G.

3.2 Operating History (3)

The cold trap was installed in 1959 and was used during the initial charging of the plant and subsequent reactor flow tests. The trap's operating history is shown in Figure 2. The trap operated for 3700 hours at an average flow rate of 50 gpm. Thus, 80 million

OPERATING HISTORY OF FERMI SODIUM COLD TRAP

Figure 2

pounds of sodium passed through the trap, which is equivalent to 160 flushes of the primary system. During the period of service experienced by the trap, the main loop temperature was maintained between 400°F and 500°F, except during two tests when it was raised to 750°F and to 1000°F, respectively.

There were two causes for the higher plugging temperatures shown in Figure 2. The solubility of sodium oxide in sodium increased with higher sodium temperatures; and the "reactornaut" maintenance program, performed on the drained system in late 1962, increased the contamination of the system. The plugging temperature for sodium containing sodium oxide was usually less than 240°F, except during the 750°F and 1000°F tests, when it rose to 325°F and 550°F, respectively. Another exception occurred in January 1963, when the plugging temperature reached 425°F while the reactor vessel was at 445°F. During the 1000°F test, decomposition of the organic binder for the graphite material in the reactor vessel rotating plug, released carbonaceous and hydrogenous materials to the cover gas. The primary sodium became contaminated with the carbon and hydrogen impurities because it was in contact with the cover gas. Just before the cold trap was removed from service on February 1, 1963 the sodium was at 430°F while the plugging temperature was 265°F.

4. COLD TRAP SAMPLING AND CHEMICAL ANALYSIS METHODS

Sodium cold traps have been removed from service at APDA, EBR-II, ANL, SRE, Hallam and other locations. However, none of these traps were examined for identification and distribution of impurities before work started on the Fermi cold trap. The available literature revealed no method for sampling sodium with mesh. Thus, two basic methods were considered for examining the Fermi cold trap: 1) taking cored samples from the trap; and 2) melting and draining the trap contents, and then disassembling the trap. The second method would have disturbed the oxide deposits on the mesh, redistributed some of the oxide during melting, and prevented physical examination of the sample material in place. Therefore, only coring methods were investigated.

The following experimental procedures had to be selected and developed to analyze and evaluate the trap:

1. A method of obtaining suitable, representative samples; and
2. Procedures for performing chemical analyses.

This section summarizes the selection and use of these procedures. The development of the sampling procedure is described in detail in

Appendix A. The chemical analysis procedures are described in detail in Appendix B.

4.1 Sampling

4.1.1 The Sampling Problem

To analyze and evaluate the cold trap, suitable samples of its contents had to be recovered. To be suitable, a sample should be:

1) physically and chemically representative of the material from which it was taken; 2) free of contaminants due to handling; and 3) be of sufficient diameter for gross physical examination. In addition, the precise location of the sample in the trap should be identified.

4.1.2 Sampling Method Used

The difficulty of obtaining suitable sample cores of sodium and mesh from the trap was underestimated. The mixture of sodium and mesh was soft and adhesive, and exhibited marked cold flow characteristics. Tools that worked well on sodium or on mesh separately did not work well on the mixture of sodium and mesh. A variety of tools and techniques were tried, including pot metal mockup, hole saws, cryogenic temperatures, wax, oscillating scissors, various drills, a harpoon, and a driven tube (described in detail in Appendix A). Since the driven tube method produced suitable sample cores, it was adopted for sampling the cold trap. This method is described below.

A 1-1/8 inch O.D. x 1 inch I.D. x 36-inch long sharp-tipped, 30⁴ stainless steel tube was hammered into the contents of the cold trap and a sample core was extracted in an inert atmosphere. The design of the tube tip is shown in Figure 3. Stainless steel, rather than carbon steel, was used because it was less apt to contaminate the sample core. A new cutting tip was tack-welded to the end of each tube and the weld was ground smooth, before each sample core was removed.

Because of the reduced I.D. at the cutting end of the tip, the radial clearance between the sample and the sampling tube was only 21 mils. This proved sufficient, however, since samples over two feet long could slide freely through the tube. The reduced I.D. section of the tip was 0.281-inches long, thus providing enough surface to twist the sample free just before removing it from the trap. In fact, some samples were cut so cleanly that when the tube was removed, the two-foot samples remained in the trap. In these cases, retrieve a complete core, a solid, 1/16-inch diameter, stainless steel rod was inserted between the inner wall of the tube and the sample to lock them together.

A five to ten pound sledge hammer proved to be more convenient than an electric hammer for driving the tube. A 1-inch bolt with a large head was placed in, and taped to, the trailing end of the sampling tube. The bolt provided a striking surface for the hammer,

CUTTING TIP FOR SAMPLING TOOL

Figure 3

thus preventing distortion of the sampling tube. The tube was driven into the trap, about one to two inches at a blow, with a five-pound sledge hammer. In an attempt to ease movement of the sample, a vacuum was applied to the inside of the tube. Although this technique was tried two or three times, its results were inconsistent and it was discarded. During each sampling and after every few hammer blows, the accumulated tool penetration and the sample length were measured by inserting a ruler in the trailing end of the sampling tube. The distance between the trailing end of the tube and the wall of the trap was measured in the same way. Generally, the tool penetration corresponded to the increase in the length of the sample. This did not always occur, however; sometimes the sample was extracted too soon. In such cases, a new tube was driven into the hole to complete the job. When a sample was taken, adjacent holes were plugged with steel tubes to prevent material movement.

The mesh region of the trap consisted of loosely woven wire cloth, tightly rolled to a diameter of four feet and having the same vertical axis as the trap. All samples from the mesh region were taken in the radial direction. To assure a clean break, the sampling tube was rotated three times before being removed. No samples were taken vertically from the mesh region because they could not be obtained without severely disturbing the mesh-sodium relationship.

Before the trap was sampled by the method described above, safety precautions were established. The outer shell of the trap was removed first. When the trap proper was removed from service, the NaK annulus was drained and capped. However, some material remained on the walls, and the bottom drain pipe was suspected of containing about a cupful of NaK.* Since it was desirable to remove the NaK shell for safety and to facilitate sampling, the following preparations were made.

The cold trap was housed in a temporary, roofless room constructed of 1/16-inch mild steel sheets and suitable angle-iron supports. (See Figures 4 and 5). The floor was a 12 foot x 12 foot seal-welded pan with an 8-inch high border. (If all the sodium in the trap had drained into the pan, the pan would only have been filled to a depth of less than six inches.) The walls were 10 feet high, with an open maze type of access at three corners of the room. This arrangement could safely contain any possible splashes of sodium, yet permitted easy access and exit. Three fans were installed in the wall next to the cold trap to exhaust smoke in case of fire. A 350-pound, MET-L-X extinguisher with a 50-foot hose was parked

*NaK exposed to air on a dry day will spark and flicker gently; on humid days, pronounced sparking will occur. When NaK or potassium is exposed to air or oxygen, potassium superoxide (KO_2) is formed. KO_2 can explode if exposed to a carbonaceous material such as oil or alcohol, and is sensitive to percussion.

Figure 4 Outside View of Isolation Room for Cold Trap

Figure 5 Interior View of Isolation Room For Cold Trap

outside the room for use either through the personnel doorways or over the top of the walls. Ten 100-pound bags of soda ash were stored just outside the room. Overhead sprinklers, gas lines, and water lines passing by the cold trap were disconnected.

Above the NaK outlet level, the NaK shell was separated from the trap by using 24 manually-operated cutoff grinding wheels. An argon purge, fed into the bottom of the shell, maintained an inert atmosphere during grinding. The gaps were covered with masking tape to conserve gas. The trap was lifted slowly, in one-foot increments (Figure 6) and wiped with dry rags. A surface coating of thin NaK film flickered occasionally when a small area was wiped. The outer surface of the cold trap had a very porous and sandblasted appearance. A thicker NaK film was observed at the bottom of the trap, and one of the centering tabs had a buildup of NaK (Figure 7), which was removed by scraping and wiping. The inside bottom of the NaK shell had a pale yellow coating, apparently of potassium superoxide. This coating did not explode or burn. The shell was moved to a nearby field where it was flushed thoroughly with a fire hose. No explosion occurred, and any fires were masked by the deluge of water.

Masking tape, although normally tenacious, would not stick to the NaK wetted wall, even after the wall had been washed with wet rags. Accordingly, most of the wall surface was hand-ground with disc sanders to obtain an adherent surface for the tape. A flexible

Figure 6 Removing Trap from NaK Shell

Figure 7 Deposits at Bottom of NaK Annulus

plastic film glove box was attached to the wall with tape and filled with argon. This box was pressurized so that any leakage would be outward.

A 1-3/16 inch, high-speed hole saw was used to cut through the 1/4-inch shell and the 1/8-inch inner liner, with a 1-inch IPS tap used to cut pipe threads in both walls. The steel chips were carefully removed to prevent sample contamination.

The sampling tube was inserted through a hole, made by cutting a finger from a glove, and was driven by the electric hammer as shown in Figure 8. (A sledge hammer was used later due to its greater convenience.) After samples were removed, the tapped holes were closed with 1-inch pipe plugs.

A Tygon tube was placed over one end of the sampling tube after its removal from the trap. The sample was then pushed through the steel tube into the Tygon tube, and the ends of the tube were sealed with rubber stoppers and clamps. All samples were similarly packaged to protect them during transfer to the Westinghouse analytical laboratory. Check samples for outside laboratories were prepackaged in Tygon tubes and were placed in steel pipes fitted with Teflon sealed pipe caps. The atmosphere in the pipe and in the Tygon was argon.

The method described above met all of the requirements for

Figure 8 Electric Hammer Sampling

taking a suitable sample. The contamination problem was solved by using an inert gas blanket, a stainless steel tool, and a Tygon transport container. The location of each sample in the cold trap was determined accurately by performing dimensional checks during sampling. The mesh concentration in the samples, plus visual examination, showed the samples were cut cleanly and were representative.

The effectiveness of the adopted sampling procedure was further checked by reacting the sample from Hole No.9-2 (refer to Section 4.1.3) with methanol, to dissolve the sodium, and to reveal the mesh configuration in the sample core. Figure 9 shows the effectiveness of the sampling technique in removing clean-cut samples containing mesh.

4.1.3 Location of Sample Cores in the Cold Trap

Fifty-five sample cores were removed from the trap by the sampling method described in the preceeding section. The length of each sample, its location in the trap, and the date on which it was removed from the trap are given in Table 1. The numbering system used to identify the locations of the samples in the trap is described below.

Figure 9 Mesh Residue from Sample 9-2 After
Methanol Dissolution

Table 1

Identification of Samples Removed From Fermi Cold Trap

Elev.	Half Plane	Sample Number	Date Removed	Length	Elev.	Half Plane	Sample Number	Date Removed	Length	Elev.	Half Plane	Sample Number	Date Removed	Length	Elev.	Half Plane	Sample Number	Date Removed	Length
2	1	1-1	6-25	.5 x 3.3	17	1	9-1	9-17	5.4	24	3	28-1	11-24	14	48	1	49-1	11-18	20
		1-2	6-25	.5 x 1.8			9-2	9-17	6.3			28-2	12-14	12	-5	2	50-1	11-30	24
		1-3	7-6	.5 x 2.5			9-3	9-17	6	36	3	29-1	11-20	18	1	2	51-1	12-1	23
		1-4	7-6	.5 x 1.9			9-4	9-17	6	48	3	30-1	11-13	20	4	2	52-1	12-1	18
52	1	2			11	1	10-1	9-18	1.8	57	3	31-1	12-2	19	48	2	53-1	12-2	19
48	1	3-1	7-7	.5 x 5.3	1	1	11-1	9-17	27	-5	3	32-1	12-18	25	24	3	54-1	12-22	19
		3-2	7-8	.5 x 7	-5	2	12-1	8-27	4.8	1	3	33-1	12-16	25	Outlet Pipe		55-1	1-4-65	27
							12-6	9-17	8										
		3-3	7-10	.5 x 3.5	1	2	13-1	8-25	2.1	2	3	34-1	12-16	25					
-5	1	4-1	7-14	3.3															
		4-2	7-22	3.8			13-2	8-25	3	4	3	35-1	12-14	20					
		4-3	7-24	.5 x 5			13-3	8-25	20										
24	1	5-1	7-15	.5 x .8	4	2	14-1	8-26	22	6	3	36-1	12-14	21					
6	1	6-1	7-16	2	6	2	15-1	8-31	4	11	3	37-1	12-11	14					
		6-2	7-16	1.6															
		6-3	7-17	2			15-2	9-1	18	24	3	38-1							
		6-4	7-17	1.5	11	2	16-1	9-16	27	36	3	39-1	12-18	22					
		6-5	7-17	.5 x .8	36	2	17-1	9-16	20	48	3	40-1	12-21	19					
		6-6	7-22	1 OD x .5 ID															
				x 2	36	1	18-1			57	3	41-1	12-21	23					
		6-7	7-22	2															
		6-8	7-23	.5 x 2.3	Outlet pipe		19	(See 55)		-5	4	42-1	11-25	26					
		6-9	7-24	1.5	Inlet pipe		20-1	1-5-65	14	1	4	43-1	11-14	8					
		6-10	7-28	1.4	Relief pipe		21					43-2	12-7	19					
4	1	7-1	7-30	3.8															
		7-2	7-30	2.4	-5	3	22-1	11-11	16	4	4	44-1	11-30	4					
		7-3	7-31	4.6	1	3	23-1	11-13	19			44-2	12-4	18					
		7-4	7-31	3.8						48	4	45-1	11-25	23					
		7-5	8-11	2.5	2	3	24-1	11-20	23	-5	1	46-1	11-19	19					
		7-6	8-12	pieces	4	3	25-1	11-12	21	1	1	47-1	11-14	15					
		7-7	8-12	4	6	3	26-1	11-19	10			47-2	12-9	6					
48	2	8-1	8-17	3			26-2	12-11	10										
		8-2	8-17	6						4	1	48-1	11-14	10					
		8-3	8-27	6.4	11	3	27-1	11-12	12			48-2	12-7	10					

NOTE: The sample length is in inches.

The diameter is 1" unless shown as .5".

Dates are 1964 unless shown otherwise.

The elevations are in inches above the lowest mesh level.

The first part of the sample number is the hole number. The second part refers to the relative position, with higher numbers being close to the center of the trap.

Figure 10, an isometric, cut-away view of the trap, shows the spatial arrangement of typical sample locations. The developed view of the trap (Figure 11) shows the half-plane location and elevation of every sample removed from the trap. The length, elevation, and half-plane location of each sample used for chemical analysis is given in Figures 12, 13 and 14

Since it was deemed impractical to examine and analyze 100 percent of the trap's contents, a scheme was devised for obtaining representative samples. To determine the vertical distribution of impurities, horizontal samples were taken at various elevations of the trap. The location of the mesh and the sodium flow path indicated the trap might have acted as a filter, with the maximum concentration of contaminants accumulating in the lower mesh region. Therefore, adjacent elevations were selected and sampled in this region, with the remainder of the trap sampled at more widely separated elevations. To determine the horizontal distribution of impurities at each elevation, it was decided to take samples along different radii for the full length of each radius selected, i e , from the vertical axis to the outer shell of the trap. Four equidistant radii were therefore selected and sampled at most elevations

Some half-inch diameter samples were not representative and it was apparent that a one-inch diameter was preferable; therefore all

FERMI COLD TRAP
TYPICAL SAMPLE HOLE LOCATIONS

Figure 10

FERMI COLD TRAP
SAMPLE LOCATIONS

NOTE: HOLE 20 IS LOCATED IN THE INLET PIPE WHICH IS VERTICAL AT THE TOP CENTER OF THE TRAP.

HOLE 55 (19) IS LOCATED IN THE OUTLET PIPE WHICH IS VERTICAL AT THE TOP OF THE TRAP AT THE MID RADIUS OF HALF PLANE 2.

Figure 11

**SAMPLE LOCATIONS
HOLES 22-31,46-49
ENRICO FERMİ COLD TRAP**

Figure 12

**SAMPLE LOCATIONS
HOLES 42-45,50-53
ENRICO FERMİ COLD TRAP**

Figure 13

**SAMPLE LOCATIONS
HOLES 32-41,46-49
ENRICO FERMI COLD TRAP**

Figure 14

subsequent samples were of this size. This diameter permitted removal of long, undistorted samples, suitable for examining the mesh.

4.2 Chemical Analyses

The analytical methods considered and finally selected for analyzing samples from the Fermi cold trap are described in detail in Appendix B.

It is considered pertinent at this point to describe the nature of sodium and the general precautions to be observed in handling sodium samples to determine successfully their impurities of interest. Sodium metal is extremely reactive and has a strong affinity for oxygen, moisture, and carbon dioxide, all of which are abundant in the air. Therefore, the analytical sample must be protected from air to minimize atmospheric contamination during analysis. Sample transfer should be conducted in a dry, inert atmosphere. This can be accomplished most conveniently by handling the analytical sample inside a leak-tight glove box. The glove box should be equipped with a recirculating gas purification unit to remove oxygen and moisture from the inert atmosphere.

It is equally important to guard against contamination during the actual analysis of sodium samples. Leak-tight conditions in all analytical apparatus are essential. These precautions were rigidly observed. The glove box and gas purification unit used by

Westinghouse to handle samples from holes 20 through 55 is shown in Figure 15.

In analyzing sodium samples extracted from the Fermi cold trap, certain modifications were made to the analytical procedures that had been selected. As a rule, these changes included adjusting the size of the analytical sample to accommodate the wide range of impurity concentration found in the cold trap material. To establish the appropriate size of the analytical samples, three samples of sodium and mesh from adjacent trap locations were analyzed for oxygen. The following results were obtained:

<u>Weight of Sample</u> <u>grams</u>	<u>Oxygen content</u> <u>ppm</u>
5.0	1200
1.0	1170
0.1	8400

These results indicated that very small analytical samples were unsuitable because they might emphasize local variations. Moreover, the presence of mesh made it difficult to remove neatly very small specimens from the sample cores. Thus, most of the sampling employed for the chemical analyses weighed between one and two grams.

A literature survey of applicable methods of chemical analyses resulted in the annotated bibliography described in Westinghouse report WCAP-2540.

Figure 15 Inert Atmosphere Glove Box for
Sodium Sample Preparation

5. EXPERIMENTAL RESULTS

To accomplish the objectives of this program as listed in Section 2, sample cores of the trap contents were removed from the trap and analyzed for the impurities of interest. At the suggestion of the AEC, an experiment was also performed to determine the density gradients in the trap by means of a gamma radiation survey technique. The results of the experimental phase of this program follow.

5.1 Physical Examination of the Cold Trap and Sample Cores

While the samples were being removed and analyzed, certain observations were made of the physical appearance of the sample cores and of the general condition of the cold trap structure.

5.1.1 Physical Appearance of Sample Cores

The appearance of the sample cores reflected their relative degree of contamination. Sample cores removed from the lower mesh region of the trap exhibited a coarse, grainy, dull appearance, and were noticeably hard and brittle. Chemical analysis later revealed a high concentration of sodium oxide in this region of the trap.

Samples removed from the mesh-free regions of the trap were characterized by a bright, silvery appearance. These samples also had a soft and putty-like consistency. Photographs of selected sample cores removed from the trap, with specific comments concerning their general appearance, are included in Appendix B.

The internal surface of Hole No. 1 in the lower mesh region was examined with a boroscope after the sample core had been removed. Figure 16 shows the general appearance of the internal surface at different depths. A granular surface was observed at the periphery. As shown by the view at the 24 inch depth, the rest of the surface was smooth, except at the 20-inch depth, where a rough surface was noted for half of the circumference.

5.1.2 Examination of the Mesh in the Sampling Cores

To evaluate the role of the mesh in the trap, experiments were performed to separate the sodium from the mesh without disturbing the sodium oxide deposits on the mesh. Details of the experimental procedures employed are included in Appendix D. Subsequent microscopic examination of the isolated oxide-laden mesh showed the pattern of the oxide deposit on the mesh. Based on 42 samples, it was observed that a uniform coating of oxide had formed on the wires, with a fillet or web-type buildup at the intersection of the wires. As the thickness of the coating increased to a few mils, the deposit

10" From Wall

11" From Wall

20" From Wall

24" From Wall

Figure 16 Boroscope Views in Hole No. 1

appeared less dense and had a fuzzy structure (see Figure 42).

This observation suggested the possibility of direct crystal growth on the wire. Metallurgical examination of ten stainless steel mesh samples, taken from various regions of the trap, showed no evidence of surface corrosion or of intergranular attack.

5.1.3 Collapsed Wall

When the samples were removed from the cold trap, it was noted that the inner liner (Figure 1), designed as an inverted cup with straight walls, was severely collapsed for half of its circumference and for almost all of its height. This deformation was found in half-planes 1 and 2 and is shown to scale in Figures 12 and 13. The maximum observed displacement was $7\frac{3}{4}$ inches. No deformation was found in half-planes 3 or 4. The possible cause and effect of the collapsed wall are discussed in Section 6.2.

5.1.4 Displaced Mesh

The trap was designed to contain 12 lb. per cu. ft. of stainless steel mesh.* Examination of the cores removed from the mesh region showed a mesh content ranging from 0 to 37.8 w/o (0 to 32.5 lbs. per cu. ft.), with most of the samples containing between 15 and 30 w/o

* The Fermi Hazards Report states 10 to 20 lb. per cu. ft.

(10 to 23.7 lb. per cu. ft.) Some of the cores, removed from the lowest mesh region, contained no mesh at the center of the trap. This suggested that the mesh was displaced upwards during operation of the trap, and was held there by the formation of a crust of sodium oxide at the perimeter of the trap before final freezing and removal. Another possibility is that the squeezing effect of the deformed wall caused the displacement of the mesh, although this effect probably would have displaced the mesh downwards.

5.1.5 Examination by Gamma Radiation Survey Technique

In an attempt to make a gross determination of the overall distribution of material in the cold trap, it was decided to use gamma radiation surveying. A radium source was inserted into the thermocouple well of the trap, and the radiation intensity was measured at various locations along the outer surface of the trap. This experiment was conducted by Applied Health Physics, Inc. of Bethel Park, Pennsylvania.

This experiment proved unsuccessful. Because of a high degree of radiation scattering, the data could not be used to indicate the distribution of material. The results showed several unexplained anomalies. Further analysis of the data or use of a more energetic radiation source could not be justified. Details of the experimental procedure, the results obtained, and an evaluation of the results are contained in Appendix E

5.2 Chemical Examination of Cold Trap Contents

The samples removed from the trap were analyzed by employing the methods and techniques described in Appendix B of this report. The following discussion includes an appraisal of the general results obtained, and specific comments on each impurity.

5.2.1 Distribution of Impurities in the Cold Trap

The concentration values of impurities in the cold trap are shown in Figures 17 through 24. These figures indicate the distribution of impurities as a function of their elevation in the cold trap. Logarithmic scales were used for the amounts of contaminants to show the scatter of data over many decades, with mesh distribution expressed on a linear scale. The analytical results for oxygen are displayed on both logarithmic and linear scales, with each dot on the figures representing an analysis. Shaded areas represent the spread of the data. The horizontal distribution is not shown in these figures. The analytical results are displayed in Figures 48 through 64, and are summarized in Tables 8 and 11 of Appendix F.

The high concentration of impurities in the lower mesh region indicates that the stainless steel mesh acted as a nucleating and filtering medium. Attempts were made to isolate and measure the sodium oxide, on and adjacent to the mesh, by mechanically separating the mesh from the sodium. These experiments proved unsuccessful.

-111-

Figure 17

Figure 19

-7-

Figure 20

Figure 21

BASIS: MESH FREE: CONTAMINANT PRESENT

VERTICAL DISTRIBUTION OF NITROGEN IN THE FERMI COLD TRAP

Figure 23

Figure 24

because the mesh could not be removed from the sample core without disturbing the oxide deposit, thus contaminating both the mesh and the remaining sodium.

Several unexplained variations were noted in the distribution of metallic impurities, i.e., a rather high concentration of copper and iron in the lower half of the mesh region plus reversal of the carbon concentration trend near the top of the mesh region. The association of copper and iron with high concentrations of oxygen in sodium indicates the existence of a concentration mechanism such as coprecipitation, flocculation or occlusion. However, the precise nature of this mechanism was not determined.

5.2.2 Material Balance for Contaminants in the Cold Trap

Although the magnitude of impurities was of interest, an accurate material balance for the impurities could not be calculated because the sodium in the trap was not homogeneous. The total weight of carbon and oxygen in the trap was therefore estimated on the basis of the analytical data. The results of this estimate are included in the discussion that follows.

The total carbon and oxygen in the Fermi cold trap was estimated by assigning mean values for total carbon and oxygen to different regions of the trap. For the purpose of this estimate,

the sodium in the trap was divided into three volumes, as follows:

Volume "A" included the bottom 12 inches of the mesh zone.

Volume "B" included the remaining portion of the mesh zone.

Volume "C" included the mesh-free zones at the top and bottom of the trap.

Based on the analytical results for total carbon and oxygen in each of these zones, a mean concentration value for each impurity was estimated and assigned, as follows:

<u>Total Carbon</u>	<u>Total Oxygen</u>
Volume "A" = 0.070%	Volume "A" = 6.1%
Volume "B" = 0.013%	Volume "B" = 0.3%
Volume "C" = 0.005%	Volume "C" = 0.005%

These figures represent estimates of the average per cent by weight of each impurity, and are intended to show only orders of magnitude of concentration. Subsequent calculation of total amounts of these impurities should be treated strictly as an estimate.

The volume of each zone was calculated, using 46 inches as the nominal diameter, while realizing that the collapsed wall would introduce some error in this calculation.

The calculated volume for each zone in the trap was as follows:

Volume "A" = 12 cu. ft.

Volume "B" = 42 cu. ft.

Volume "C" = 16 cu. ft.

The calculated total carbon and oxygen content for each zone, assuming a sodium density of 57.9 lb. per cu. ft. (density at the freezing point) was as follows:

<u>Total Carbon</u>	<u>Total Oxygen</u>
Volume "A" = 0.49 lb.	Volume "A" = 42.50 lb.
Volume "B" = 0.32 lb.	Volume "B" = 7.30 lb.
Volume "C" = 0.04 lb.	Volume "C" = 0.05 lb.

Total carbon in trap = 0.9 lb. Total oxygen in trap = 50 lb.

5.2.3 Preparation of Samples for Analyses

The samples to be used for determination of carbon, hydrogen, oxygen, nitrogen, metallic impurities and mesh content were cut out of the sodium cores removed from the trap. First, the oxidized outer surface of the cores was removed. Specimens were then cut out of the cores, either with a clean knife or with a stainless steel cork borer and plunger. These samples were transferred in a glove box filled with purified argon (see Figure 15).

5.2.4 Determination of Specific Impurities

This section includes observations and general comments on the determination of each specific impurity of interest

5.2.4.1 Stainless Steel Mesh

Sample cores removed from the middle portion of the cold trap contained stainless steel mesh. During the examination of these cores, data was obtained on the concentration of this mesh in the cold trap sodium.

Throughout this report, mesh concentrations are stated in weight per cent (w/o) of the cold trap material. The vertical distribution of the mesh is shown in Figure 25.

Figure 25 also shows, on non-linear scales, the mesh distribution in lb. per cu. ft. and in mesh-to-sodium ratio. The conversion factors employed in Figure 25 were based on a stainless steel mesh density of 490 lb. per cu. ft. and a sodium density of 57.9 lb. per cu. ft. The term "sample" here refers to stainless steel mesh plus contaminated sodium. The conversion factors were calculated as follows.

$$\text{Let } A = \text{w/o mesh} = \frac{\text{lb. stainless steel} \times 100}{\text{lb. sample}}$$

$$\text{Let } B = \frac{\text{mesh}}{\text{sodium}} = \frac{\text{lb. stainless steel}}{\text{lb. sodium}}$$

VERTICAL DISTRIBUTION OF S. S. MESH IN THE FERMI COLD TRAP

Let $C = \text{mesh packing density} = \frac{\text{lb. stainless steel}}{\text{cu. ft. sample}}$

Then

$$A = \frac{100}{\frac{1}{B} + 1}$$

$$A = \frac{113.5}{1 + \frac{65.6}{C}}$$

$$B = \frac{8.45}{\frac{490}{C} - 1}$$

$$B = \frac{1}{\frac{100}{A} - 1}$$

$$C = \frac{65.6}{\frac{113.2}{A} - 1}$$

$$C = \frac{490}{\frac{8.45}{B} + 1}$$

The data for mesh concentration can be interpreted to define the uniformity of the sample core. It can also be used to check the actual mesh concentration against the design concentration.

5.2.4.2 Analytical Round Robin and Check Analyses

In the early stages of the analytical work, it was decided to confirm the accuracy of the WAPD version of the amalgamation procedure. Accordingly, three sets of "round robin" samples were submitted to independent laboratories for oxygen determination. The results obtained by two laboratories did not agree either with each other or with results obtained by WAPD. At a meeting between representatives of these two laboratories and WAPD personnel, it was generally agreed that the difference in sample size might have caused the variation in results. It was also recognized that a glove box, as originally

used by WAPD, was not suitable for handling samples having a very low oxide content. The glove box wasn't tight enough to maintain the low water and oxygen contents required for handling high purity samples. This was confirmed by high value results on samples of Fermi primary sodium containing a nominal 10 to 20 ppm oxygen. Modifications of the WAPD analytical procedures were recommended and adopted. The WAPD glove box was tightened and a third "round robin" was conducted with samples from the upper mesh region. These had a nominal oxygen content of about 0.3 w/o. Considering the homogeneity of oxygen concentration in that region, there was good agreement between results obtained by the laboratories. The results of this analytical program are summarized in Table 2.

To maintain the necessary atmosphere for handling high purity sodium samples, a new, vacuum-type glove box was installed and outfitted with a recirculating gas purification unit. Figure 15 shows this glove box and its control panel. A Westinghouse oxygen meter was used to monitor the oxygen in the argon atmosphere, with a Consolidated Electrodynamics moisture monitor used to indicate the water content. The purification unit consistently maintained the oxygen below 1 ppm, and the moisture below 15 ppm. These conditions were adequate for processing samples from the Fermi cold trap, as was demonstrated by the results obtained for oxygen in a second sample of Fermi primary sodium. This sample was supplied by APDA.

Table 2

Oxygen in Sodium Round-Robins

Round Robin	Laboratory	Sample Identification																
		1-1-A	1-1-C	1-2-A	3-1-A	3-1-B	3-1-C	3-2-B	3-2-C	3-3-A	6-1-A	6-1-B						
First	WAPD	17.6 (2.34)		29.6 (2.55)	0.44 (1.42)	0.82 (1.14)		1.24 (1.43)		1.51 (1.15)	1.30 (0.90)							
	APDA		10.54				0.27		2.54		9.44							
		4-2-1	4-2-2	4-2-3	4-2-4	8-2-1	8-2-2	8-2-3	8-2-4	8-2-6	8-3-1	8-3-3	8-3-4	8-3-5	12-1-1	12-1-2	12-1-3	12-1-5
Second	WAPD		3.06 (0.86)		1.84 (0.60)		0.24 (0.97)		0.28 (0.76)	0.67 (0.34)	4.56 (0.675)	0.52 (0.936)		0.50 (0.800)	2.09 (0.856)		0.153 (0.741)	0.098 (0.862)
							0.18 (1.42)			0.45 (2.48)								
							0.17 (1.71)											
	APDA	0.30 (0.124)		0.57 (0.100)		0.04 (0.105)		0.06 (0.112)										
		0.31 (0.145)		0.54 (0.082)		0.03 (0.114)		0.06 (0.110)										
		0.32 (0.084)		0.60 (0.067)		0.08 (0.122)		0.05 (0.056)										
	ORNL		0.40 (0.16)		0.54 (0.24)		0.15 (1.00)		0.16 (0.60)									
	MSAR											0.11 (2.03)					0.00083 (0.768)	
		8-4-1	8-4-2	8-4-3	8-4-4	8-4-5	8-4-6	8-4-7										
	Third	WAPD	0.59 (5)				0.26 (5)			0.27 (5)								
		0.49 (5)				0.31 (5)			0.33 (5)									
APDA					0.26 (1)				0.08 (1.8)									
					0.31 (0.1)				0.10 (0.88)									
									0.31 (0.112)									
	ORNL		0.32 (9)				0.31 (9)											

- NOTES: 1. WAPD = Westinghouse Atomic Power Division
 2. APDA = Atomic Power Development Associates
 3. ORNL = Oak Ridge National Laboratory
 4. MSAR = Mine Safety Appliance Research

5. Results are in weight percent oxygen in sodium.
 6. Sample weight in grams, when known, is in parentheses.
 7. The first part of each sample number is the hole number.
 8. First round robin held in July 1964.

9. Second round robin held in August 1964.
 10. Third round robin held in September 1964.

They had analyzed the sample material, and reported oxygen concentrations of 20, 20, and 24 ppm. The WAPD results were 19, 20, 29, and 30 ppm oxygen. The new glove box was used to prepare the analytical samples from holes 20 through 55 of the cold trap.

Similar samples of cold trap material were submitted to independent laboratories for check analyses. WAPD analyzed samples from holes 32 through 41 ("32 series"). Samples from holes 22 through 31 ("22 series") were analyzed by LeDoux for nitrogen and by APDA for oxygen, total carbon, carbonate carbon and hydrogen. As shown in Figure 11, the "32 series" holes were very near the "22 series" holes. APDA analyzed additional samples to provide extensive data for all trap locations.

The following laboratories either participated in the analytical round robin, or provided check analyses for Westinghouse on samples from the Fermi cold trap:

Oak Ridge National Laboratory	Atomic Power Development
P. O. Box X	Associates
Oak Ridge, Tennessee	1911 First Street
	Detroit 26, Michigan
LeDoux & Co., Inc.	MSA Research Corporation
359 Alfred Avenue	Callery, Pennsylvania
Teaneck, New Jersey	

Westinghouse Electric Corp.
Technical Services Laboratories
Atomic Power Division
P O. Box 355
Pittsburgh, Pennsylvania 15230

5 2.4.3 Oxygen

The mercury amalgamation procedure was employed to determine the oxygen content in sodium samples from the Fermi cold trap. The limitations of this method of analysis are discussed in Appendix B, Selected Methods of Chemical and Spectrographic Analyses.

The oxygen content in the cold trap samples was not known precisely at the beginning of this program, but the suspected range was between 10 ppm and 20 per cent. Actual results show that this estimate was correct. The maximum value for sodium monoxide is 25.8%. Samples with a low oxygen content required special handling during analysis. Discussed below are some of the more important observations noted during the processing of all the samples through the analytical procedure for the determination of oxygen.

Samples from the lower mesh region, where the oxygen content was high, were analyzed by the amalgamation procedure. This proved difficult to accomplish because the presence of a black residue, floating on the surface of the amalgam, made complete extraction of the sodium

metal almost impossible. For example, on some samples 12 extraction steps were required, as compared to 6 steps on low oxide sodium samples. A sample of the black residue from amalgamation of high oxide sodium samples was examined by X-ray fluorescence techniques. The results showed the matrix to be mercury containing appreciable amounts of copper. Microscopic examination of the black residue showed small droplets of mercury coated with a black film, possibly a copper compound. Figure 26 shows extraction vessels containing sodium samples having high and low oxygen contents. A black film completely covers the walls of the vessel on the left, which contains the high oxygen sodium sample. Approximately 1/4-inch of black powder is floating on top of the amalgam, but is obscured from view by the heavy black film on the vessel wall. Olfactory evidence of metallic carbides was detected after water was added to the residue from amalgamation. Generally phosphine is evolved with acetylene during the hydrolysis of certain metallic carbides.

The role of the mesh as a nucleating agent was studied by attempting to measure the amount of oxide contained in the sodium deposited on the mesh and between mesh wires. Two methods were employed. Two adjacent sodium samples were taken from hole number 5, located in the middle of the mesh region. Each sample was amalgamated, and the contaminated mesh removed with tweezers. Then, each sample was reacted with water and titrated. The remaining oxide in the amalgamation vessel was also determined. For one of the samples, 0.001% of

Figure 26 Amalgamation and Separation Equipment
for Oxygen Determination

the total oxygen appeared to be on the mesh. The corresponding value for the other sample was 0.2%. This low concentration of oxide could be explained by the washing action of the extraction procedure and the tendency of oxide residues in mercury to migrate to the walls of the amalgamation vessel.

In the second method, a sample was taken from hole number 30, located in the upper mesh region. The mesh, covered with sodium and sodium oxide, was removed from the sample with tweezers and analyzed for oxygen by the amalgamation method, as was the rest of the original sample. About 20% of the total oxygen appeared to be on the mesh.

This work was discontinued for two reasons: the sample apparently had become contaminated while the mesh was being removed with tweezers; and second, the mesh could not be separated completely from the sodium matrix. Rapid distillation of the sodium by induction heating was considered. However, this was not attempted, because sufficient temperatures could have been generated in the mesh, even with its low susceptibility, to either volatilize the sodium oxide or possibly cause a reaction between the mesh and the oxide residue.

5.2.4.4 Carbon

The results for total carbon and for carbonate carbon show that the carbon was distributed in the trap in about the same pattern as

the oxygen. Nearly 95% of the carbon estimated to be in the trap was found in the mesh region. Over 50% was found in the bottom 12 inches of the mesh region. The association of carbon and oxygen could be inferred from the analytical results for these two impurities. However, no attempt was made to verify the existence of a common chemical specie containing both carbon and oxygen.

5.2.4.5 Hydrogen

Hydrogen was also distributed in the trap in about the same pattern as the oxygen, i.e., high concentrations in the lower mesh region and relatively low concentrations in the mesh-free sodium. It was observed that a considerable amount of hydrogen evolved when the sodium samples were analyzed by the amalgamation procedure. This indicated the presence of hydrogen in the form of hydride and/or dissolved hydrogen, rather than as sodium hydroxide. The results are reported as total hydrogen and include the forms H_2 , NaH and NaOH.

5.2.4.6 Nitrogen

The nitrogen was distributed in the trap in the same general pattern as the other impurities described above. While previous workers ⁽⁴⁾ have stated that nitrogen must be carried by calcium in a sodium matrix, the spectrographic examination of the sodium from the Fermi cold trap showed that the calcium concentration was below

detection limits. The nitrogen in the cold trap sodium might have been combined or coordinated with other metallic impurities in the sodium; possibly being present as dissolved nitrogen.

5.2.4.7 Metallic Impurities

Samples of the material removed from the cold trap were processed for metallic impurities in accordance with the procedure given in Appendix B. The concentration of some thirty elements could be detected and measured by this procedure with the limits of detection for the thirty elements given in Appendix B. Results of the analyses are summarized in Tables 8 and 11 of Appendix F. Using the results for a sample of Fermi primary sodium (Table 11) as a base-line, a significant increase was evident in the concentration of copper, iron and potassium in the samples from the cold trap. The vertical distribution of copper in the trap is shown in Figure 23. Distribution of the metallic impurities tended to follow the vertical oxygen distribution curve (Figure 17). The increased solubility of metallic impurities in sodium containing significant amounts of sodium oxide may cause the accumulation of the metallic impurities in the trap. However, there is no immediate explanation for the presence of the copper in the system since copper-free brazing material was employed in manufacturing the support cages for the Fermi fuel pins.

5.2.5 Number of Samples

To be useful, a sample must have all the characteristics of the material from which it is taken. Frequently, a small sample can accurately represent a very large amount of homogeneous material. However, when a heterogeneous mixture is involved, such as the contents of the cold trap, the sample should reveal all the components of the mixture and their distribution. The more heterogeneous the distribution, the larger the number of samples needed, with economics and use of remaining material being limiting factors.

Since the oxides were expected to be concentrated in the mesh region, the first samples were taken at the top, middle, and bottom of the mesh region. The oxygen contents of the top and middle samples were low and close together. The oxygen concentrations in the bottom samples ranged from very low to very high. Therefore, the sampling elevations in the lower mesh region were selected close together and many samples were taken at each elevation. Since the horizontal distribution at any given level was expected to be rather uniform, samples were taken at four equally spaced radii at most elevations. The oxygen concentrations outside the mesh region were expected to be low and uniform, thus only one elevation was selected above the mesh, and one below the mesh.

The analytical results for oxygen in the Fermi cold trap are displayed in Figures 17 and 18. The envelopes of these values are

indicated by a shaded area. In the case of the upper two-thirds of the mesh region, all of the samples contained less than 1 w/o oxygen. It is inferred from this pattern that results of further analyses would also lie in the shaded area. Therefore, further sampling at the given elevations or between them, was unwarranted. In the lower third of the mesh region, the interpretation of widely-scattered oxygen concentration values was confounded by the displaced and compacted mesh. It is unlikely that further analyses in the lower mesh region would have revealed a different pattern for oxygen distribution.

Most of the analyses were for oxygen (151) because the basic function of a cold trap is to remove oxygen from sodium. Fewer samples were analyzed for total carbon (86), carbonate carbon (92), hydrogen (31), nitrogen (40), and metallic impurities (37). However, the sampling pattern for these impurities followed that for oxygen.

6. EVALUATION OF COLD TRAP PERFORMANCE

After achieving the experimental results described in Section 5, the following steps were taken to meet the program objectives of : 1) evaluating the operational performance of the trap; and 2) developing design criteria for future sodium cold traps.

First, the experimental observations and results were analyzed and a theory was developed to explain the sodium oxide precipitation mechanism. This theory is supported by evidence from the cold trap examination. The cause of the collapsed wall was postulated and the overall actual performance of the trap was calculated. This performance was then compared with that expected by the MSA Research Corporation, the designer of the Fermi Cold Trap.

Necessary design criteria and desirable design criteria were listed and discussed for cold traps in general. The applications of the criteria to the Fermi cold trap design were noted.

6.1 Sodium Oxide Precipitation Mechanism

To understand the operation of the Fermi cold trap, the processes of filtration and crystallization were considered in order to determine their role in the oxide retention mechanism.

Filtration may be defined as the separation of a heterogenous mixture of a fluid and solid particles by a filter medium that permits fluid to pass, but retains the solids. Normally, the original filter material is not the true filter medium since the average particle size is usually considerably finer than the average opening between the filter elements. Thus, the real filter medium is the layer of filtered solids

Crystallization processes in stagnant fluid normally occur very slowly. Any solid in contact with its solution will either grow at the expense of the solution if the solution is super-saturated, or will dissolve if the solution is unsaturated. In either case, equilibrium is represented by the solubility of the solid in the solvent. Seed crystals or foreign matter in the solution act as crystal growing sites. The degree of supersaturation is determined by the cooling rate. At high cooling rates there is a high degree of supersaturation and many small crystals, at low cooling rates, there is a small degree of supersaturation and the crystals are large. Crystal formation and growth depend on solution agitation, distance between crystals, and purity of solution.

An oxide deposit was found in only one area in the lower plenum of the cold trap. The sample next to the wall in Hole 42 contained 15.5 w/o oxygen, indicating that some oxide had precipitated before reaching the mesh area. Furthermore, most of the wire in the trap

had a thin black coating of oxide except in the lower mesh region, where the oxide coating was thick, white, and fuzzy.

Based on the following theory the sodium between mesh wires should have contained very little oxygen. The highest plugging temperature noted was 650°F, equivalent to 150 ppm oxygen. Precipitated oxide particles could adhere to the mesh, be carried out of the trap, or sink. It is unlikely that the oxides would be suspended in flowing sodium and accumulate at any given point. Therefore, the oxygen concentration in the spaces between mesh wires should not have exceeded 150 ppm. When samples of sodium and mesh were analyzed the oxygen contents were between 100 and 1000 times more than 150 ppm. Therefore, virtually all of the oxygen in the mesh region was attached to the wire mesh. An attempt to verify this theory by chemical analysis was unsuccessful. (See section 5.2.4.3)

Based on the filtration and crystallization mechanisms and on examination of the oxide deposits, the following process is believed to have occurred. As the oxide-laden sodium entered the cold trap, a sudden reduction in temperature caused the formation of very small particles of sodium oxide. These particles were carried by sodium, through the annular space at a relatively high velocity. When they reached the mesh area, however, their flow velocity was drastically reduced. As the particles passed through the mesh, some hit and

adhered to the wires. When new particles passed through the same area, they probably deposited on an oxide surface, rather than on a base wire. In this manner, a thick layer and high concentration of sodium oxide accumulated on the upstream face of the mesh region. This theory was verified by the chemical and physical examinations, which were described in Section 5.

6.2 Collapsed Wall

Deformation of the inner liner (Figure 1) was noted when the samples were removed from the cold trap. The inner liner, designed as an inverted cup with straight walls, was severely collapsed along half of its circumference and for most of its height. Figures 12 and 13 show the deformation, which had a maximum displacement of 7-3/4 inches.

There are three possible explanations for the collapsed wall. The first, and most probable explanation, is based on the application of pump pressure against the wall as the result of a dense oxide formation in the mesh area. The second explanation is similar, but is based on oxide blockage of the annular space near the bottom of the trap. The third possibility is that the wall collapsed and the mesh became distorted during fabrication of the cold trap.

The first theory is as follows. Sodium entered the cold trap; flowed down the 3/4-inch annulus; passed up through the mesh region; and left the trap. The mesh at the bottom of the mesh region was displaced upward about 2 to 4 inches (see Figures 12 and 13). The

oxygen profiles shown in Figures 17 and 18, show oxygen concentrations greater than 10% in the lower mesh region. According to its designer, the useful life of the trap would end when the flow resistance increased suddenly and rapidly and its oxygen concentration exceeded 8% (about 30% of the maximum value). The maximum oxygen concentration would exist when all the sodium became converted to sodium monoxide. It is postulated that a layer of sodium oxide accumulated in the lower mesh region, thus permitting application of pump pressure across the innermost wall of the cold trap and causing that wall to collapse. The pressure was applied to the mesh and caked oxide with an equivalent force of 1800 pounds. This force lifted the mesh until an area free of oxide opened, allowing the sodium to pass. The formula for the collapsing pressure of a perfect thin cylinder of infinite length is $2.2E \left(\frac{T}{D}\right)^3$. For the inner shell of the cold trap, $E = 30 \times 10^6$, $T = 0.125$, and $D = 48$. The resulting calculated value of collapsing pressure was 1 psi. In this calculation, the restraining end effect of the top head was considered to be more than offset by the ovality of the cylinder. The initial operating conditions for the annulus and the mesh region at a maximum sodium flow rate of 100 gpm were as follows:

	<u>Annulus</u>	<u>Mesh Region</u>
Area, sq. ft.	0.8	13.0
Velocity, fps.	0.3	0.02
Pressure drop, psi	0.001	<<0.001

Pressure drop is proportional to the square of the velocity, and velocity is proportional to the cross sectional flow area.

Therefore, most of the flow area at the bottom of the mesh had to be plugged before the 1 psi value was reached.

In the second theory, the annular space near the bottom of the trap became blocked with oxide, causing the wall to collapse. This theory, however, does not explain the displacement of the layer of mesh. Also, all the sodium removed from the annular space with hole saws was soft and shiny. The typical hard crusty texture of sodium with a high oxygen content was not observed. Such an absence of crusty material might be explained by postulating a temporary plug in the annulus which caused the wall to collapse. The plug then broke free and dropped into the lower plenum.

The third possibility, i.e., that the wall collapsed and the mesh became distorted during fabrication of the cold trap, is considered improbable.

Fortunately, the collapsed wall did not affect the integrity or operation of the cold trap.

6.3 Actual Performance of the Cold Trap

The actual performance of the cold trap was determined from operating data, from analytical results, and from physical examination of the trap and sample cores.

The total oxygen introduced into the Fermi primary system was estimated from the following information derived from the operating

history of the trap as shown in Figure 2. The initial supply of sodium was received in five tank cars containing a total of 342,000 lbs. of sodium. This sodium was transferred to storage tanks and cold-trapped for 50 hours. During cold-trapping, the bulk sodium temperature was dropped from 600°F to 450°F. The initial plugging temperature was 480°F, equivalent to 60 ppm oxygen. After cold-trapping, the bulk temperature was raised to 600°F. At this point, the plugging temperature was about 300°F, equivalent to 10 ppm oxygen. Thus, 17 lbs. of oxygen were removed at an average rate of 0.34 lb per hr., or 80% of the design value. Later, the final tank car load of sodium was added to the system, and an additional 2.5 lbs of oxygen were removed by cold-trapping. When the sodium was transferred to the reactor for flow tests, some oxygen was removed during the high temperature tests and during routine operation. During a seven-day period, with the bulk sodium at 425°F, the plugging temperatures dropped from 425°F (35 ppm oxygen) to 300°F (10 ppm oxygen). Since the system contained 500,000 lbs. of sodium, 12.5 lbs. of oxygen were removed at a rate of 0.07 lb. per hr., or 15% of the design value. Information on plugging temperatures is reviewed in Section 3.2.

There was no evidence of increased pressure drop during the operation of the trap. This fact was determined by monitoring the flow vs. power values for the electro-magnetic pump that circulated the sodium through the cold trap. The final use of the cold trap was in January, 1963. The cold trap was then removed from service so

that a new one could be installed to assure maximum capability for operation under radioactive conditions.

6.4 Engineering Analysis of Cold Trap Performance

6.4.1 Design Performance

The design of the Fermi cold trap was based on the stoichiometric conversion of sodium and oxygen to sodium monoxide. In accordance with the equation $2 \text{ Na} + 1/2 \text{ O}_2 = \text{Na}_2\text{O}$, 1 lb. of sodium can be converted to 1.35 lbs. of sodium monoxide containing 0.35 lbs. of oxygen.

Based on a sodium volume of 54 cu. ft. in the mesh region, equivalent to 3000 lbs. of sodium, the trap theoretically can remove about 1050 lbs. of oxygen. When the apparent density of the sodium oxide approaches 30% of the theoretical value, the flow resistance of the trap increases rapidly and the trap's useful life is considered to be expended.

The Fermi cold trap was designed by MSA Research Corporation. The operating manual for the cold trap contained the following design data. (8) Additional information is given in Appendix G.

Flow = 100 gpm

Oxygen removal rate = 0.438 lb. per hr.

Oxygen removal capability = 290 lb. (9)

<u>Plugging Temperature</u>	<u>Temperature Difference Between Sodium Entering the Trap, and Sodium Entering the Mesh Region</u>
700°F	5°F
650	6
600	13
550	25
500	33
450	33
400 or less	50

The temperature relationship shown above results in a constant oxygen removal rate. This prevents isolated oxide precipitation, which could lead to premature plugging of the trap. The plugging temperature was associated with the amount of dissolved oxygen, according to the curve in Figure 27. The flow rate and temperature of the sodium were established by adjusting the flow rate and temperature of the NaK coolant.

6.4.2 Comparison of Actual Performance and Design Performance

The total oxygen concentration in the trap was estimated to be 50 lbs. This amount was calculated by integrating the oxygen concentrations measured in all the zones of the trap. A material balance of the whole system indicated that 32 + lbs. of oxygen were removed from the primary sodium system. The two numbers are of the same order of magnitude. Since the designed oxygen removal capability was 290 lbs, less than 20% of the expected life of the cold trap was achieved. However, since the oxygen concentration in one part of the trap

SOLUBILITY CURVE FOR SODIUM MONOXIDE IN SODIUM

Figure 27

approached the maximum theoretical value and there was a very low oxygen concentration in the rest of the trap, the designed removal capacity might have been achieved with further operation.

The low level of impurities in the Fermi primary sodium system and the accumulation of oxygen, carbon, hydrogen, nitrogen, copper and iron in the trap show the cold trap to be a suitable device for general sodium purification.

At any given elevation of the trap, the horizontal distribution of impurities and mesh concentrations were relatively uniform. This indicates there was no flow maldistribution.

6.5 Design Criteria for Sodium Cold Traps

6.5.1 Design Criteria of the Fermi Cold Trap

Examination of the Fermi cold trap and its contents, plus an evaluation of the performance of the trap, indicated that its design met most of the criteria listed below.

Necessary Design Criteria for Sodium Cold Traps

1. Suitable sodium oxide removal capacity and rate. The oxide removal capacity of a cold trap is proportional to the total volume of the trap. The geometry factor, employed in designing the trap, should result in a flow path of reasonable cross-section and length. Small flow path cross-sections are undesirable as they may allow the formation of an oxide layer

which can cause early plugging of the trap. A long flow path is not necessary to achieve high sodium purity, e.g., 10 ppm oxygen.

The residence time for the sodium in a cold trap is equal to the trap volume divided by the trap volumetric flow rate. Typical residence times are between five and ten minutes. The Fermi cold trap was designed for a five-minute residence time. However, the trap normally operated at half of its design flow rate, so the typical residence time was ten minutes. Short residence times, resulting from high flow rates, decrease the rate of oxide removal because some oxygen passes through the trap instead of being retained. Long residence times, resulting from low flow rates, also decrease the rate of purification because a smaller fraction of the sodium in the primary system is cold-trapped in a given period.

The size of a cold trap for a given sodium system should be selected on the basis of the amount of oxygen to be removed in a given period. Small size traps can be used either in parallel or one after another. Sequential operation requires more frequent replacement of the traps. A disadvantage of small size traps is the handling expense and inconvenience incurred during replacement. A small cold trap can prove to

be more economical than a large trap if the large trap has to be removed because of surface gamma radiation conditions rather than exhaustion of purification ability. Wire mesh in a cold trap is very effective as a nucleating area for sodium oxide. In the Fermi cold trap, essentially all the sodium oxide was deposited on the mesh. While the 11-mil diameter wire only occupied 2.5% of the volume in the mesh region, the accumulative wire length was 380 miles, and the total surface area of the wire was 5800 square feet.

- 2 Compatibility of materials with sodium. Stainless steel is commonly used as a construction material for cold traps, although carbon steel has also been used. Since cold traps normally operate at about 300°F , carbon steel would not experience severe corrosion and could be used satisfactorily even with a moderate amount of surface rust. However, continued operation at high temperatures should be avoided with carbon steel because of the increased corrosion problem. All parts of the Fermi cold trap in contact with sodium were made of stainless steel.
- 3 Capable of operating at high temperatures. If, because of a large accumulation of contaminants in the sodium, the plugging temperature of a primary sodium system is very high, say 800°F , the primary sodium system and the cold trap must be at

an even higher temperature for the sodium to flow. The maximum design temperature of the Fermi cold trap was 1000^oF.

4. Suitable for credible external and internal pressures. If a vacuum occurs inside the trap, the pressure differential across the outer shell can be 15 psi. The pressure inside the trap might be the total pressure of the flowing sodium caused by the sodium pumps. Sub-structures inside the main shell should be designed for abnormal but credible pressures.
5. Provision for expansion of the sodium. When the cold trap heaters are turned on, the sodium will expand. A trap must be able to accommodate the expansion that will occur if the trap is accidentally isolated. The Fermi cold trap had a 5 cu. ft. expansion space in the upper head, and was equipped with an 80 psig rupture disc.
6. Heaters. When a cold trap is installed, it must be heated above the melting point of sodium (208^oF) for the sodium to flow. Electric heaters are usually employed for this purpose. Hot gas, oil or steam can also be used in tracer lines although they are not as desirable because of the possibility of introducing contaminants into the sodium system. Heat is not required during continuous trap operation, but is needed if trapping is intermittent and the cold trap sodium is permitted to cool until it is frozen.

7. Flow meters. Electromagnetic flow meters are used to measure the flow of sodium through a pipe line. A flow meter is an important device in a cold trap system. It shows the amount of sodium being purified and indicates the residence time of the sodium in the trap. The meter can also be used to measure the flow resistance of the trap. The flow resistance of a trap during most of its operating life is very small compared to that of the flow regulating valve. Therefore, the flow rate will be proportional to the valve opening. When a cold trap approaches the end of its useful life, the flow resistance increases rapidly and the relationship between valve opening and flow rate changes drastically.
8. Temperature indicators. The temperature of the sodium at the inlet of a trap must be known and must be maintained above the plugging temperature of the primary sodium. Otherwise, the oxide will precipitate from the sodium prematurely, and deposit in regions of the system outside the cold trap. The coldest sodium temperature in the trap must also be monitored so that the maximum oxide removal rate is not exceeded. A high rate may plug the trap prematurely. The Fermi cold trap had a vertical thermocouple well, extending from the region below the mesh to the outside of the trap (through the top head). A thermocouple placed in this well, measured

the temperature in the region below the mesh where the coldest sodium was located.

9. Capability for handling radioactive materials. Primary system cold traps should be designed to contain radioactive fission products. Even if vented fuels are not used, an accidental fuel pin rupture will release fission products to the primary sodium. The trap should be monitored for gamma rays so that it can be removed conveniently before the radiation level becomes too high.
10. Integrity. The cold trap must be tested for leaks. It should also be tested and certified for the maximum design temperature and pressures. The Fermi cold trap had an ASTM code stamp for an unfired pressure vessel.
11. Variable cooling rate. To establish the desired oxide removal rate in the trap, provision must be made for adjusting the flow and/or temperature of the coolant so that the rate of heat removal can be varied.

Desirable Design Criteria for Sodium Cold Traps

1. Low Cost. A single large unit will not necessarily cost less than several smaller units with a combined equivalent capacity. The higher labor costs incurred with multiple small traps would tend to be offset by the special heads,

jigging fixtures, structure thickness, and installation and removal costs of a single large unit.

2. Easy to remove from service, especially when radioactive.

Pipe and electrical connections should be easily disconnected. The trap should be located so it can be removed easily when necessary. Disposal of the radioactive cold trap should be considered. The most practical disposal method is burial on-site or at a commercial burial ground. Nuclear Engineering Co.* will accept radioactive cold traps for burial. However, Nuclear Fuel Services ** cannot legally handle sodium.

3. Reliable operation. Since cold traps are not examined frequently, they should operate reliably during their normal

lifetime. The collapsed wall and upheaved mesh in the Fermi cold trap are indications that it did not operate reliably.

Despite these two problems, the trap would probably have achieved its designed oxide accumulation provided it remained in service. If the trap had operated reliably, that result would have been more certain.

4. Self-venting. When molten sodium first enters a trap, it displaces gas which should be vented to the sodium discharge

*Nuclear Engineering Co., Moorhead, Kentucky.

**Nuclear Fuel Services, Box 124, West Valley, New York

line until the trap is almost full. A permanent gas reservoir space is necessary for sodium expansion in case the trap is isolated and heated.

5. Gravity draining. A capped valve should be installed at a low point of the cold trap system for gravity draining of the trap. The sodium then can be drained at about 250°F, with essentially all of the impurities remaining in the trap. This feature would be useful for salvaging the sodium in a spent trap, for preparing a spent trap for cleaning and reuse, and to facilitate trap removal.
6. Capable of intermittent operation. Unless there is a persistent leak of contaminants into the sodium, a cold trap is normally bypassed when high purity of the sodium is achieved (about 10 ppm oxygen). It should be possible to isolate a cold trap and allow its contents to freeze to reduce the operating cost of the cold trap cooling system.
7. Measurement of trap condition. The use of flow meters for indicating the end of trap life was described above under necessary criterion No. 7. However, since the pressure drop only starts to increase substantially just before the trap is to be removed, other methods of determining the amount of oxide in the cold trap would be desirable. Two direct

methods are: 1) weighing the trap; and 2) using a sensitive differential pressure meter between the inlet and outlet sodium lines. A less direct method would be to measure the plugging temperatures at the inlet and outlet of the trap, and use the sodium throughput to evaluate the amount of oxygen deposited. The least direct method would be to note the change in oxygen content of the primary system during cold trap operating periods. The disadvantage of the latter method is that temperature changes in the primary sodium would tend to nullify the results.

The design of the Fermi cold trap met most of the necessary and desirable criteria listed above. The design did not meet the following criteria:

Suitability for credible internal pressure

Operational reliability

Provision for direct measurement of trap condition

6.5.2 Recommended Design Improvements

Consideration of the dense oxide layer, the collapsed wall, and the upheaved mesh in the Fermi cold trap led to changes in the design of the trap as shown in Figure 28.

In a trap of this design, precooled sodium enters at the top and flows down the sides, as in the Fermi trap. The cross-sectional

FERMI SODIUM COLD TRAP

PROPOSED SODIUM COLD TRAP

PROPOSED CHANGES FOR FERMI COLD TRAP DESIGN

Figure 28

flow area is increased substantially by using radial flow from the outer wall of the trap to the center of the trap. The flow pattern is short-circuited at the top mesh area initially; but, as the sodium oxide accumulates there, the maximum flow pattern moves to a lower elevation. The cycle progresses in this manner until the trap is filled. The transient flow pattern and the large frontal area of the outside periphery of the mesh region are expected to contribute to a uniform, dense oxide distribution. In the Fermi cold trap, the upstream, six-inch layer of mesh collected oxides and other impurities very efficiently. Therefore, a long sodium flow path through the mesh region is unnecessary, and the shorter path in the proposed design should be adequate. Since this design eliminates the thin wall, the collapsed wall problem would not occur.

It is desirable to know if a cold trap is purifying the sodium as expected. It is necessary to know when its useful life is expended, so that a new trap can be substituted. The performance can be measured in several ways: 1) by monitoring the plugging temperature of the inlet sodium; 2) by determining impurities in the primary sodium by chemical analyses, 3) by suitable in-line electrical devices, such as a resistance meter or electro-chemical cell; or 4) by a differential pressure drop meter. If local plugging occurs, as happened in the Fermi trap, the sodium oxide can be

redistributed more uniformly by raising the temperature to a high value and maintaining it for a few days. During this period, the flow of sodium through the cold trap must be discontinued to prevent the oxide from returning to the primary system.

7.0 RECOMMENDATIONS FOR FURTHER STUDIES

Several additional studies are recommended to learn more about the performance of sodium cold traps:

1. A more comprehensive program of chemical analyses should be conducted to identify the specific compounds involved in the precipitation and concentration processes that occur in cold traps. The various chemical compounds associated with oxygen, carbon, hydrogen and nitrogen, as well as with copper and iron, should be identified. This will require the development of: 1) more specific analytical methods for determining oxygen, nitrogen and hydrogen in sodium; and 2) techniques for isolating and identifying the copper and iron compounds.
2. The solubility of impurities in sodium is of importance in the study of corrosion in sodium systems. Accurate solubility data for oxygen, hydrogen, nitrogen and metallic impurities would be very valuable in explaining how a cold trap works, in defining the limits of cold trapping as a purification technique, and in determining how the design of future cold traps can be improved.

A recommended study would consist of artificially loading each impurity of interest, separately and in combination, into a flowing isothermal sodium system, and determining the basic solubility limits of the impurities. The tests would be repeated for a range of temperatures.

3. A physical chemistry program should be conducted to determine the mechanisms of oxide precipitation and accumulation on metal surfaces.
4. A program should be conducted, in conjunction with the solubility and physical chemistry studies, to evaluate the effectiveness of cold traps, and the mechanism involved, in removing fission products and metallic impurities from radioactive sodium. Such a program could yield valuable information for designing advanced sodium cooled, fast reactors employing vented fuel elements. This recommendation is based on the fact that the SRE cold traps have accumulated fission products.
5. Examination of the EBR-II, Hallam, and SRE cold traps is recommended as a logical extension of the Fermi cold trap examination. The configurations of these traps are shown in Figure 29.

SODIUM COLD TRAPS

Figure 29

The EBR-II trap was removed from service because sodium could not be pumped through it. It is important to determine the amount and distribution of oxide precipitated in a plugged trap, such as the EBR-II trap. If the oxide distribution is uniform, the total amount of oxide would probably indicate the maximum capability of the EBR-II cold trap design and size.

The eight SRE cold traps were removed from service when their surface radiation values reached the 10-20 R/hr range. It is suggested that the fission products were associated with, and accumulated in, the high oxygen region of these traps. The information on fission product removal, obtained from examination of an SRE cold trap, would provide criteria for using cold traps to remove fission products from sodium.

The non-radioactive Hailam trap is of the same design as the radioactive SRE trap. Therefore, it could be used in the development of sampling techniques that may be needed to examine the SRE unit.

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9. Personal Communication with K. Barker, Mine Safety Appliance Research Company, Callery, Pa.

APPENDIX A - DEVELOPMENT OF SAMPLING PROCEDURE

The development of a suitable method for obtaining representative sample cores of sodium and mesh from the cold trap is described below. The desired samples had to be free of contaminants due to handling; they had to be undistorted; long lengths were desirable; and their diameter had to be suitable for physical and chemical examination

The following tools and techniques were tried and evaluated, in the course of developing the sampling procedure:

Pot metal mock-up	Elox
Hole saw	Straight drills
Cryogenic temperatures	Twist drills
Oscillating scissors	Auger drills
Screw retainer	Harpoon
Wax	Jackhammer

These tools and techniques were used with prototypes of the trap, to avoid disturbing the actual cold trap

The first prototype was a piece of 6-inch steel pipe, filled with 11-mil stainless steel wire mesh rolled to a density of 15 lb.

per cu. ft., and a low-melting pot metal. A short twisted sample was obtained with an ordinary 1-inch diameter hole saw used in the axial and transverse directions. A carbide-tipped hole saw (Figure 30) was substituted, but chips and metal particles clogged the cutting teeth. When the prototype was cooled to -150°F by liquid nitrogen (see Figure 30), the pot metal became very hard and a clean cut sample was obtained.

A second prototype was built with mesh and sodium and the carbide-tipped hole saw was tested on this prototype at room temperature, at -150°F , and at -300°F . Again, the results were unsatisfactory because the mesh in the sample was twisted and packed to a concentration of 50 w/o, whereas the initial concentration of the mesh was 22 w/o. The sodium consistency remained like putty, even at -300°F .

A more sophisticated tool was then designed and built. It consisted of a pair of concentric hole saws that oscillated with respect to each other to produce a scissor-like action. The inner and outer cutters had 12 triangular teeth each, with a 30° cutting angle between the teeth. The inner saw had a 1-inch inside diameter. When this tool was used on clean mesh alone, a satisfactory mesh core was obtained from the transverse direction. However, when only the inner hole saw oscillated or rotated, the mesh became twisted. When the tool was used on sodium alone, a 7.5-inch penetration resulted

Figure 30 Sampling Prototype with Carbide Tipped
Hole Saw and Liquid Nitrogen Refrigeration

in a sample 6.5 inches long, indicating a satisfactory sample. However, when this tool was tried on a combination of sodium and mesh, with the scissors oscillating at one cycle per second, an 8-inch penetration resulted in a core only 1-1/4 inches long. This sample contained 33% mesh, indicating a compacted condition. The mesh content was determined by dissolving the sodium in alcohol and weighing the mesh residue.

The third prototype consisted of mesh in four layers of sodium. Each layer contained a different mixture of sodium and sodium oxide, ranging from pure sodium to equal parts of sodium and sodium oxide. The mixtures were used to simulate the range of material expected to be found in the cold trap. Sodium, and mixtures of sodium and sodium oxide, were bought from commercial sources. Since they were received in a heterogeneous, rather than a homogeneous, condition the materials were screened, reweighed, and mixed with pure sodium. When the materials were heated to 300°F for the sodium to flow into the voids, the sodium oxide agglomerated and impeded succeeding operations. The final distribution of oxide was not established. Fine wires of different metals were laced with the mesh to serve as tracers for determining mesh movement during sampling. The first test on this prototype, using the oscillating scissors, resulted in a penetration of 1-7/16 inches and a sample 2-3/16 inches long. The sodium displaced by the tool had distorted and extended the sample.

A second test resulted in a 6-inch penetration and a 2-inch long sample, which was also unsatisfactory.

Before additional sampling, screws were inserted into the core region of the third prototype to prevent relative motion between the mesh and the sodium during coring. With a 6-inch long by 3/16-inch diameter screw in place, a 6-inch long core was obtained. However, this result could not be duplicated in later tests. The sodium-mesh adhered to the inner wall of the oscillating scissors during sampling and impeded movement of the sample into the tool. The problem was solved partially, but not satisfactorily, by adding a 1/16-inch relief at the cutting tip.

A fourth prototype was built with mesh, and with wax as hard as solid sodium. The purpose of this test was to employ a material that behaved like sodium but could be used more conveniently. An inert atmosphere was not required during melting. The oscillating scissors and a 10-24 bolt were tried in the mesh-wax prototype. The metal volume of the tool displaced the wax into the tool during sampling. Unfortunately, hardness was not the important sodium property to be prototyped. Sodium-mesh is difficult to core because it has high shear strength and adhesiveness. It was decided to confine all subsequent tests to sodium-mesh systems.

The oscillating scissors tool had the drawback of extruding sodium, displaced by the tool volume, through the desired sample.

A few 1/4-inch holes were drilled in the third prototype, along a 1-inch diameter circle, to provide relief for sodium displaced by the tool. A sample core, 2-1/4 inches long, was then obtained. A special boring jig would have been needed to achieve a deeper penetration without skewing, but this was considered to be impractical.

The use of electrical discharge machining (EDM) to obtain a suitable sample core was investigated. This technique was rejected because it required complex equipment and dielectric fluid to flush the separated particles.

A conventional 1/4-inch twist drill was used to extract chip samples from the third prototype. The full 6 inches were easily penetrated, and a clean hole was cut in the test pot. However, bits of sodium and mesh stuck to the drill and were hard to remove. Better results were obtained with straight flute drills (especially with a 1/2-inch drill). A 1-inch auger drill proved unsatisfactory when the mesh became tangled by the lead screw. Although various size drills could have penetrated the trap easily and quickly, their use was rejected because the resulting samples had three deficiencies: 1) each sample had a large surface area, and therefore required elaborate contamination protection; 2) the samples were not necessarily representative; and 3) the samples were not suitable for metallurgical-mount examination for particle size.

The harpoon or stud gun technique (Figure 31) was evaluated next. A blank cartridge was used to drive a tube, at high velocity, into the sample material. A .32-caliber shell drove a 1-1/8 inch diameter tube 1-1/2 inches into the third prototype. Using a regular .44-caliber shell resulted in a sample 2 inches long; a .44 magnum resulted in a 3-1/2 inch long sample. The tube penetrated another inch when a second .44 magnum shell was fired. A 3/8-inch diameter tube, driven by a .44 magnum shell, penetrated 6 inches of sodium-mesh. Tubes with weights added tended to penetrate more deeply. However, when the mass was substantially increased, a high velocity was not obtained and the penetration was shallow. All sample tubes were easily removed, leaving straight clean holes. In each case, the sample was about as long as the depth of penetration. A sample obtained by the harpoon technique was placed in a wire basket, and the sodium was dissolved gently in alcohol. The mesh that remained had been cut cleanly, and the layers were cleaned neatly (see Figures 32 and 33).

With multiple shots in the same hole, the harpoon technique could have achieved the two-foot penetration needed to reach the center of the Fermi cold trap. This technique, however, had the drawback that when the second tube was fired into the trap, it captured material from the walls made by the first shot. This foreign material mixed with material from the desired region; it also

Figure 31 Harpoon Sampling

Figure 32 3/8" Diameter Sodium-Mesh Sample
From Harpoon Prototype

Figure 33 Mesh Residue from Sample 9-2 After
Methanol Dissolution

retarded the penetration of the tube. To overcome this drawback, the hole was reamed with a 1-3/16 inch hole saw, but some material was still transferred.

To overcome the disadvantage of the harpoon technique, a simpler method was considered, namely, using an electric hammer to provide a series of small harpoon shots in quick succession. This technique, it was thought, would have the same advantage as the harpoon technique but would be simpler and faster. The first electric hammer test was made by driving a 1-1/8 inch tube into the third prototype. The tube easily penetrated 6 inches and produced a satisfactory core. This method was adopted as a standard procedure for sampling the Fermi cold trap. Figure 8 shows the experimental set-up.

Tubes with stainless steel tips, as well as hardened carbon steel tips, were used for sampling the trap (see Figure 3 for tip design). Although the carbon steel tips remained sharp longer, the stainless tips proved less apt to contaminate the sample. Therefore, stainless steel was selected as the preferred tip material. Despite only slight wear of the tip, a new tool was used to extract each sample. A tip was spot-welded in four places to the end of each tube and the weld was ground smooth. With the reduced inside diameter at the end of the tip, the radial clearance between the sample and the sampling tube was only 31 mils. This small clearance, however, permitted samples over two feet long to slide freely through the tube.

The section of the tip with the reduced inside diameter was 0.281 inches long. As a rule, this section had sufficient surface area to twist the sample free just before removing it from the trap. Some samples were cut so cleanly that when the tube was removed, the two-foot sample remained in the trap. To retrieve a complete core, a solid 1/16-inch diameter, stainless steel rod was inserted between the tube and the sample to lock them together

It was found that a 5 to 10 pound sledge hammer could be used more conveniently than an electric hammer. A 1-inch bolt with a large head was placed in the trailing end of the sampling tube and taped to it. This bolt provided a striking surface for the hammer, preventing distortion of the sampling tube. Each blow of the five-pound sledge hammer drove the tube in about 1 to 2 inches. Vacuum, applied to the inside of the tube, was tried to ease the movement of the sample into the tube. This technique was sometimes helpful, but was not useful enough for routine use.

The sledge hammer technique, described above, was selected and used to remove the sample cores from the Fermi cold trap.

APPENDIX B - SELECTED METHODS FOR CHEMICAL AND SPECTROGRAPHIC
ANALYSES, WITH REFERENCES FOR THE DETERMINATION OF CARBON,
HYDROGEN, NITROGEN, OXYGEN, AND METALLIC IMPURITIES IN SODIUM
SAMPLES FROM THE ENRICO FERMI COLD TRAP

1. GENERAL CONSIDERATIONS

The selection of analytical methods for determining impurities of interest in sodium samples from the Fermi cold trap was based on a review of the available literature, and on experience with methods currently used by laboratories engaged in chemical analysis of alkali metals. The metallic impurities in the sodium samples from the trap were determined by emission spectrography. The method of analysis, the equipment and calibration techniques, and the limits of detection for each element of interest are described completely in the attached copy of a tentative procedure used to examine the cold trap samples.

A comprehensive review of the literature resulted in the publication of "Annotated Bibliography for Analytical Methods for Alkali Metals", WCAP-2540. The analytical methods listed in this bibliography were evaluated for suitability in analyzing the sodium samples from the trap. Basic analytical procedures for this program were

selected from the methods listed. Each chosen analytical method was modified, to some extent, to produce more satisfactory results on the sodium samples processed during this work.

2. SELECTION OF ANALYTICAL METHODS

2.1 Total Carbon in Sodium

It was suspected that carbon concentration in the sodium samples from the trap ranged between 0.005% and 5%. This estimate was based on the carbon content in recent samples of primary sodium as reported by APDA, and on the total amount of carbon introduced into the primary sodium by outgassing of the graphite plug. Table 3 shows the analyses of the surface scum on the reactor sodium that resulted from this incident.

Three basic procedures for determining total carbon in sodium are reported in the literature. Pepkowitz and Porter¹ employed wet combustion following water dissolution of the sodium sample. The oxidizing agent was a mixture of chromic acid in sulfuric and phosphoric acid. The resultant CO₂ was measured manometrically. Several modifications of this basic procedure have been reported. However, in every case, questionable results were obtained because of a high, erratic reagent blank, and a lack of evidence that the procedure successfully oxidized all the carbon in the sample. Eng, Bingham, and Meyer² partially overcame these defects in the procedure by

Table 3

Analyses of Scum on Reactor Sodium

<u>December 1960</u>		<u>May 16, 1961</u>	<u>May 19, 1961</u>
Na ₂ O (as NaOH)	90.7%	NaOH 31.7%	NaOH 37.2%
Fe (as Fe ₂ O ₃)	7.6%	Na ₂ CO ₃ 47.6%	Na ₂ CO ₃ 40.7%
Silica	.34%	Carbon 20.6%	Carbon 13.2%
Cr	17 ppm	B .5 ppm	B none
K	24 ppm		
Ni	<1 ppm		
		Sodium at 800 to 1000°F Cover gas CO ₂ = 3.8% Plugging temperature = 325°F	Sodium at 800°F Cover gas CO ₂ = .25% Plugging temperature <240°F

adding a known amount of carbon-13 to the sample, and determining by mass spectrometry the ratio of carbon-12 to carbon-13 in the evolved CO_2 . Neither procedure, however, determined if elemental carbon was converted to carbonate carbon during water dissolution.

Kalman and Liu³ reported a method for determining total carbon in sodium metal, based on low temperature combustion of the carbon in the sodium sample to carbon dioxide which, in turn, forms sodium carbonate. Subsequent acid decomposition of the combined sodium carbonate releases the CO_2 , which is condensed in a liquid nitrogen trap and measured by a conductometric cell. The conductometric measurement used in this procedure is not sensitive enough for analyzing sodium samples that contain low concentrations of carbon. Hence, the procedure did not appear suitable for processing the large number of samples that had to be analyzed for this program.

The method finally selected for determining the total carbon in the sodium samples from the cold trap was a modification of the procedure reported by Stoffer and Phillips⁴, and incorporated some of the modifications of this same procedure as reported by Mungall and Johnson⁵, and as described in detail in APDA Report No. 164⁶. The carbon in the sodium sample is converted to sodium carbonate in a 10% oxygen-in-helium atmosphere. In the presence of silicon dioxide, the sodium carbonate is thermally decomposed at 1100°C . The carbon dioxide resulting from the decomposition of the sodium carbonate is condensed in a trap cooled by liquid nitrogen, with the

amount subsequently determined by gas chromatography. The precision of this method is approximately ± 5 ppm in the 10 to 250 ppm range. The recovery of known additions of carbon in the presence of sodium suggests that this analytical method is accurate to within $\pm 5\%$.

2.2 Carbonate Carbon in Sodium

Kalman and Liu³ described a procedure for acid decomposition of dissolved sodium carbonate with the evolution of carbon dioxide, which is condensed in a liquid nitrogen cooled trap and subsequently measured by a conductometric cell. A modification of this method was used to detect and measure carbonate carbon in the sodium samples from the Fermi cold trap.

The sodium sample was reacted with water at 0°C under argon. The water solution of sodium and sodium carbonate was then acidified with sulfuric acid, and the evolved CO_2 was swept into a liquid nitrogen cooled trap. Gas chromatography was used to detect and measure the carbon dioxide in the trap.

2.3 Sodium Oxide in Sodium

The concentration of sodium oxide in sodium samples from the Fermi cold trap was estimated to be in the range of 0.003% to 100%. The equivalent oxygen content of these samples could conceivably range from 0.001% to 25.8%. This estimate was based on a reported value of 10 ppm for the oxygen content of the primary sodium, and on

the assumption that areas of the cold trap could contain 100% sodium oxide.

Several methods for determining the sodium oxide content of sodium metal were reviewed. Pepkowitz and Judd⁷ were the first to report the use of the amalgamation procedure for separating sodium oxide from a sodium metal matrix. In this procedure, the sodium sample is treated with mercury in a suitable apparatus under an inert gas atmosphere until all the sodium metal is converted to an amalgam. The sodium oxide in the sample is not soluble, and floats on the surface of the amalgam. Repeated extractions with fresh mercury complete the separation of the sodium monoxide, which is dissolved in water and is measured by titrating the water solution with a standard acid. Although many refinements and modifications have been developed for the basic amalgamation procedure, the method is still deficient in that it is not specific for sodium monoxide. If the sodium sample contains sodium carbonate, sodium hydride, sodium hydroxide, or any other alkaline compounds insoluble in the amalgam, the results will be in error for true sodium monoxide content.

White, Ross, and Rowan⁸ substituted n-butyl bromide to separate sodium monoxide from sodium metal. This method was based on the fact that n-butyl bromide reacts with sodium to form sodium bromide, but does not react with sodium oxide. The sodium oxide residue is reacted with water, the phases separated, and the aqueous phase titrated with

standard nitric acid. This method, however, has the same deficiencies as the amalgamation method in that it is not specific for sodium monoxide. Other workers have substituted such solvents as ammonia and alcohol for mercury in separating the oxides from sodium, but all attempts have led to use of a non-specific analytical method. T. C. White⁹ employed vacuum distillation as a means of removing the sodium matrix from the residual oxides. This method eliminates the interference from sodium hydride, but would be subject to interference from elements that can reduce sodium monoxide.

On samples from the Fermi cold trap, an improved amalgamation procedure was used to separate the oxides from the sodium matrix. This method consisted of amalgamating and extracting the sodium in a simple separatory funnel inside an argon-filled glove box. A recirculating gas purification apparatus maintained the oxygen level below 1 ppm inside the glove box. The oxygen level in the glove box atmosphere was monitored by a W oxygen gauge calibrated against a certified mixture of oxygen in argon produced by Air Products Co. The separated oxides were reacted with water, and the alkalinity of the resultant solution was measured by potentiometric titration with a standard acid. Although this method is not specific for sodium monoxide, it did provide consistent results for "gross oxides". The precision of the method on aliquots from the same batch of sodium was ± 3 ppm in the 10-30 ppm range.

2.4 Nitrogen in Sodium

A modified Kjeldahl method for the determination of nitride nitrogen in sodium was reported by Ward, Gardner, et al.¹⁰, wherein the sodium sample was dissolved in ethanol in an argon atmosphere. The solution was then diluted with water and the nitrogen was distilled as ammonia. The ammonia was then absorbed in dilute boric acid solution and titrated with standard hydrochloric acid. Kirtchik and Melzer¹¹ attempted to demonstrate the function of nascent hydrogen in the formation and release of ammonia by reacting the sodium with phosphoric acid under hexane. They employed Nessler's reagent in conjunction with colorimetry, to measure the distilled ammonia. The method used by Westinghouse to determine nitrogen in sodium samples from the Fe-Mn lad trap incorporated features of both these methods. The sodium sample was reacted with methanol in an argon atmosphere, and the nitrogen was separated by steam distillation. Nessler's reagent was employed to develop the color from the resultant ammonia solution, and the color was measured on a Beckman D. U. Spectrophotometer. The average recovery on samples containing known amounts of nitrogen in the 5-100 ppm range was 95%. Figure 34 shows the laboratory apparatus used to determine nitrogen in sodium.

2.5 Hydrogen in Sodium

Several methods of determining the hydrogen content in sodium have been reported. Pepkowitz and Proud¹² used the selective diffusion

Steam Distillation Apparatus for Nitrogen Determination

Figure 34

of hydrogen through the walls of an iron capsule at 700°C to separate hydrogen from sodium in the 2%-100% range. This method, however, is not reliable for hydrogen in the 10-100 ppm range. Evans and Herrington¹³ employed the isotope dilution method, where a known amount of tritium-enriched hydrogen is allowed to contact the sodium sample. Equilibrium between the tritiated hydrogen and the hydrogen in the sample is established by heating the samples for 10 minutes at 420°C . Naud and Sanner¹⁴ reported the use of the amalgamation procedure (described above, in the oxygen determination procedure) to partially release the hydride and dissolved hydrogen from the sodium. The remaining hydride hydrogen is collected and measured during the water addition to the oxide residue.

Work at AFRL and WAFD indicates that a method based on vacuum refluxing of the amalgam will quantitatively release the dissolved and hydride hydrogen. Refluxing at approximately 700°F and one atmosphere of final pressure will indicate the hydroxide hydrogen. There is insufficient data to determine whether sodium hydroxide is decomposed partially or completely by this procedure. This method was used on most of the sodium samples from the cold trap. The results are expressed as total hydrogen, which includes the forms H_2 , NaH , and NaOH .

An alternate method, currently in the development stage at AFDA, was used to determine the hydrogen in two cold trap samples. This method is based on the principle that all the hydrogen in the

sodium will be evolved when the sample is heated to 700°F at a pressure of less than 1 micron. The evolved hydrogen diffuses through a palladium diaphragm, and is converted to water. The amount of water thus formed is then measured by the change in the weight of magnesium perchlorate located on the pan of an automatic electrobalance.

Both of these methods are still in the development stage. Calibration data is not available in sufficient quantity to warrant a statement here as to the accuracy of either method.

2.6 Spectrographic Analysis

A complete description of the spectrographic procedure for determining metallic impurities in the sodium samples from the Fermi cold trap is included in this report. This was considered necessary to provide background information for interpreting the results on iron, and copper in the high oxide area of the trap.

DETERMINATION OF IMPURITIES IN SODIUM BY OPTICAL
EMISSION SPECTROGRAPHY

I. ABSTRACT

This method provides for the determination of some 30 elements in sodium, in concentrations of 1 to 1,000 parts per million. The sample is hydrolyzed and converted to the chloride. It is then ground and mixed with an equal part of graphite and excited in a deep, thin-walled electrode with a programmed direct current arc. The spectra are recorded on a photographic plate by a large grating spectrograph. Standard spectra are recorded concurrently with those of samples. Standards are produced by blending fine NaCl with graphite containing varying amounts of impurities. Accuracies of 10 to 50 per cent of the amount present are attained, depending upon the element determined and its concentration.

II APPARATUS AND REAGENTS

A. Apparatus

1. 3.4-meter Ebert spectrograph, JACO #7132, with a 30-inch camera and a 14,400 line per inch Farrand grating of second order blaze.

2. Arc stand, JACO #1930, with controlled exhaust filter.
3. Photographic plate processing unit, JACO #3410.
4. Excitation source, NSL #KE 1234.
5. Recording microphotometer, NSL #MM-103.
6. Calculating board, Seidel, JACO #3001.
7. Mechanical Blender, SPEX Industries, #5000 Mixer Mill.
8. Electrode holder block, stainless steel for 1/8" electrodes.
9. Infrared heater, dual.

B. Reagents

1. Spectrographically-pure NaCl, vacuum-dried at 200°C.
2. Spectrographically-pure graphite, less than 200 mesh.
3. Four spectrographic standards in graphite, G standards, SPEX Industries #1002.
4. Electrodes graphite.
 - a Upper - 1/8-inch diameter, pointed with a 15 degree included angle.
 - b Lower - 1/8-inch diameter preformed with a 0.096 by 0.50-inch crater (UCP 7175 or equivalent).
5. Reagent grade Methanol.
6. Reagent grade Hydrochloric Acid

III PROCEDURE

A. Preparation of Standards and Standard Curves

1. Dilute each of the four graphite standards with graphite to obtain dilutions as per Table 4

Table 4

Schedule of Dilution of SPEX Industries - "G" Standards

Standards are 2 gram quantities of graphite containing oxides and carbonates of the 43 elements, Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, F, Fe, Ge, K, Ki, Mg, Mn, Mo, Na, Nb, Ni, P, Pb, Rb, Sb, Si, Sn, Sr, Ta, Th, Ti, Tl, U, V, W, Zn, and Zr.

G-1 = 1000 ppm of each element

G-2 = 100 ppm of each element

G-3 = 10 ppm of each element

G-4 = 1 ppm of each element

Add 0.4500 grams of each standard to 1.0500 grams of UCP-2 graphite to make the following series of standards.

	<u>Final Weight Grams</u>
G-1 = 1000 ppm	1.55
G-1-1 = 300 ppm	1.50
G-2 = 100 ppm	1.55
G-2-2 = 30 ppm	1.50
G-3 = 10 ppm	1.55
G-3-3 = 3 ppm	1.50
G-4 = 1 ppm	1.55
G-4-4 = 0.3 ppm	1.50
Blank	

2. Blend one part of NaCl with one part of each graphite standard in a plastic vial, using the mechanical blender with three 1/8-inch Lucite ball pestles.
3. Weigh 50 mg of this mix into a lower electrode (UCP 7145), and tamp the charge firmly.
4. Heat all charged electrodes in a stainless steel block under infrared lamps at 200°C for 15 minutes before starting exposures.
5. Under the conditions in Table 5, obtain a spectrogram for the ultraviolet region.
6. Under the conditions in Table 6, obtain a spectrogram for the visible region.
7. Against the clear developed emulsion, measure the per cent transmission of the line of the element of interest in Table 7.
8. Plot the per cent transmission as a Seidel function against the concentration of each element to obtain standard curves.

B. Analysis of Samples

1. From 5 to 10 grams of sodium metal are placed in a 400-ml polypropylene beaker. Reagent grade methanol is added in 10-ml aliquots until dissolution is complete. If there is any stainless steel mesh or inclusions of metallic nature, the sodium methylate solution is filtered through a 100-mesh nylon screen and the filtered material is washed with deionized water. The mesh or foreign residue, is then placed in a plastic vial, and a dilute HCl (0.001N) solution is added to the mesh

Table 5

Procedure - Impurities in Sodium as NaCl - Ultraviolet Region

Sample Conditions

Wgt. 50 mg
Electrodes: Upper - 1/8" pointed 15° angle
 Lower - UCP 7175
Tamped
Preheated 15 min. at 200°C

Source

DC arc
Hi-lo Switch on
Timer - 15 sec.
Resistance Ratio - 4:1

Source Conditions

Current - 6 to 14 amps programmed
Pretimer - 0 sec.
Total timer - 60 sec.

Spectrographic Conditions

Plate holder - three
Emulsion (L)SA-1, (C)SA-1, R(SA-1)
Focus - 18 mm
Slit angle - 1.5 R
Slit width - 10 microns
Slit height - 2 mm

Anal. Gap - 2 mm

Draft - .01 inch

Filter - none

Time on sector - 30%

Grating mask - 2"

Grating angle 10.75°

Order - second

Developing Conditions

Temperature 68°F

Developer - D-19 for 3 min.

Fixer - X-ray for 3 min.

Stop bath - 5% acetic for 30 sec.

Washing time - 15 min.

Photoflow - 1 min.

Densitometer

Scanning speed .17 mm/min.

Slit width - 12 microns

Note: Source; N.S.L. KE 1234
Spectrograph; JACO 7132 3.4 meter Ebert
Densitometer; N.S.L. MM 103

Table 6

Procedure - Impurities in Sodium as NaCl - Visible Region

Sample Conditions

Wgt. 50 mg
Electrodes: Upper 1/8" pointed 15° angle
 Lower - UCP 7175
Tamped
Preheated 15 min. at 200°C

Source

DC arc
Hi-lo Switch on
Timer - 15 sec.

Source Conditions

Current - 6 to 14 amps programmed

Spectrographic Conditions

Plate holder - three
Emulsion (L)IN, (C)IN, R(IN)
Focus - 18 mm
Slit angle - 1.5 R
Slit width - 10 microns
Slit height - 2 mm

Anal. gap - 2 mm
Draft - .01 inch
Filter - corning 3389
Grating mask - 2"
Grating angle - 11.00°

Developing Conditions

Temperature - 68°F
Developer - D-19 for 3 min.
Fixer - X-ray for 3 min.
Stop bath - 5% acetic for 30 sec.
Washing time - 15 min.
Photoflow - 1 min.

Densitometer

Scanning speed - .17 mm/min.
Slit width - 12 microns

Note: Source; N.S.L. KE 1234
Spectrograph; JACO 7132 3.4 meter Ebert
Densitometer; N.S.L. MM 103

Table 7

Impurities in Na As NaCl

<u>Element</u>	<u>Est. Limit of Detection</u>	<u>Wave Length A</u>	<u>Element</u>	<u>Est. Limit of Detection</u>	<u>Wave Length A</u>
Ag	1	3280.7	Na		
Al	10	3082.1	Nb	800	3094.2
As			Ni	10	3012.0
Au			P		
B	30	2497.7	Pb	30	2833.1
Ba	3	4554.0	Rb	10	7947.6
Be	30	3131.1	Sb	250	2598.1
Bi	10	3067.7	Si	10	2516.1
Ca	3	3968.5	Sn	30	3175.0
Cd	50	3261.1	Sr	3	4607.3
Co	3	3453.5	Th	800	2837.3
Cr	3	2849.8	Ti	30	3372.8
Cs	20	8521.1	Tl		
Cu	3	3247.5	U		
Fe	10	2598.4	V	10	3184.0
In	50	3256	W	800	2946
K	30	7968.0	Zn	100	3302.6
Li	1	6707.8	Zr	20	3438.2
Mg	3	2795.5			
Mn	2	2794.8			
Mo	10	3132.6			

Note: Limits of detection may vary slightly with photographic emulsion.

for three minutes, at room temperature, to ensure removal of any adhering oxide film. The dilute HCl is then added to the filtered sodium methylate solution through the 100-mesh nylon screen. A few drops of phenolphthalein are added to the sodium methylate, and enough deionized water is added to hydrolyze the sodium methylate. Concentrated HCl is then added until the solution is acid, with approximately 1-ml of excess HCl. The solution is then evaporated under infrared lamps. When the sodium chloride is visually dry, it is transferred to tared 250-ml Pyrex beakers and dried in an oven at 130°C for one hour. The beakers are cooled and reweighed, and the sodium chloride is transferred into plastic vials with 3/8" diameter plastic balls, and is blended for two minutes.

2. Proceed with steps 2 through 7 of the Preparation of Standards and Standard Curves, making three exposures of each sample.

C. Calculations

1. Determine the average per cent transmission of the three exposures for each analytical line of interest.
2. Determine the concentration of each element, based on NaCl from the simultaneously prepared standard curve.
3. Multiply the results by 2.5, and report in parts per million based on mesh-free sodium metal.

IV. PRECISION AND ACCURACY

Accuracies of 10 to 50 per cent of the amount present are estimated.

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APPENDIX C - PHYSICAL APPEARANCE OF SAMPLE CORES

Figures 35 through 41 show photographs of typical sample cores removed from the cold trap. The layout of the photographs corresponds to the sample locations in the trap. In each figure, the same cores are shown in their vertical order for a half plane of the trap. Thus, the figures provide a representative view of the contents of the trap. Brief observations of the sample cores follow.

Figure 35 shows samples 6-6, 7-1 and 4-2. Sample 4-2 was removed from the relatively pure sodium found below the mesh region. Although sample 7-1 was removed from the mesh region, it actually contained no mesh. Sample 6-6 has a 1-inch hole where a 1/2-inch diameter specimen was removed.

Figure 36 shows samples of the material removed from the inlet and outlet pipes of the trap.

Figures 37 through 41 show samples from half planes 1 through 4. Sample 48-2 was located closer to the center of the trap than 48-1. The series of samples, 32-1 to 41-1, were removed from a region adjacent to the series of samples, 22-1 to 31-1. (Note the black areas on samples 39-1 and 25-1).

6-6

THIS END WAS TOWARD
OUTSIDE OF TRAP

7-1

4-2

SAMPLES FROM HALF PLANE I

Figure 35

55
OUTLET

THIS END WAS TOWARD
OUTSIDE OF TRAP

20
INLET

SAMPLES FROM INLET AND OUTLET PIPES

Figure 36

THIS END WAS TOWARD
OUTSIDE OF TRAP

49-1

48-2

48-1

47-1

46-1

SAMPLES FROM HALF PLANE I

Figure 37

SAMPLES FROM HALF PLANE 4

Figure 38

THIS END WAS TOWARD
OUTSIDE OF TRAP

53-1

52-1

51-1

50-1

SAMPLES FROM HALF PLANE 2

Figure 39

SAMPLES FROM HALF PLANE 3

Figure 40

SAMPLES FROM HALF PLANE 3

Figure 41

APPENDIX D - EXAMINATION OF THE MESH IN SAMPLE CORES

As part of the physical examination of the trap contents, and to determine the oxide precipitation mechanism, the impurities on the mesh were examined by the following techniques.

1. An examination of the mesh, after the sodium had been removed by alcohol dissolution, showed no appreciable amount of oxide on the mesh. The agitation produced by the gas bubbles accompanying the alcohol-sodium reaction may have mechanically displaced any oxide deposits from the mesh. Both methanol and butanol were used in these experiments.
2. An examination of the mesh after liquid ammonia dissolution of the sodium showed sodium oxide deposits adhering to the mesh. This experiment was repeated on a sample extracted from hole No. 14, where the oxygen content ranged between 3 and 6%. The oxide-covered mesh was mounted in clear epoxy resin* and examined at 15 X magnification. Figure 42 shows the general appearance of the mesh after ammonia dissolution. Based on a wire diameter

* Maraset Type A resin
The Marblette Corporation, Long Island City, New York

Figure 42 Sodium Oxide on Wire Mesh

of 11 mils, the average coating thickness was estimated at 3 mils. Sodium samples from other parts of the trap were treated similarly, and the mesh showed varying degrees of oxide buildup. In most cases, only a thin black film was evident.

3. A vacuum distillation technique for removing sodium from the mesh was evaluated. A sample of sodium, supported on a wire frame inside a cylindrical glass vessel, was heated at a pressure of 50 microns to slightly above the melting point of sodium. After the sodium melted and deposited in the bottom of the vessel, examination showed a voluminous black residue adhering to the mesh. Attempts to mount the mesh sample in Maraset Type A epoxy resin resulted in extensive bubble formation in the mount because the sodium-sodium oxide residue probably accelerated the epoxy polymerization reaction. The resulting epoxy mount was not suitable for microscopic examination of the mesh. Controlled distillation at a lower pressure might have yielded better results on these epoxy-mounted samples. Rapid distillation of the sodium by induction heating was considered, but it was not attempted because sufficient temperatures could have been generated in the mesh, even with its low susceptibility, to volatilize the sodium oxides or, possibly, to react with the oxide residue.

All the samples examined exhibited a similar pattern of oxide deposit on the mesh. A fairly uniform coating was observed on the wire, with a fillet or web type buildup at the intersection of the wires. As the coating thickness approached a few mils, the deposit had a fuzzy structure and a less dense appearance. This indicates the possibility of direct crystal growth on the wire. These experiments were not sufficiently sophisticated to prevent mechanical displacement of sodium oxide particles from sites where bridging might have occurred. Metal-lurgical examination of the stainless steel mesh samples from the cold trap showed no evidence of surface corrosion or inter-granular attack.

APPENDIX E - EXAMINATION BY GAMMA RADIATION SURVEY TECHNIQUE

The examination of the Fermi cold trap by gamma radiation survey, and the results of this experiment, are described below. The experiment was performed by Applied Health Physics, Inc. of Bethel Park, Pennsylvania. The results were analyzed by Westinghouse.

I. Examination

The first step in the procedure was to establish the points where the readings would be taken. The method used to locate these points was as follows. The cylindrical surface area of the outer wall of the trap was divided into four faces, I through IV, of equal area. The vertical boundaries of each face were the pertinent two of the trap's four vertical support columns. The horizontal boundaries of each face were defined by the top and bottom dished heads of the trap, with a grid of vertical and horizontal lines marked on each face. Each intersection of the grid formed a point for a reading. The elevation of each horizontal grid line and its code letter are shown in the lower view of Figure 43. The upper view of this figure shows the location and code numbers of the vertical lines in each face. Thus, each grid intersection was

ENRICO FERMI COLD TRAP, GAMMA SURVEY

Figure 43

coded, e.g., A-5. The grid was centered on each face except on Face III, where it was shifted 3 inches towards Face II so that the sample hole plugs would not interfere with the detector. The location of each face is also identified below by relating each face to the sample holes and the thermocouple tube:

Face I was nearest the thermocouple tube

Face II contained sample holes 2 through 7, 9, 10, 11, 18, and 46 through 49

Face III contained sample holes 8, 12 through 17, and 50 through 53

Face IV contained sample holes 22 through 41, and hole 54.

Before taking readings, the following safety precaution was observed. The 68.22 mc radium source of gamma radiation employed Ra^{226} in equilibrium with its daughter products. When this source was positioned in the thermocouple tube of the trap, the maximum gamma radiation at the outer wall of the trap was 80 mr/hr. The area was restricted around the steel room that housed the trap. Outside of the restricted area, radiation measured less than 0.2 mr/hr.

A reading was taken at each intersection of the grid on each face. For each reading, the source and the detector were positioned at the same level of the trap. Each trap face was scanned completely before taking readings on the next face. The following steps

illustrate the scanning procedure employed. The source was positioned at the N level in Face I, and the radiation was measured at the five points on this level. The source was then moved to the M level in Face I, and the radiation was measured at the five points on this level. This procedure was repeated until Face I had been completely scanned. Before scanning each face, an equipment calibration point, based on the average of twenty one-minute counts, was established on the face at the 5-N location. Whenever three levels (say L, M, N) of one face had been scanned, the calibration point was rechecked to confirm the stability of the equipment.

In addition to the radiation source mentioned above, the following equipment was employed in performing this experiment:

<u>Item No.</u>	<u>Description</u>
1	CS-200 Amplifier Analyzer and High-Voltage Supply
2	CS-905 Timer
3	Scaler and Counter
4	CS-210 Pre-Amplifier (Employing an End-Window GM Tube)
5	Victoreen Survey Meter, Model AGB-50B SR

A Sola power transformer was also employed to increase the stability of the equipment.

II. Results

The data obtained from this experiment is shown graphically in Figures 44 through 47, with one graph for each face. Each graph shows the gamma count rate detected at each point. In these graphs and in the discussion below, the vertical grid lines are referred to as "columns". The count rate is a function of the radiation transmission between the source, located in the thermocouple well, and the detector, located at the reading point on the outer surface. Since sodium oxide is denser than sodium, the transmission through high oxide regions should have been less than through low oxide regions. An attempt to interpret this data proved unsuccessful because the gamma radiation, measured at the outer surface of the trap, could not be related to the distribution of sodium oxide in the trap. The difficulty was caused mainly by gamma radiation scattering and buildup effects.

Below are some general observations on the data from each face.

Face I (see Figure 44)

The count rate varied widely at each trap level along different columns. Although column 3 was nearest the radiation source, data along this column was severely depressed between trap levels B and H. Similarly, although columns 2 and 4 were closer to the source than columns 1 and 5, the count rates along columns 2 and 4 were less

-941-

Figure 44

- 147 -

Figure 45

GAMMA SCAN TEST, FACE III

Figure 46

GAMMA SCAN TEST, FACE IV

Figure 47

than those along columns 1 and 5. The severe drop at levels B through H in the count rate along columns 2, 3, and 4 suggested the presence of a structural member. However, examination of the engineering drawings for the trap did not reveal a structural member in this region.

A severe drop in the count rate was not observed at the M level in Face I as it was in the other faces. In Face I, this was probably due to the proximity of the points on the M level to the source, which precluded scattering and shielding.

Face II (see Figure 45)

The reason for the non-horizontal trends in columns 4 and 5 was not determined. The relative intensity of radiation along the five columns corresponded with the distance of each column from the source.

Face III (see Figure 46)

The depression of the count rate at the M level as detected along all five columns was probably caused by the presence of the lower support plate at this level. Peaking at levels L and N was caused by scattering and reflection of the gamma rays from the M level support plate into the detector. Unfortunately, the region where the largest variation in material was expected proved to be that where the greatest perturbation in count rate occurred from

the buildup and scattering effects. The peaking at level A was probably caused by reflection from the dished head at the top of the trap, and from lower density material in this mesh-free region.

Face IV (see Figure 47)

Gamma flux peaking at levels A, L, and N, as well as the depression at level M, were noted in Face IV, the same as in Face III, and can be explained in a similar way. Since Face IV was a mirror image of Face II, data from these two faces were expected to have a corresponding relationship. The comments given above for face II are also applicable to Face IV.

APPENDIX F - ANALYTICAL RESULTS

This appendix presents the analytical results in Figures 48 through 64, and in Tables 8 through 11.

Figures 48 through 64 show the vertical and radial distribution of impurities found in the Fermi cold trap. In these figures, the data points are located along the sample cores at the approximate radial trap location of the analytical samples; the sample cores are located at the approximate vertical trap locations of the analytical samples and the half plane origin of each sample core is noted. The analytical sample is defined as the portion of the sample core used in each analysis.

Table 8 gives the amount of impurity determined for each analytical sample, and identifies the location of each sample in the trap. Table 9 gives the weight of each analytical sample used for each determination; this table also gives the location of each sample in the trap.

As background information, Table 10 gives the results of the chemical and spectrographic analyses of the Fermi primary sodium that were performed by APDA, Dupont and LeDoux, between July, 1959

and May, 1961 before examination of the Fermi cold trap was started. The values obtained for carbon and uranium are particularly interesting.

Table 11 gives the amount of metallic impurities determined by spectrographic analyses of samples from the cold trap and from the Fermi primary sodium.

The data is presented in a manner that provides a clear understanding of the location of the analytical sample inside the trap, and facilitates comparison of concentrations of the different impurities at approximately the same axial and radial positions. The location of the sample core in the trap can be derived by noting the half plane involved and the relative vertical elevation from Figure 11 and Table 1. Data in Tables 8 and 9 are presented by hole number and sub-sample number. The numbering system used to identify the trap locations of the analytical samples is based on assigning the number (1) to the specimen nearest the external wall of the trap in each sample core, and progressively higher numbers toward the center of the trap.

OXYGEN DISTRIBUTION HOLES 22-31,46-49

Figure 48

OXYGEN DISTRIBUTION HOLES 32-41,46-49

Figure 49

OXYGEN DISTRIBUTION HOLES 42-45,50-53

Figure 50

TOTAL CARBON DISTRIBUTION HOLES 22-31,46-49

Figure 51

TOTAL CARBON DISTRIBUTION HOLES 22-31,46-49

Figure 51

TOTAL CARBON DISTRIBUTION **HOLES 42-45,50-53**

Figure 52

CARBONATE CARBON DISTRIBUTION HOLES 32-41,46-49

Figure 54

CARBONATE CARBON DISTRIBUTION HOLES 42-45,50-53

Figure 55

HYDROGEN DISTRIBUTION HOLES 22-31,46-49

Figure 56

HYDROGEN DISTRIBUTION HOLES 42-45,50-53

Figure 57

NITROGEN DISTRIBUTION HOLES 22-31,46-49

Figure 58

NITROGEN DISTRIBUTION HOLES 32-41,46-49

Figure 59

NITROGEN DISTRIBUTION HOLES 42-45,50-53

Figure 60

COPPER AND IRON DISTRIBUTION HOLES 32-41,46-49

Figure 61

**MESH DISTRIBUTION
HOLES 32-41,46-49**

Figure 63

MESH DISTRIBUTION HOLES 42-45,50-53

Figure 6h

Table 8

Analytical Results

Hole No.	Impurity Measured	Sample Site Number									
		1	2	3	4	5	6	7	8	9	10
20	Total Carbon										
	Carbonate Carbon	18									
	Total Hydrogen										
	Nitrogen										
	Oxygen	34		29							
22	Percent Mesh	0		0							
	Total Carbon	87	25	72	72	66					
	Carbonate Carbon	20		6		53					
	Total Hydrogen			7							
	Nitrogen										
23	Oxygen	32	34	45	30	54					
	Percent Mesh	0	0	0	0	0					
	Total Carbon	100			13		24		14		99
	Carbonate Carbon	0.09%					4		9	4	14
	Total Hydrogen						13				
24	Nitrogen	6	8								
	Oxygen	13.5%	13.4%	11.1%	143	0.33%	1.5%	0.12%	46	33	55
	Percent Mesh	14.4%	0	0	0	0	0	0	0	0	0
	Total Carbon	0.06%	495	39	34	28					
	Carbonate Carbon	251		20		7					
25	Total Hydrogen										
	Nitrogen										
	Oxygen	7.1%	9.2%	15.8%	12	23					
	Percent Mesh	18.2%	16.4%	0	0	0					
	Total Carbon	0.09%			0.09%		0.06%		0.20%		290
26	Carbonate Carbon	286				239					427
	Total Hydrogen						0.67%				
	Nitrogen	61	57				(0.54%)				
	Oxygen	50.7%	7.5%	6.0%	6.1%	10.8%	10.3%	3.3%	2.3%	3.2%	1.5%
	Percent Mesh	15.7%	23.0%	23.8%	23.5%	16.1%	20.4%	22.2%	29.4%	25.3%	27.4%
27	Total Carbon	365	385	0.08%	0.12%	317					
	Carbonate Carbon	199		245		403					
	Total Hydrogen										
	Nitrogen										
	Oxygen	3.6%	4.0%	3.9%	3.2%	6.2%					
28	Percent Mesh	10.3%	14.6%	19.5%	22.1%	19.6%					
	Total Carbon	201	230	301	225	386					
	Carbonate Carbon	101		207		78					
	Total Hydrogen										
	Nitrogen										
29	Oxygen	1.6%	1.4%	0.66%	1.0%	1.6%					
	Percent Mesh	37.2%	18.8%	27.7%	28.0%	27.0%					
	Total Carbon	154	168	37	80	210					
	Carbonate Carbon	29		21		19					
	Total Hydrogen										
30	Nitrogen										
	Oxygen	0.35%	0.58%	0.40%	0.63%	0.40%					
	Percent Mesh	18.2%	20.6%	15.6%	24.0%	17.6%					
	Total Carbon	68	74	80	98	116					
	Carbonate Carbon	22		18		32					
31	Total Hydrogen										
	Nitrogen										
	Oxygen	0.21%	0.37%	0.32%	0.28%	0.31%					
	Percent Mesh	20.0%	16.7%	18.7%	20.7%	16.9%					
	Total Carbon	135	186	138	70	482					
32	Carbonate Carbon	22		47		45					
	Total Hydrogen										
	Nitrogen										
	Oxygen	0.07%	0.20%	0.12%	0.24%	0.11%					
	Percent Mesh	17.8%	21.7%	17.3%	18.0%	20.4%					

Table 8 (Cont)

Hole No.	Impurity Measured	Analytical Results									
		1	2	3	4	Sample Site Number					
						5	6	7	8	9	10
31	Total Carbon	18	22	24	39	28					
	Carbonate Carbon	3		3		3					
	Total Hydrogen										
	Nitrogen										
	Oxygen	40	39	28	27	61					
	Percent Mesh	0	0	0	0	0					
32	Total Carbon										
	Carbonate Carbon	22	38	19	14	16					
	Total Hydrogen										
	Nitrogen	5	14	9	12	11	9	3			
	Oxygen	57	62	33	45	32					
	Percent Mesh	0	0	0	0	0					
33	Total Carbon										
	Carbonate Carbon	494	0.05%	24	20	51					
	Total Hydrogen										
	Nitrogen	10	9	2							
	Oxygen	11.9%	13.8%	0.04%	0.01%	0.03%					
	Percent Mesh	0	0	29.6%	0	0					
34	Total Carbon										
	Carbonate Carbon	375	340	240		80					
	Total Hydrogen										
	Nitrogen										
	Oxygen	8.0%	6.0%	4.4%	5.8%	0.89%					
	Percent Mesh	15.8%	16.7%	15.2%	22.3%	0					
35	Total Carbon										
	Carbonate Carbon	0.12%	0.07%	490	390	273					
	Total Hydrogen										
	Nitrogen	6									
	Oxygen	8.0%	12.1%	4.1%	4.2%	2.2%					
	Percent Mesh	20.6%	17.6%	36.8%	26.1%	27.9%					
36	Total Carbon										
	Carbonate Carbon	340	0.06%	0.06%	0.05%	340					
	Total Hydrogen										
	Nitrogen										
	Oxygen	7.1%	5.3%	6.6%	5.9%	1.6%					
	Percent Mesh	28.2%	34.4%	34.6%	20.8%	24.0%					
37	Total Carbon										
	Carbonate Carbon	144	245	154	103	98					
	Total Hydrogen										
	Nitrogen	102									
	Oxygen	1.5%	2.2%	1.6%	0.69%	0.48%					
	Percent Mesh	25.7%	25.1%	18.8%	28.2%	31.7%					
39	Total Carbon										
	Carbonate Carbon	57	63	76	116	56					
	Total Hydrogen										
	Nitrogen	53									
	Oxygen	0.17%	0.45%	0.40%	0.25%	0.32%					
	Percent Mesh	20.5%	21.4%	15.1%	23.6%	21.3%					
40	Total Carbon										
	Carbonate Carbon	41	119	53	85	56					
	Total Hydrogen										
	Nitrogen										
	Oxygen	0.15%	0.16%	0.20%	0.34%	0.14%					
	Percent Mesh	11.1%	20.4%	18.1%	20.7%	16.3%					
41	Total Carbon										
	Carbonate Carbon										
	Total Hydrogen										
	Nitrogen										
	Oxygen	39	20	<10	31	27					
	Percent Mesh	0	0	0	0	0					

Table 8 (Cont.)

Analytical Results											
Hole No.	Impurity Measured	Sample Site Number									
		1	2	3	4	5	6	7	8	9	10
42	Total Carbon	0.21%	52	43							
	Carbonate Carbon										
	Total Hydrogen		15								
	Nitrogen	24	30								
	Oxygen	15.5%	41	15							
	Percent Mesh	0	0	0							
43	Total Carbon	0.08%	444	45							
	Carbonate Carbon										
	Total Hydrogen	358	0.08%	21							
	Nitrogen	73	27								
	Oxygen	4.8%	4.1%	23							
	Percent Mesh	0	0	0							
44	Total Carbon	315	0.14%	0.14%							
	Carbonate Carbon										
	Total Hydrogen	353	0.11%	0.07%							
	Nitrogen	45	82								
	Oxygen	4.7%	4.1%	23.0%							
	Percent Mesh	23.5%	29.7%	3.7%							
45	Total Carbon	120		164							
	Carbonate Carbon	87		31							
	Total Hydrogen			25	(115)						
	Nitrogen	10		23							
	Oxygen	0.09%		0.13%					0.24%		
	Percent Mesh	20.8%		18.5%					20.4%		
46	Total Carbon	75		92							
	Carbonate Carbon	3		4							
	Total Hydrogen			6							
	Nitrogen	1	6	30	25						
	Oxygen	29		25							
	Percent Mesh	0		0							
47	Total Carbon	0.43%	0.13%	0.16%							
	Carbonate Carbon	0.05%	100	0.04%							
	Total Hydrogen	0.21%	46	0.24%							
		(0.38%)*		(1.1%)*							
	Nitrogen	65	29	49	42						
	Oxygen	14.1%	8.2%	9.5%							
48	Percent Mesh	16.9%	0	0							
	Total Carbon	0.07%	0.15%	0.08%							
	Carbonate Carbon	21	164	138							
	Total Hydrogen	0.11%	0.07%	0.05%							
	Nitrogen	66	49	55	86						
	Oxygen	4.4%	5.9%	5.3%							
49	Percent Mesh	37.8%	18.4%	25.4%							
	Total Carbon	179		203							
	Carbonate Carbon	30		21							
	Total Hydrogen			9							
	Nitrogen	11	10	10	37						
	Oxygen	148		0.12%					0.30%		
50	Percent Mesh	19.1%		23.4%					20.5%		
	Total Carbon	104	44	41							
	Carbonate Carbon										
	Total Hydrogen										
	Nitrogen	5	9								
	Oxygen	9.9%	21	207							
51	Percent Mesh	0	0	0							
	Total Carbon	341	72	51							
	Carbonate Carbon										
	Total Hydrogen										
	Nitrogen	11	11								
	Oxygen	16.0%	101	90							
	Percent Mesh	10.3%	0	0							

Table 8 (Cont.)

<u>Analytical Results</u>										
<u>Hole No.</u>	<u>Impurity Measured</u>	<u>Sample Site Number</u>								
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u> <u>10</u>
52	Total Carbon	0.31%	83	0.13%						
	Carbonate Carbon									
	Total Hydrogen									
	Nitrogen	56	26							
	Oxygen	3.0%	221	6.4%						
	Percent Mesh	27.5%	0	13.4%						
53	Total Carbon	142	97	68						
	Carbonate Carbon									
	Total Hydrogen									
	Nitrogen	29	21							
	Oxygen	114	0.15%	0.27%						
	Percent Mesh	21.0%	17.8%	20.2%						
54	Total Carbon									
	Carbonate Carbon	43		55	77	63				
	Total Hydrogen									
	Nitrogen									
	Oxygen	0.50%	0.29%	0.22%	0.36%	0.37%				
	Percent Mesh	24.7%	16.4%	20.2%	23.3%	30.5%				
55	Total Carbon									
	Carbonate Carbon	16								
	Total Hydrogen									
	Nitrogen									
	Oxygen	26	25	20						
	Percent Mesh	0								

NOTES:

- (a) All results reported in parts per million impurity in mesh-free sample unless otherwise stated.
- * (b) Results for hydrogen in parentheses were obtained by catalytic oxidation procedure.
- (c) A typical calculation for obtaining an oxygen value and mesh percentage on a sample from the lower mesh area is shown below:

- (1) wt. of sample - 2.3010 grams
- (2) wt. of mesh - 0.4180 grams
- (3) wt. of mesh-free sample - 1.8830 grams
- (4) wt. of oxygen found by amalgamation procedure - 0.1330 grams

$$(5) \text{ wt. percent mesh in sample} = \frac{\text{wt. mesh}(2)}{\text{wt. sample}(1)} \times 100 = 18.2\%$$

$$(6) \text{ wt. percent oxygen in mesh-free sample} = \frac{\text{wt. oxygen}}{\text{wt. mesh-free sample}} \times 100 = 7.1\%$$

Table 9

Weights of Analytical Samples

Hole No.	Sodium Wt.* (g)	Sample Site Number									
		1	2	3	4	5	6	7	8	9	10
20	Carbon										
	CO ₃ -Carbon	1.09									
	Hydrogen										
	Nitrogen										
	Oxygen	1.09		1.18							
	Mesh										
22	Carbon	0.1257	0.1354	0.1610	0.1189	0.1343					
	CO ₃ -Carbon	1.2130		1.1510		0.9210					
	Hydrogen			0.2180							
	Nitrogen										
	Oxygen	1.3100	1.3040	1.6230	1.3230	1.6760					
	Mesh	0	0	0	0	0					
23	Carbon	0.0823			0.2734		0.2004		0.2703		0.2238
	CO ₃ -Carbon	0.5649					1.4220		0.7380	0.6820	0.8510
	Hydrogen						0.4250				
	Nitrogen	14.4060	14.5260								
	Oxygen	1.2830	2.1170	1.6630	1.1590	1.0780	1.0550	1.6980	0.8680	1.1460	1.1010
	Mesh	0	0	0	0	0	0	0	0	0	0
24	Carbon	0.1723	0.1896	0.1296	0.1311	0.1447					
	CO ₃ -Carbon	1.8910		1.5590		1.4490					
	Hydrogen										
	Nitrogen	14.5260	14.4060								
	Oxygen	1.8830	2.0310	2.5600	2.8470	1.8560					
	Mesh	0.4180	0.3970	0	0	0					
25	Carbon	0.1660			0.1889		0.1220		0.0697		0.1548
	CO ₃ -Carbon	1.1860				1.4590					1.3345
	Hydrogen						0.0906				
							(0.0072)*				
	Nitrogen	19.1060	24.0754								
	Oxygen	1.6680	1.3795	1.2820	1.6740	1.2590	1.1450	1.4000	1.2840	1.9985	0.9650
	Mesh	0.3110	0.4125	0.4010	0.5140	0.2420	0.2930	0.3990	0.5350	0.6775	0.3650
26	Carbon	0.1167	0.1199	0.0994	0.1457	0.0875					
	CO ₃ -Carbon	1.1770		1.3380		0.7755					
	Hydrogen										
	Nitrogen										
	Oxygen	1.9464	1.5930	1.6340	1.4890	1.6190					
	Mesh	0.2236	0.2730	0.3950	0.4220	0.3950					
27	Carbon	0.2140	0.1058	0.1010	0.1228	0.1417					
	CO ₃ -Carbon	1.4700		0.7414		1.3615					
	Hydrogen										
	Nitrogen										
	Oxygen	1.3250	1.7468	1.3715	1.2980	1.4360					
	Mesh	0.7850	0.4042	0.5245	0.5037	0.5319					
28	Carbon	0.0946	0.1200	0.1066	0.1033	0.1284					
	CO ₃ -Carbon	1.2485		1.0965		1.4957					
	Hydrogen										
	Nitrogen										
	Oxygen	1.3775	0.9563	1.2116	1.6379	1.4371					
	Mesh	0.3055	0.2487	0.2234	0.5171	0.3059					
29	Carbon	0.1484	0.2011	0.1306	0.1830	0.1549					
	CO ₃ -Carbon	1.3750		1.8135		1.0510					
	Hydrogen										
	Nitrogen										
	Oxygen	1.9563	1.5465	2.0980	1.4780	1.2839					
	Mesh	0.4877	0.3095	0.4820	0.3860	0.2620					
30	Carbon	0.1293	0.1274	0.1321	0.1403	0.1311					
	CO ₃ -Carbon	1.1568		1.0362		0.7382					
	Hydrogen										
	Nitrogen										
	Oxygen	1.0550	1.7440	1.2350	1.3580	0.3041					
	Mesh	0.2280	0.4820	0.2590	0.2980	0.0781					

Table 9 (Cont.)

Weights of Analytical Samples

Hole No.	Sodium Wt.*(g)						<u>Sample Site Number</u>		7	8	9	10
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>					
31	Carbon	0.2175	0.1864	0.2416	0.1093	0.1286						
	CO ₃ -Carbon	2.6250		1.5860		1.4430						
	Hydrogen											
	Nitrogen											
	Oxygen	1.3650	1.5650	2.5790	1.7180	1.1510						
	Mesh	0	0	0	0	0						
32	Carbon											
	CO ₃ -Carbon	1.19	1.01	1.00	1.53	1.41						
	Hydrogen											
	Nitrogen	1.89	1.06	1.32	1.14	1.53	1.37	1.28				
	Oxygen	1.19	1.01	1.00	1.53	1.41						
	Mesh	0	0	0	0	0						
33	Carbon											
	CO ₃ -Carbon	2.43	2.09	1.02	1.98	1.11						
	Hydrogen											
	Nitrogen	1.29	1.01	1.27								
	Oxygen	2.43	2.09	1.02	1.98	1.11						
	Mesh	0	0	0.42	0	0						
34	Carbon											
	CO ₃ -Carbon	3.37	3.00	3.24		1.81						
	Hydrogen											
	Nitrogen											
	Oxygen	3.37	3.00	3.24	2.33	1.81						
	Mesh	0.63	0.60	0.58	0.67	0						
35	Carbon											
	CO ₃ -Carbon	3.82	1.36	1.46	1.39	1.76						
	Hydrogen											
	Nitrogen	2.44										
	Oxygen	3.82	1.36	1.46	1.39	1.76						
	Mesh	0.99	0.29	0.85	0.49	0.68						
36	Carbon											
	CO ₃ -Carbon	1.73	1.35	1.51	1.79	1.68						
	Hydrogen											
	Nitrogen											
	Oxygen	1.73	1.35	1.51	1.79	1.68						
	Mesh	0.68	0.71	0.80	0.47	0.53						
37	Carbon											
	CO ₃ -Carbon	1.42	1.91	2.34	2.09	1.72						
	Hydrogen											
	Nitrogen	1.15										
	Oxygen	1.42	1.91	2.34	2.09	1.72						
	Mesh	0.49	0.64	0.54	0.82	0.80						
39	Carbon											
	CO ₃ -Carbon	2.10	1.91	1.57	1.55	2.14						
	Hydrogen											
	Nitrogen	1.08										
	Oxygen	2.10	1.91	1.57	1.55	2.14						
	Mesh	0.54	0.52	0.28	0.48	0.58						
40	Carbon											
	CO ₃ -Carbon	1.45	1.21	1.58	1.42	1.49						
	Hydrogen											
	Nitrogen											
	Oxygen	1.45	1.21	1.58	1.42	1.49						
	Mesh	0.18	0.31	0.35	0.37	0.29						
41	Carbon											
	CO ₃ -Carbon											
	Hydrogen											
	Nitrogen											
	Oxygen	1.11	1.44	1.66	1.17	1.45						
	Mesh	0	0	0	0	0						

Table 9 (Cont.)

Hole No.	Sodium Wt.* (g)	Weights of Analytical Samples				
		1	2	Sample Site Number		5
				3	4	
42	Carbon	0.1204	0.1024	0.1160		
	CO ₃ -Carbon					
	Hydrogen		0.3016			
	Nitrogen	11.7720	12.9399			
	Oxygen	1.7810	1.8040	2.3950		
	Mesh	0	0	0		
43	Carbon	0.1787	0.1087	0.1481		
	CO ₃ -Carbon					
	Hydrogen	0.2148	0.2288	0.6327		
	Nitrogen	11.3007	11.0856			
	Oxygen	2.7630	3.2290	2.5460		
	Mesh	0	0	0		
44	Carbon	0.0942	0.1223	0.1015		
	CO ₃ -Carbon					
	Hydrogen	0.1503	0.1285	0.0909		
	Nitrogen	11.3539	11.5785			
	Oxygen	1.4520	1.2880	0.9107		
	Mesh	0.4460	0.5430	0.0346		
45	Carbon	0.1236		0.1481		0.1267
	CO ₃ -Carbon	1.3142		1.3456		1.1310
	Hydrogen			0.3565	(1.3629)	
	Nitrogen	12.0336		12.6927		
	Oxygen	1.2855		1.3155		1.3555
	Mesh	0.3385		0.2995		0.3465
46	Carbon	0.1305		0.1161		0.1190
	CO ₃ -Carbon	0.9140		0.8270		1.0210
	Hydrogen			0.4952		
	Nitrogen	15.4680	12.2050	12.8120	13.4097	14.4680
	Oxygen	1.2480		1.4810		1.5470
	Mesh	0		0		0
47	Carbon	0.1421	0.2607	0.1543		
	CO ₃ -Carbon	0.3050	0.3430	1.5460		
	Hydrogen	0.1850	0.2750	1.4500		
		(0.0420)**		(0.0411)**		
	Nitrogen	21.3540	29.9160	9.8193	24.0026	21.2230
	Oxygen	2.4420	1.9400	0.7640		
48	Carbon	0.1468	0.0916	0.1158		
	CO ₃ -Carbon	1.1000	0.8898	2.1815		
	Hydrogen	0.1122	0.1050	0.0980		
	Nitrogen	17.9675	20.8190	21.8700	18.5170	
	Oxygen	0.8510	1.0130	0.9590		
	Mesh	0.5178	0.2881	0.3272		
49	Carbon	0.1520		0.1022		0.1422
	CO ₃ -Carbon	1.4170		1.3150		0.9155
	Hydrogen			0.4029		
	Nitrogen	10.5282	12.2489	11.4923	12.9022	10.7138
	Oxygen	0.9490		1.1970		1.1220
	Mesh	0.2880		0.2820		0.2890
50	Carbon	0.1090	0.1292	0.1013		
	CO ₃ -Carbon					
	Hydrogen					
	Nitrogen	12.3169	12.6474			
	Oxygen	1.6600	2.0060	1.1300		
	Mesh	0	0	0		
51	Carbon	0.1444	0.2463	0.1116		
	CO ₃ -Carbon					
	Hydrogen					
	Nitrogen	12.2001	11.2562			
	Oxygen	0.7840	1.5970	1.4500		
	Mesh	0.0900	0	0		

Table 9 (Cont.)

Weights of Analytical Samples

<u>Hole No.</u>	<u>Sodium Wt.* (g)</u>	<u>Sample Site Number</u>				
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
52	Carbon	0.1206	0.1740	0.1454		
	CO ₃ -Carbon					
	Hydrogen					
	Nitrogen	13.0746	16.8603			
	Oxygen	1.1170	1.2250	1.9020		
	Mesh	0.4230		0.2950		
53	Carbon	0.1186	0.1938	0.1080		
	CO ₃ -Carbon					
	Hydrogen					
	Nitrogen	11.5037	11.3301			
	Oxygen	1.1900	1.9110	1.3870		
	Mesh	0.3170	0.4150	0.3510		
54	Carbon					
	CO ₃ -Carbon	1.40		1.54	1.55	1.14
	Hydrogen					
	Nitrogen					
	Oxygen	1.40	1.58	1.54	1.55	1.14
	Mesh	0.46	0.31	0.39	0.47	0.50
55	Carbon					
	CO ₃ -Carbon	1.32				
	Hydrogen					
	Nitrogen					
	Oxygen	1.32	1.00	1.09		
	Mesh	0				

*Sample weight in grams of mesh free sample used for each specific analysis.

**Values for hydrogen in parentheses were for catalytic oxidation procedure.

Note: For each hole the samples were approximately evenly spaced from the center to the outside of the trap for the number of sites listed.

Table 10

FERMI PRIMARY SODIUM ANALYSES

	<u>APDA¹</u> <u>Specifications</u>	<u>Tank Cars²</u>	<u>Cold Trapped³</u> <u>in Storage Tanks</u>	<u>After Six⁴</u> <u>Months in</u> <u>Reactor</u>
Alkalinity, %	99.97 $\pm$. 1	99.97 to 100.02	---	---
Chlorides, ppm	30 max	1 to 10	25	23
Sulfur, ppm	10 max	1 to 5	<10	<10
Carbon, ppm	50 max	11 to 37	<10	77
Calcium, ppm	10 max	<5	5	7
Boron, ppm	5 max	<1	< 2	<2
Lithium, ppm	20 max	<1	< 1	<1
Silver, ppm	20 max	<2	1	.5
Uranium, ppm		3.3 to 3.6	< 1	<1
Iron, ppm			5	8
Nickel, ppm			< 2	<2
Chromium, ppm			1	1
Silicon, ppm			3	10
Cobalt, ppm			< 2	<2
Zirconium, ppm			< 2	<2

¹ Atomic Power Development Associates

² Analyzed by DuPont in July, 1959, and December, 1960.

³ Analyzed by LeDoux in November, 1960, after cold trapping for 190 hours. The plugging temperature was less than 250°F.

⁴ Analyzed by LeDoux in May, 1961.

Table 11

Metallic Impurities in Sodium Samples from Fermi Cold Trap

Core Sample No.	4	4	3	6	6	7	7			
<u>Subsample No.</u>	2	3	1	2	10	1	4	Baker's No. 6266	Commer. Na. Avg.	U.S. Data Avg.
Al	< 3	3	7.6	7.6	3	7.6	10	3	10	10
Ca	< 80	< 80	< 80	< 80	< 80	< 80	< 80	100	120	190
Cr	< 50	< 50	50	64	50	50	64	50		5
Cu	< 3	3	20	430	280	300	480	3		2
Fe	30	150	180	200	180	200	300	25	20	2.5
K	230	230	150	130	180	150	100	13		100
Li	0.3	2.5	0.5	0.8	< 0.3	< 0.3	< 0.3	< .3		20
Mg	3.5	13	5.6	24	5.6	4.1	5.1	3		10
Mn	< 3	3	5	10	7.6	7.6	5.1	3		2
Ni	< 3	5.1	9.1	12	10	3.8	10	3	4	2
Si	15	48	46	71	15	10	43	7.6	70	5
Ti	< 7	< 7	8.9	7.6	10	< 7	7.6	< 7		
Sample Weight, g.	1.4	.8	2	5.8	6.8	10	9.8			

Notes: Results are in ppm [±] 30% relative on the basis of Na metal.
 The following elements were not detected in any of the samples.
 Numbers in parenthesis are detection limits in ppm.
 Ag (3), Au (800), B (25), Ba (7), Be (3), Bi (25), Cd (250), Co (26),
 Cs (80), In (25), Mo (7), Nb (800), P (800), Pb (25), Rb (25), Sb (80),
 Sn (80), Sr (25), Th (250), Ta (800), V (5), W (250), Zn (800), Zr (25).

September 1964

Table 11. (Cont.)

Metallic Impurities in Sodium Samples From Fermi Cold Trap

Core Sample No.	32			33			34			35			36		
Sub-Sample No. (3)	1	2	3	1	2	3	1	2	3	1	2	3 ⁽¹⁾	1	2	3
Al	< 8	< 8	< 8	< 8	< 8	< 8	8.5	4.0	4.3	15	5.0	110	8.8	< 8	< 8
Ca	< 3	< 3	< 3	< 3	< 3	< 3	< 8	< 8	< 8	< 3	< 3	< 3	< 3	< 3	< 3
Cr	< 3	< 3	< 3	3.8	< 3	< 3	22	< 3	< 3	28	30	20	25	15	16
Cu	5	3	4	230	4.5	30	340	< 3	260	360	360	320	320	260	240
Fe	7.5	< 3	< 3	40	3.8	5.0	110	< 3	35	165	100	120	160	78	90
K	150	180	180	240	150	180	300	125	250	230	170	170	200	220	180
Mg	< 3	< 3	< 3	2.5	0.5	< 0.5	2.8	< 0.5	< 0.5	3.0	2.0	5.0	2.5	1.5	3.2
Mn	< 2	< 2	< 2	< 2	< 2	< 2	11	0.9	7.0	11	8.5	8.5	12	8.0	< 2
Ni	< 5	< 5	< 5	< 5	< 5	< 5	4.3	< 3	< 3	5.3	5.8	4.5	6.3	3.3	7.5
Si	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15	< 15
Ti	4	< 3	< 3	< 3	< 3	< 3	8.5	3.3	4.8	7.8	5.0	5.3	6.0	< 3	3.3
Sample Wt., g.	6.58	9.11	11.86	8.76	5.66	14.70	11.78	9.21	12.06	15.71	12.59	11.76	13.39	13.24	7.65

Core Sample No.	37			54			39			41			20	55	Ethyl	(4) APDA #200	
Sub-Sample No. (3)	1	2	3	1	2	3	1	2	3	1	2	3					
Al	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	10	< 8	
Ca	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	250	< 3	
Cr	20	17	18	< 3	< 3	< 3	6.8	9.8	7.5	< 3	< 3	< 3	8.2	13	< 3	27	
Cu	250	340	230	53	30	80	63	38	140	< 3	< 3	< 3	5	3	3.5	< 3	
Fe	120	41	58	18	13	110	27	24	25	5	< 3	< 3	48	30	6.3	11	
K	210	130	240	150	130	200	140	100	75	250	75	140	140	75	250	75	
Mg	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	3	< 3	
Mn	2.7	2.2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	
Ni	7.5	7.5	8.8	< 3	< 3	9	< 3	3	3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	
Si	< 15	< 15	< 15	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	< 8	25	< 8	
Ti	6.3	< 3	< 3	< 3	< 3	< 3	< 25	< 25	< 25	< 25	< 25	< 25	< 3	< 3	< 3	< 3	
Sample Wt., g.	11.92	10.84	12.41	8.56	10.38	10.17	8.84	9.98	14.35	9.98	9.09	9.34	8.51	9.38	10.33	7.65	

NOTES:

- (1) Zn was detected in this sample at 380 ppm
- (2) Sr was detected in this sample at 21 ppm. Sample came from a pound can of sodium made by Ethyl Corp.
- (3) Actual location of sample along core can be determined by referring to copper and iron data points on Figure 1.
- (4) Sample came from the primary sodium system of the Fermi plant about October 1964. RESULTS IN FPM $\pm$ 30% RELATIVE ON THE BASIS OF Na METAL.

The following elements were not detected in any of the samples. Numbers in parentheses indicate detection limits in ppm.

Ag(2), B(25), Ba(3), Be(8), Bi(3), Cd(50), Co(3), Cs(15), In(50), Mo(8), Li(1), Nb(250), Pb(8), Rb(8), Sb(250), Sn(8), Sr(3), Th(250), V(8), W(250), Zn(80), Zr(15).

DESIGN DATA FROM FERMI COLD TRAP OPERATING MANUAL

Designer: MSA Research Corporation
 Manufacturer: Alloy Manufacturing Company
 Serial Nos.: AMC-8426-1; Natl Bd - 330
 Code Stamp: U - ASME Code for Unfired Pressure Vessels
 Type: Circulating Cold Trap with Cooling Jacket
 Heat Duty: 707,000 Btu/hr
 Heat Transfer Rate: 224 Btu/hr-sq. ft.- °F
 Heat Transfer Area: 43.8 sq. ft., minimum required;
 79 sq. ft., used
 LMTD; 72°F
 Fouling Resistance: None
 Allowable Leak Rate: None
 Materials: Process, Type 304 stainless steel
 Structural, carbon steel
 Size (approx.): 4 ft. diam x 7-1/2 ft. high, sodium
 vessel 4-1/2 ft. diam x 9 ft. high,
 OA 3500 lb empty; 8000 lb full.

<u>Item</u>	<u>Jacket</u>	<u>Vessel</u>
Fluid	NaK-78	Sodium
Flowrate, lb/hr	31,600	43,600
Velocity, ft/sec	0.194	0.291
Temperatures, °F		
Inlet	200	350
Outlet	300	300
Design, Maximum	1,000	1,000
Pressures, psig		
Operating	0-10	0-15
Pressure Drop	0.5	0.5
Design	15	60
Pneumatic Test	48	192

Table 12 (Cont.)

DESIGN DATA FROM FERMI COLD TRAP OPERATING MANUAL

Fluid Properties	Jacket	Vessel
Density, lb/cu ft	52.4	56.9
Viscosity, lb/ft-sec	2.92×10^{-4}	3.47×10^{-4}
Conductivity, Btu/hr-sq ft-°F/ft	13.2	47.9
Specific Heat, Btu/ lb-°F	0.224	0.323
Activity, curies/cc max	-	0.1
Construction Detail		
Size, nominal	50 in. ODx 3/16 in. th. x 6 ft. high	48 in. ODx 1/4 in. th. x 7-1/2 ft. high
Baffles	None	46 in. ODx 1/8 in. th. x 5 ft. high
Annular Width, in.	13/16	3/4
Packing	None	54-3/4 in. ODx 4 ft ¹ in. high at 12 ² lb/cu ft.
Nozzles		
Inlet	one-2 in.	one-3 in.
Outlet	three-2 in.	one-3 in.
Thermowell	None	one-1/4 in.
Rupture disc	None	one-3 in.
Supports	Attached to vessel only	four-4 in. I beams & 45-5/8 in. sq x 6 in. deep base and pan

¹MSAR engineering drawing shows 4'8".

²10 to 20 lb/ft³ given in Enrico Fermi Atomic Power Plant Hazards Report.