

SODIUM MASS TRANSFER: I  
TEST LOOP DESIGN

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June, 1962

Prepared for the  
SODIUM COMPONENTS DEVELOPMENT PROGRAM  
of the  
UNITED STATES ATOMIC ENERGY COMMISSION  
UNDER CONTRACT AT(04-3)-189  
PROJECT AGREEMENT #15

ATOMIC POWER EQUIPMENT DEPARTMENT

**GENERAL ELECTRIC**

SAN JOSE, CALIFORNIA

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TABLE OF CONTENTS

	<u>Page</u>
LIST OF FIGURES . . . . .	v
LIST OF TABLES . . . . .	vi
LIST OF FIGURES IN APPENDICES . . . . .	vii
LIST OF TABLES IN APPENDICES . . . . .	viii
ACKNOWLEDGMENT . . . . .	ix
I. ABSTRACT . . . . .	1
II. INTRODUCTION . . . . .	3
III. DESIGN, FABRICATION AND ASSEMBLY . . . . .	7
A. General . . . . .	7
B. Design of Test Loop Components . . . . .	14
1. Sample Holders . . . . .	14
2. Main Heaters . . . . .	22
3. Coolers . . . . .	27
4. Surge Tanks . . . . .	29
5. Electromagnetic Pumps . . . . .	30
6. Purification System . . . . .	30
7. Valves . . . . .	34
8. Dump Tanks . . . . .	34
9. Preheaters . . . . .	35
C. Assembly and Installation . . . . .	37
1. Weld Preparation and Procedure . . . . .	37
2. Stress Relief . . . . .	39
3. Quality Control . . . . .	40
4. Final Assembly and Installation . . . . .	43
D. Electrical Power Supply . . . . .	48
E. Control and Instrumentation . . . . .	52
1. General . . . . .	52
2. Alarm and Shutdown Systems . . . . .	55
3. Temperature Control . . . . .	55
4. Indicating Instruments . . . . .	59
5. Operating and Monitoring Panels . . . . .	64

	<u>Page</u>
IV. PROCEDURES FOR INITIAL OPERATION AND SAMPLE PREPARATION . . .	69
A. Sodium Charging and Filling . . . . .	69
B. Loop Start-Up . . . . .	69
C. Steel Sample Preparation . . . . .	70
D. Sample Loading and Removal . . . . .	72
E. Steel Sample Analysis . . . . .	75
F. Sodium Sampling and Analysis . . . . .	76
V. PRE-OPERATIONAL TESTS . . . . .	78
A. Level Gage Calibration . . . . .	78
B. Main Flowmeter Calibration . . . . .	79
C. Observed Pressure Drop Data . . . . .	85
VI. PROJECT STATUS (DECEMBER, 1960) . . . . .	88
APPENDICES:	
A. LITERATURE SURVEY ON MASS TRANSFER IN LIQUID METAL SYSTEMS . . . . .	90
B. ANALYTICAL PROCEDURES . . . . .	107
C. STEEL ACTIVATION . . . . .	132
D. STEEL SAMPLE REMOVAL UNDER OPERATING CONDITIONS . . . . .	145
E. PRESSURE DROP CALCULATIONS . . . . .	165
F. REFERENCE DRAWINGS . . . . .	187

LIST OF FIGURES

<u>Figure</u>	<u>Title</u>	<u>Page</u>
III-1	FLOW SHEET . . . . .	9
III-2	SAMPLE HOLDER . . . . .	18
III-3	TYPICAL HOT LEG SAMPLE HOLDER . . . . .	20
III-4	TYPICAL COLD LEG SAMPLE HOLDER . . . . .	21
III-5	SAMPLE LOADING PATTERN . . . . .	23
III-6	MAIN HEATER . . . . .	24
III-7	HEATER FITTING CURVES . . . . .	25
III-8	PUMP ASSEMBLY . . . . .	31
III-9	FOUR PASS PUMP CELL . . . . .	32
III-10	WELD PREPARATION & FIT UP FOR MASS TRANSFER PIPING . . . . .	38
III-11	LOOP ARRANGEMENT . . . . .	44
III-12	PARTIALLY INSULATED LOOP . . . . .	45
III-13	COVER GAS FLOW SHEET . . . . .	47
III-14	MASS TRANSFER LOOP ARRANGEMENT . . . . .	49
III-15	POWER SUPPLY SYSTEM . . . . .	50
III-16	LOOP POWER DISTRIBUTION . . . . .	53
III-17	INSTRUMENTATION DIAGRAM . . . . .	56
III-18	TEMPERATURE CONTROL . . . . .	58
III-19	LEVEL INDICATOR . . . . .	63
III-20	INSTRUMENT PANEL INSTALLATION . . . . .	65
III-21	OPERATING PANEL ARRANGEMENT . . . . .	66
III-22	MONITORING PANEL ARRANGEMENT . . . . .	68
IV-1	SAMPLE FLOW SHEET . . . . .	73
V-1	SURGE TANK LEVEL INDICATOR CALIBRATION . . . . .	79
V-2	FLOW- $\Delta$ P TEST EQUIPMENT . . . . .	81
V-3	LOOP #3 FLOW CALIBRATION CURVE . . . . .	83
V-4	LOOP PRESSURE DROP . . . . .	87

LIST OF TABLES

<u>Table</u>	<u>Title</u>	<u>Page</u>
III-1	MATERIAL SPECIFICATIONS . . . . .	41
III-2	ALARMS AND SAFETIES FOR THE SODIUM MASS TRANSFER LOOPS . . . . .	57
III-3	SELECTOR SWITCH NUMBER IDENTIFICATION . . . . .	61
V-1	LOOP #1 FLOW DATA . . . . .	78
V-2	LOOP #3 FLOW DATA . . . . .	82
V-3	FLOW METER CALIBRATION DATA FOR MASS TRANSFER LOOPS . . . . .	84
V-4	LOOP #3 PRESSURE DROP DATA . . . . .	86



LIST OF FIGURES IN APPENDICES

<u>Figure</u>	<u>Title</u>	<u>Page</u>
B-1	SODIUM CORROSION DATA - HOT ZONE SPECIMENS . . . . .	112
B-2	POSSIBLE FORM OF MASS TRANSFER DATA WEIGHT . . . . . CHANGE VS TIME	113
C-1	ISOTOPIC ACTIVATION IN 316 STAINLESS STEEL . . . . .	138
C-2	ISOTOPIC ACTIVATION IN $2\frac{1}{4}$ Cr-1Mo ALLOY . . . . .	139
C-3	ISOTOPIC ACTIVATION IN $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$ ALLOY . . . . .	144
D-1	FREEZE VALVE ARRANGEMENT . . . . .	151
D-2	VALVE - BELLOWS ARRANGEMENT . . . . .	152
D-3	PISTON ASSEMBLY - DRYBOX ARRANGEMENT . . . . .	154
D-4	EM PUMP - DRYBOX ARRANGEMENT . . . . .	155
E-1	SAMPLE LOADING PATTERN . . . . .	167
E-2	HOT LEG SAMPLE HOLDER - PRESSURE DROP LOCATIONS . .	173
E-3	COLD LEG SAMPLE HOLDER - PRESSURE DROP LOCATIONS . .	174
E-4	CALCULATED LOOP PRESSURE DROP . . . . .	178
E-5	CALCULATED PRESSURE DROP WITHOUT SAMPLES . . . . .	180
E-6	CALCULATED ISOTHERMAL LOOP PRESSURE DROP . . . . .	182
E-7	CALCULATED ISOTHERMAL PRESSURE DROP WITHOUT . . . . . SAMPLES	183
E-8	PLUGGING OF A COOLANT PASSAGE . . . . .	184
E-9	CHANGE IN SAMPLE PRESSURE DROP VS SAMPLE LOSS . . . . . FROM HOT LEG AND DEPOSIT ON COLD LEG	186

LIST OF TABLES IN APPENDICES

<u>Table</u>	<u>Title</u>	<u>Page</u>
C-1	EMISSION SPECTROMETER ANALYSIS RESULTS . . . . .	134
C-2	NUCLIDE PROPERTIES . . . . .	135
C-3	RADIOCHEMICAL ANALYSIS RESULTS . . . . .	136
C-4	RADIOACTIVE ISOTOPE ACCUMULATION REQUIREMENTS . . .	140
C-5	COMPARISON OF ACTIVATION BETWEEN THE TWO METALS . .	140
C-6	RADIOCHEMICAL ANALYSIS RESULTS . . . . .	143
E1	SAMPLE HOLDER PRESSURE DROP - SAMPLES . . . . . INSTALLED - 0.5 gpm FLOW	177
E2	PRESSURE DROP WHEN FLOW IS MEASURED AT 700 F . . . . . AND SAMPLE HOLDERS ARE AT OPERATING TEMPERATURES	179
E3	SAME AS TABLE E2 EXCEPT SAMPLES ARE REMOVED . . . .	179
E4	LOOP ISOTHERMAL AT 700 F -- SAMPLES INSTALLED . . .	181
E5	LOOP ISOTHERMAL AT 700 F -- SAMPLES REMOVED . . . .	181
E6	PRESSURE DROP (psi) @ 0.5 gpm AS A FUNCTION . . . . . OF MILS DEPLETION OF HOT LEG AND DEPOSITION ON COLD LEG SURFACES	185

ACKNOWLEDGMENT

The authors gratefully thank the many people at the Atomic Power Equipment Department of the General Electric Company that applied special skill and effort to design, procure, fabricate, install and start up the test loops; in particular to the authors of the various sections of the appendix that made this report more complete and to Dr. L. F. Epstein, GE-VAL, H. W. Savage and J. H. DeVan, ORNL, for the encouragement, consultation and advice that they provided during the development of the test equipment.

## SODIUM MASS TRANSFER: I TEST LOOP DESIGN.

### I. ABSTRACT

Higher system temperatures are required to reduce the operating cost of nuclear power plants cooled with sodium. However, to reliably design and operate such a plant, experimental determination of the mass transfer and metallurgical effects under representative conditions are necessary. This report presents the design, fabrication, assembly, operating procedures, and start-up data for six experimental test loops to examine the effect of steel exposed to sodium at temperatures as high as 1300 F. Until the loops are sectioned at the end of the program, corrosion data will be obtained from metallic tabs exposed to sodium at various locations in the loops under prescribed test conditions. The loops are identical in design and differ only in the material used in fabrication.

One complete loop is fabricated from Type 316 Stainless Steel, one from  $2\frac{1}{4}\text{Cr}-1\text{Mo}$  alloy steel and one from  $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$  alloy steel; two loops are constructed of Type 316SS in the heated or hot leg portion, and  $2\frac{1}{4}\text{Cr}-1\text{Mo}$  alloy steel in the cooled or cold leg portion. The purification side stream on these two loops is also fabricated from  $2\frac{1}{4}\text{Cr}-1\text{Mo}$  steel. The sixth loop is constructed of type 316SS in the hot leg and  $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$  in the cold leg and purification leg. In each loop the sodium coolant is purified in a side stream by means of a forced convection cold trap. In parallel with the cold trap is a plugging indicator for determining the oxygen impurity in the sodium. The test loops that contain low alloy steel are designed to operate at a maximum temperature of 1200 F, and the type 316SS loop is designed for operation at 1300 F.

All loops have sample holders located in the maximum and minimum temperature regions, as well as in two intermediate regions in the hot leg and also in the cold leg. In addition, there is a special sample holder in the maximum temperature zone that can be biologically shielded. An activated steel specimen will be located in this sample holder when the radioactive tracer technique is being employed. The corrosion samples are fabricated from the same material as the sample holder, and are  $1/16$  of an inch thick by  $3/16$  of an inch wide by 3 inches long. After cleaning they

will be weighed before and after test to  $\pm 0.1$  mg in order to determine their change in weight. This weighing accuracy will provide a sensitivity of less than 0.06 mils per year of uniform loss of steel sample material.

In order to obtain prescribed test temperatures in the sample holders, there are electric heaters preceding the first three sample holders in the hot leg and extended surface air coolers preceding the three cold leg sample holders. Manual adjustments are made to establish test conditions. Automatic control of some of the electric power to the heaters and of some of the air to the coolers provides a trim control to maintain prescribed temperature conditions during daily changes in line voltage and ambient conditions. This control permits unattended loop operation for a period of several work shifts.

To provide independent operation, each loop has an individual power transformer and electrical distribution system. A contactor interrupts all power to the loop if the stop button or any one of three over temperature trips are activated. Electrical power distribution, circuit breakers, automatic temperature controllers, inert gas control system and temperature indication for about 30 locations on each loop are provided on the individual operating panels. The seventh instrument panel contains a level, temperature and flow recorder for all loops and a two pen recorder for cold trap or plugging indicator data.

This is the first of a series of reports on this project. This report contains detailed information from the conceptual design stage, through fabrication and installation of the test equipment, and includes pre-operational test data. The test loops are ready for the first corrosion test run. Results from operating the test loops will be contained in future reports.

## II. INTRODUCTION

Ever since the U. S. Atomic Energy Commission was established, the Commission has supported the development of sodium cooled reactor systems with the promise of economic electric power generation or nuclear ship propulsion. By the time this project was proposed, some twelve years later, a considerable amount of coolant technology and corrosion data was available for temperatures up to about 1000 F. In addition, certain classified military projects afforded a small amount of short term data at temperatures up to 1600 F.

Based on the detailed design and construction cost estimates of several prototype sodium cooled central station type plants, it became apparent that to be competitive, sodium cooled power plants would have to develop improved plant efficiency (operation at higher temperatures) to overcome coolant system equipment costs.

Thus, in early 1958, the Atomic Energy Commission established the Sodium Component Development Program. The objectives of this program are to develop less expensive and more reliable components available to transfer heat from a reactor to a working fluid system. As with any power conversion system, the higher the temperature, the more efficient the thermal cycle. A detailed program was established to design and operate equipment with maximum temperatures from 1000 to 1300 F. AEC personnel responsible for the Sodium Component Development Program established a Working Group advisory committee composed of representatives from industry, national laboratories, and the AEC to recommend experimental and developmental programs to produce the desired coolant technology and equipment design criteria. The sodium Mass Transfer Project is one of the projects recommended by the Working Group. The test conditions and materials established in this contract are complementary to other projects sponsored by the Sodium Component Development Program.

The work reported herein was performed under USAEC contract AT(04-3)-189-Project Agreement No. 15 - Mass Transfer Investigations in Liquid Metal Systems at Moderately High Temperatures. This project consists of designing, fabricating, operating and evaluating data from six experimental sodium test loops designed to determine the rate of metallic mass transfer for a group of steels.

The material composition of the loops are as follows:

- a. One loop of type 316 stainless steel.
- b. One loop of  $2\frac{1}{4}\text{Cr}-1\text{Mo}$ .
- c. One loop of  $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$ .
- d. One bimetallic loop of 316 stainless steel and  $2\frac{1}{4}\text{Cr}-1\text{Mo}$ .
- e. One bimetallic loop of 316 stainless steel and  $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$ .
- f. One loop will duplicate item d above but will vary either in velocity or operating philosophy.

Included in this report is a description of the design, fabrication, installation and check-out of these test loops.

As stated in the contract, the first five loops are to run initially as a material screening test to determine mass transfer and/or corrosion of materials under the following conditions:

- a. Sodium velocity up to 30 ft/sec.
- b. Maximum temperatures 1200 F for all loops, except the 316SS loop which will be initially run at 1300 F.
- c. Temperature differential from hot to cold leg: 500 F.
- d. Sodium purity: 50 ppm oxygen. Other contaminants shall be measured and held constant as far as possible.

The second bimetallic  $316\text{SS}-2\frac{1}{4}\text{Cr}$  loop (which will be called #2) will be operated at the above conditions except the sodium purity will be reduced to about 10 ppm oxygen.

The steels to be tested are the same as were used to fabricate the loops. These include 316SS,  $2\frac{1}{4}-1\text{Mo}$  and  $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$  alloy steel. Corrosion and metallurgical data is to be obtained from removable corrosion samples located in various temperature regions around the loop, and from various segments of the loop itself. A radioactive tracer method is to be used to obtain additional data by placing an activated foil sample in the maximum temperature region. Analysis of sodium and metallic samples are described in the text and appendices.

The test variables include material, maximum temperature, differential temperature and oxygen impurity of the sodium passing over the samples. The test variables described above, are consistent with the primary variables that have been determined in previous projects and are anticipated to have the most significant effect on the corrosion rates determined by this project. Other test variables include impurities in the sodium other than oxygen, velocity of the coolant past the sample and the length of time that the steel samples are subjected to the sodium.

The construction (or test) materials and sodium were purchased in accordance with commercial specifications and will be used in the initial screening tests. The effect of shutting down and starting up the loop for removing samples or inadvertent stoppage of loop operation, may be significant. At this time, however, there appears to be no evidence in available literature that interrupted operation will affect the corrosion rate or metallurgical results; so it is planned to drain the loops and cool them to about 300 F prior to removing samples at scheduled periods. The loops are to be operated as continuously as possible within the test conditions and scheduled shutdowns. Throughout this report there are frequent references to the "Hot Leg", "Cold Leg" and "Purification Leg" regions of the test loops, they are defined as follows:

- a. The hot leg is that portion of the loop where the temperature is increasing. It contains the three main heaters and sample holders H1, H2, H3 and H3R.
- b. The cold leg is that portion of the loop where the temperature is decreasing. It contains the three main coolers and sample holders C1, C2 and C3.
- c. The purification leg consists of the bypass inlet valve, bypass flow meter, auxiliary cooler, plugging indicator, cold trap, and the outlet valves for the indicator and trap.

Detailed descriptions of these components are contained in later sections of this report. These definitions of the three major loop subassemblies, are presented to clarify such references.



One of the criteria for designing the test loops was to simulate, or prototype on a miniature scale, conditions that might be obtained during the operation of a liquid metal cooled reactor system. These loops could be representative of either the primary or the secondary circuit in a reactor system. For example, the hot leg would represent the reactor (with no nuclear effects on the steel) or the secondary side of an intermediate heat exchanger. The cold leg, correspondingly, would represent either the primary side of the intermediate heat exchanger or the steam generator. The emphasis on the low alloy steels is deemed necessary due to the fact that most of the presently installed steam generators are fabricated from this type of water side corrosion resistant and less expensive material, and that future steam generators probably also will be fabricated from this type of steel.

It was recognized from the beginning that these test loops would have to include a pump for forcing circulation of the sodium in order to obtain the velocities required by the contract. In fact, the pressure drop is in the order of 100 psi which is comparable with that of a reactor cooling system. Purification of the sodium with a forced convection cold trap and the ability to monitor the plugging temperature (oxide content) with a plugging meter will provide good control of the sodium purity. Much of the existing corrosion data is questionable with regard to control and monitoring of the sodium impurities.

Appendix A contains data from reports which were reviewed during a short term literature survey to obtain pertinent information for the design of this type of equipment. Four additional letter reports and a set of component fabrication drawings are also contained in the Appendices. Reference to appropriate appendices appear in the text of this report.

This report provides information on the design, fabrication and pre-operational tests of the six test loops. The effective cut off date for the information in this report was December 1960. Subsequent reports will provide the results obtained from operating the test loops and a descrip-

tion of specialized techniques used to analyze the sodium and metallic samples. After initial data becomes available the continuing test program will be developed.

The following section in this report described the design philosophy, detailed design and assembly of the test loops and control panels; installation in the test building; pre-operation tests and procedures for initial operation.

### III. DESIGN, FABRICATION AND ASSEMBLY

- III A General
- III B Design of Test Loop Components
- III C Assembly and Installation
- III D Electrical Power Supply
- III E Control and Instrumentation

#### III A General

Supplemental test loop specifications and operating conditions were established during design review meetings with the AEC. These included the following:

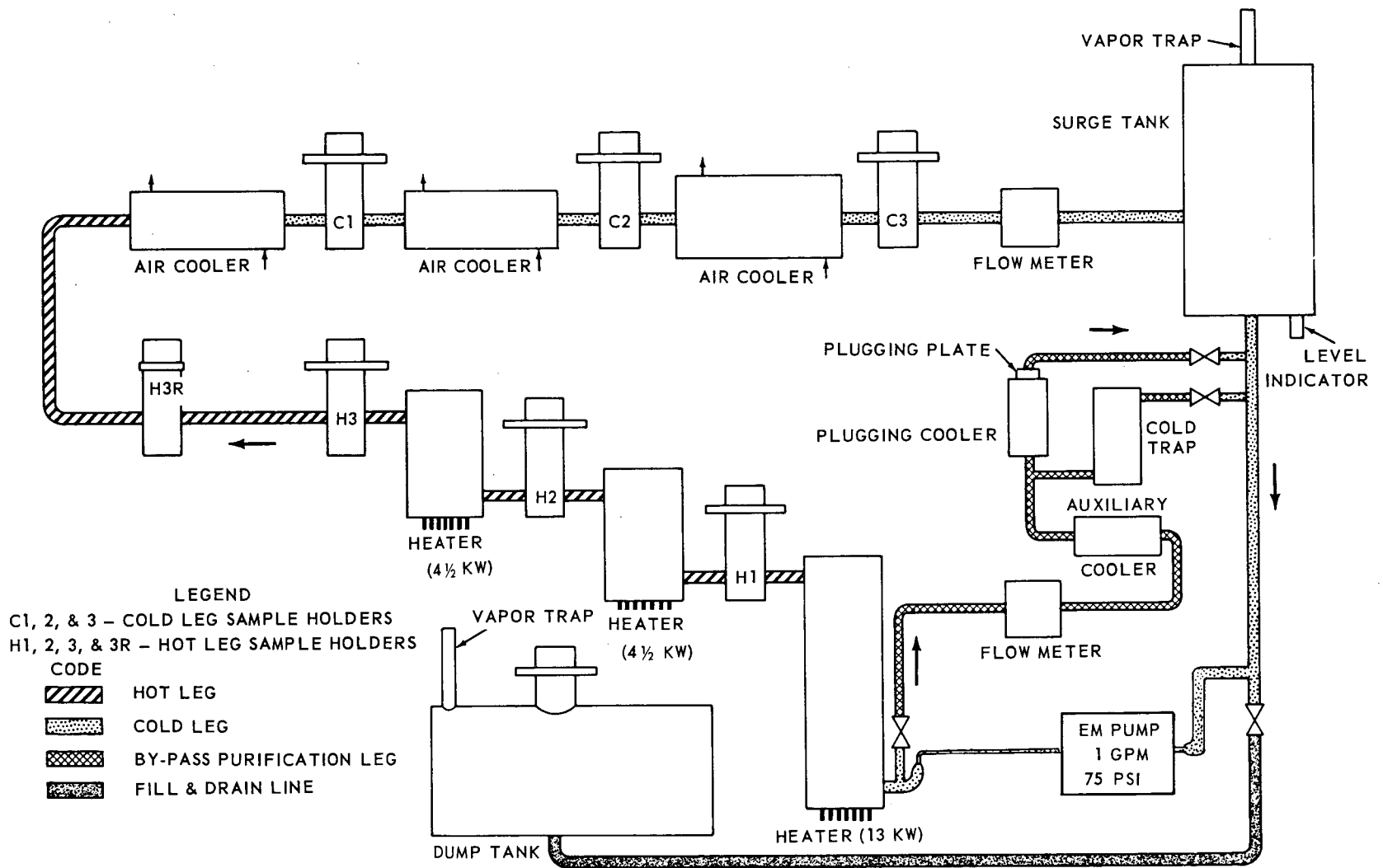
1. Figure 8 type loops should not be considered.
2. Bimetallic loops should have the ferritic material in the cold leg and the austenitic material in the hot leg.
3. The loops should have one-half of the wetted area in the increasing temperature region (hot leg) and the other half in the decreasing temperature zone (cold leg).
4. Heat flux in the heaters should be nominal; preferably low.
5. Corrosion rates should be determined by tabs (or samples) located at various temperature regions in both the hot leg and in the cold leg regions.
6. Weight change of the samples should be obtained as a function of operating time. (Remove samples during the test run.)
7. Samples should be changed during the run under operating conditions.

8. Radioactive tabs (foil samples) to be inserted in the maximum temperature region of the 316SS loop initially.
9. All stainless loops to be designed for 1300 F operation while other loops containing ferritic alloy steels to be designed for 1200 F maximum temperature.
10. Initial test operation to have intermittent cold trap operation to maintain prescribed oxygen levels. Later operation may include continuous cold trap operation.

During the conceptual design stage of the test loops, a flow sheet and various design philosophies were developed to meet these specifications. The final flow sheet with superimposed, idealized component outlines and relative elevations of the parts could, in general, be substituted for an elevation view of the test loop. This flow sheet is referenced frequently in this design report and is presented as Figure III-1.

The six forced circulation test loops are geometrically identical except for the material of fabrication. This concept provides direct comparison of test results between the loops. Primary components of each loop include seven flanged sample holders interspersed with three heaters and three air coolers. Service functions are provided by an electromagnetic pump, surge tank, dump tank, cold trap, and plugging indicator. Interconnecting lines are principally  $\frac{1}{2}$ -inch piping with 1-inch piping used in the cold leg to facilitate finned cooler connections.  $\frac{1}{4}$ -inch piping was used in the flow meters to permit more accurate flow measurements in the low flow (0.2 to 1.0 gpm) range planned for the experiments. There are no valves located in the main circulating stream, but four valves are used to isolate the drain and purification systems. They are oriented so that the bellows side of the valve is away from the main flow stream. During operation these four valves are normally closed.

As described in the sample holder design section, the design flow rate was selected to be one gpm. This was a compromise between power and pump requirements and practical sample and flow passage dimensions. The heaters



276-41

FIGURE III-1 FLOW SHEET

and coolers were designed to provide specified temperature at the sample holders with the design flow rate. Flexibility in applied heating and cooling rates (sample holder temperatures) is accomplished by manually adjusting the electrical power input to the heaters and the cooling air flow to the extended surface coolers.

The basic loop design criteria was operating reliability. In order to minimize interference from neighboring test loops each loop was provided with individual power distribution and operating panels. Common utilities provided for each loop included electrical power from the test building 480 volt distribution panel, instrument power from the 240/120 volt building panel and a common low pressure (15 psig) argon gas supply. Each loop has its individual over temperature power cut off (safety) system and is provided with limited range (less than 10%) automatic control of the maximum temperature (heater controls) and minimum temperature (air damper controls). This feature provides more nearly constant test conditions during unattended loop operation. Manual data logging is supplemented by two multi-point recorders that monitor the maximum and minimum temperature, level, and flow of each loop.

In addition to normal functions, the surge tank was designed to disengage any gas in the sodium. This was done by providing a flow through type design with a very low flow velocity in the liquid phase and a liquid level indicator so that the operators can maintain sufficient liquid submergence.

The purification leg consists of a flow meter, auxiliary air cooler, cold trap, plugging indicator and necessary electrical heating and air cooling facilities. An economizer heat exchanger was not installed because of the small amount of heat to be dissipated; the anticipated infrequent use of the equipment; and to minimize the amount of surface area in the portion of the loop.

Electrical resistance heat was chosen for the main heaters to provide the maximum flexibility of heat input to each of the three units. Immersion type heaters were chosen for high reliability. However, to maintain material continuity the inconel clad units were fitted into thimbles fabricated

from the same material as that of the hot leg. The units are similar to a shell and tube exchanger with the heaters inserted in the tubes. The coolant flow was purposely designed to be low in order to provide natural convection assistance in equalizing the sodium temperature in the heaters. It is anticipated that some of the seven heating units will not be energized or will be operated at very low power during some test operations.

Considerable effort was devoted to the design of the samples and sample holders. Design details are presented in the next subsection of this report. Presented below, is a list of the functions that the sample holder is required to provide:

1. access for inserting and removing corrosion samples
2. support and positioning of the samples
3. flow passages for the sodium around the samples
4. probe for measuring temperature
5. pre-heating facility

Once the approximate size of the samples and flow passages were established accessibility for changing the samples under three conditions were studied. These conditions were as follows:

1. Drain loop, open holders to room air at about 300 F. Manually remove and replace samples.
2. Same as above but exclude air by means of a protective (inert gas) atmosphere.
3. Change samples during operation at test conditions.

Provisions for the first two conditions were achieved by a double (concentric) flange design. The smaller flange provided access to the sodium system and samples. For condition 1 this is the only flange used. For the second condition a portable dry box that could be evacuated and purged with argon prior to opening the inner flange was attached to the sample holder with the outer flange.

Preliminary consideration of the problems associated with the third condition (opening the system with 1300 F sodium at about 75 psi pump discharge) encouraged a review of the necessity of this requirement. See the feasibility report on this subject in Appendix D for some concept of the equipment required. As a result of this report and the lack of evidence in available literature that draining and cooling down a test loop is detrimental to the corrosion data, it was recommended that no further work should be devoted to developing such a mechanism. If at a later time it becomes desirable to have such a device it is possible that the double flange sample holder could be modified to form a component of a future sample removal device.

The loops and components were designed for operation under the following conditions:

Temperature

Main Loop -- Maximum Temperature

316 Stainless steel (monometallic)	1300 F
All other loops	1200 F
Surge Tank	1000 F
Dump Tank	1000 F
Bypass purification leg	
inlet	800 F
outlet	300 F

Pressure

Surge tank	50 psi
Sodium valves	40 psi
Remainder of system	100 psi

System  $\Delta T$

500 F

Velocity

Hot leg samples	
high velocity	30 fps
low velocity	10 fps
Cold leg samples	10 fps

Flow

Main loop	1.0 gpm @ 1000 F
Cold trap (with auxiliary cooler)	0.2 gpm @ 800 F

<u>System Pressure Drop</u>	70 psi @ 1 gpm
-----------------------------	----------------

Total Heat Input

Preheat	4 KW
Test conditions	25 KW

Allowable Stress

The loops were designed in accordance with the code for pressure piping, ASA B31.1-1955 and with Section VIII of the ASME boiler code. (Stress values for the ferritic materials were extrapolated.)

<u>Material</u>	<u>Temperature</u>	<u>Allowable Stress</u>
TP 316SS	1300 F	4000 psi
2 $\frac{1}{4}$ Cr-1Mo	1200 F	2000 psi
5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti	1200 F	1200 psi

In subsequent parts of this section the design of each of the loop components will be considered individually. Shop drawings of the major components and loop assembly are found in Appendix F.



### III B Design of Test Loop Components

The following text and associated illustrations present a description and discussion of the design of the various test loop components.

B1 Sample Holders	B6 Purification System
B2 Main Heaters	B7 Valves
B3 Coolers	B8 Dump Tanks
B4 Surge Tanks	B9 Preheaters
B5 Pumps	

#### B1 Sample Holders

As the name implies the sample holders were designed to hold the corrosion samples. The design of the holders was governed largely by the following conditions which were established for this test program.

- a. Minimum number of sample holders - six (actually seven installed\*).
  - 1) Hot leg holders located at  $T_{max}$ ,  $T_{max}-100$  F,  $T_{max}-200$  F regions.
  - 2) Cold leg holders located at  $T_{max}-100$  F,  $T_{max}-200$  F, and  $T_{min}$  ( $T_{max}-500$  F) regions.
- b. Samples located at  $T_{max}$  and  $T_{min}$  are to be removed initially at the end of one, two, and four months. Other samples to be removed at end of the four month period only.
- c. Samples to be loaded and discharged with negligible contamination.
- d.  $T_{max}$  holder location to be suitable for containing an irradiated specimen ( $\sim 0.1$  curie).
- e. Velocity past hot leg samples: 30 ft/sec. maximum.
- f. System flow: 1 gpm.
- g. Temperature of sample holder to be measured.

---

\* A total of seven sample holders were developed by the design conditions. Items d and e indicate that three 30 ft/sec samples should be provided at  $T_{max}$ . In order to satisfy item d, one of the  $T_{max}$  holders must be capable of storing an activated specimen and hence should be biologically shielded. A practical way of meeting these conditions was to provide two separate sample holders at the  $T_{max}$  location.

The corrosion sample size was selected to provide sufficient area for weight change measurements. Thickness requirements for all but the irradiated samples was established at 1/16 inches in order to facilitate subsequent metallographic examinations. A rectangular flow passage with the sample oriented on the diagonal was selected as the most convenient method of achieving maximum sample area without encountering excessively small flow passages. In order to minimize the cost of the power supply and loop, and yet provide adequate corrosion data, the system was designed for a nominal flow of 1 gpm. Hence, to provide 30 fps velocity the cross-sectional flow area surrounding the samples was fixed at 0.0107 in square inches.

Numerous configurations were considered in establishing a reference sample and flow passage design. A cylindrical flow passage was originally proposed due to the simplicity of fabrication. However, maximum sample area, compatible with the system design limitations, was considered highly desirable in an effort to minimize weight measurement errors. Corrosion specimens of various cross sections such as square, triangular, circular and annular were evaluated. All of these configurations provided less exposed surface area and/or smaller clearances between the sample and flow passage wall than the rectangular cross-section selected. The other configurations considered were undesirable because:

- a. Extremely small clearance between the tube and flow passage surfaces.
- b. Difficulty in mechanically supporting and/or positioning the sample while retaining convenient removability and,
- c. Sample alignment and location with uniform clearances.

One of the advantages of the rectangular sample in a rectangular coolant passage is the extreme variation of flow velocity across each face of the sample. If velocity is a significant factor associated with metallurgical effects or the corrosion rate, the appearance of the sample after exposure should indicate such an effect.

A design corrosion rate of  $10 \text{ mg/dm}^2\text{-mo}$  (equivalent to  $0.06 \text{ mils/yr}$  uniform metal loss) was estimated from extrapolation and interpolation of available mass transfer data. See Appendix B for a discussion of estimated corrosion rates. This value with consideration of the minimum test period (1 month) was used in establishing a sample area which corresponded to a minimum weight change value which was greater than the combined weighing errors. Utilizing a balance capable of measurements to  $0.05 \text{ mg}$  it was estimated that the gross error, including variations due to cleaning and handling samples was less than  $0.25 \text{ mg}$ .

Since a weight change measurement is based upon a two weight determination, a maximum weighing error of  $0.5 \text{ mg}$  was used to design the corrosion samples. On this basis the minimum exposure surface area for each corrosion sample should be  $5 \text{ cm}^2$ . To meet this requirement with a small safety factor, and to provide a sample thick enough for metallurgical examination the corrosion samples are designed to be  $1/16$ -inch thick by  $0.165$  wide by  $2-15/16$ -inches long with a top extension  $5/16$ -inch wide by  $7/64$ -inch long. The total exposed area of each sample is  $9.3 \text{ cm}^2$ . The top extension serves to position the sample in the flow passages by:

- a. Preventing the sample from slipping downward due to the overhang beyond the edge of the insert slot.
- b. Limiting upward movement as a result of the small clearance between this sample extension and the bottom of the blank flange sample holder cover.
- c. Limiting movement in the diagonal position as a result of being restricted by a  $1/16$ -inch diameter pin attached to the center of the insert and the corners of the slot.

The activated steel sample is designed to have the same exposed surface area with a minimum mass (thickness) to reduce biological handling and shielding problems. Foil samples  $1/10$  the thickness of the corrosion samples ( $1/16$ -inch) seemed to be a reasonable compromise between ruggedness

and radioactivity. The resulting foil design is  $0.005 \pm 0.002$ -inch thick and 3 inches long. The foil is crimped along its center line to form a "V" to make it more rigid and less susceptible to vibration during exposure conditions. The width of this sample is sized to fit within a special rectangular passage  $1/16 \times 3/16$ -inch in cross-section. A rectangular passage was utilized for the same reasons described above. The foil is restricted from vertical motion by the close fitting sample holder cover and by a  $1/16$ -inch diameter wire located across the bottom of the coolant passage. The wire is welded to the insert prior to assembly into the holder body.

Based upon the foil dimensions, an assumed transport rate of  $10 \text{ mg/dm}^2\text{-mo}$  and irradiation data given in Appendix C, it was concluded that an activation level of 0.10 curies/foil was sufficient to provide measurable deposition at the standard corrosion samples. For personnel protection, approximately 4" of lead shielding around the radioactive foil sample holder is calculated to be adequate for reducing the dose rate to less than 2 mr/hr in the operating region around the loops. For tests not utilizing the radiotracer technique non-activated samples will be located in the H3R sample holder. Foils, activated or not, will be fabricated of the same material as the sample holder (hot leg of the loop).

A partially exploded view of a typical sample holder is illustrated in Figure III-2. All components of the sample holder exposed to sodium are machined from bar stock of the material as that selected for that particular section of the loop. The upper part of the body is attached to a 2-inch Marman Conoseal flange assembly to facilitate insertion or removal of corrosion samples. The female flange of the conoseal is welded to the holder while the male flange is part of the removal cap. Each sample holder is designed to contain two steel inserts 0.625 inch diameter x 3 inches long which are pressed into reamed holes in the body.

Three different slot (coolant passage) configurations were utilized for the sample holder insert design. The dimensions are based on 1 gpm loop

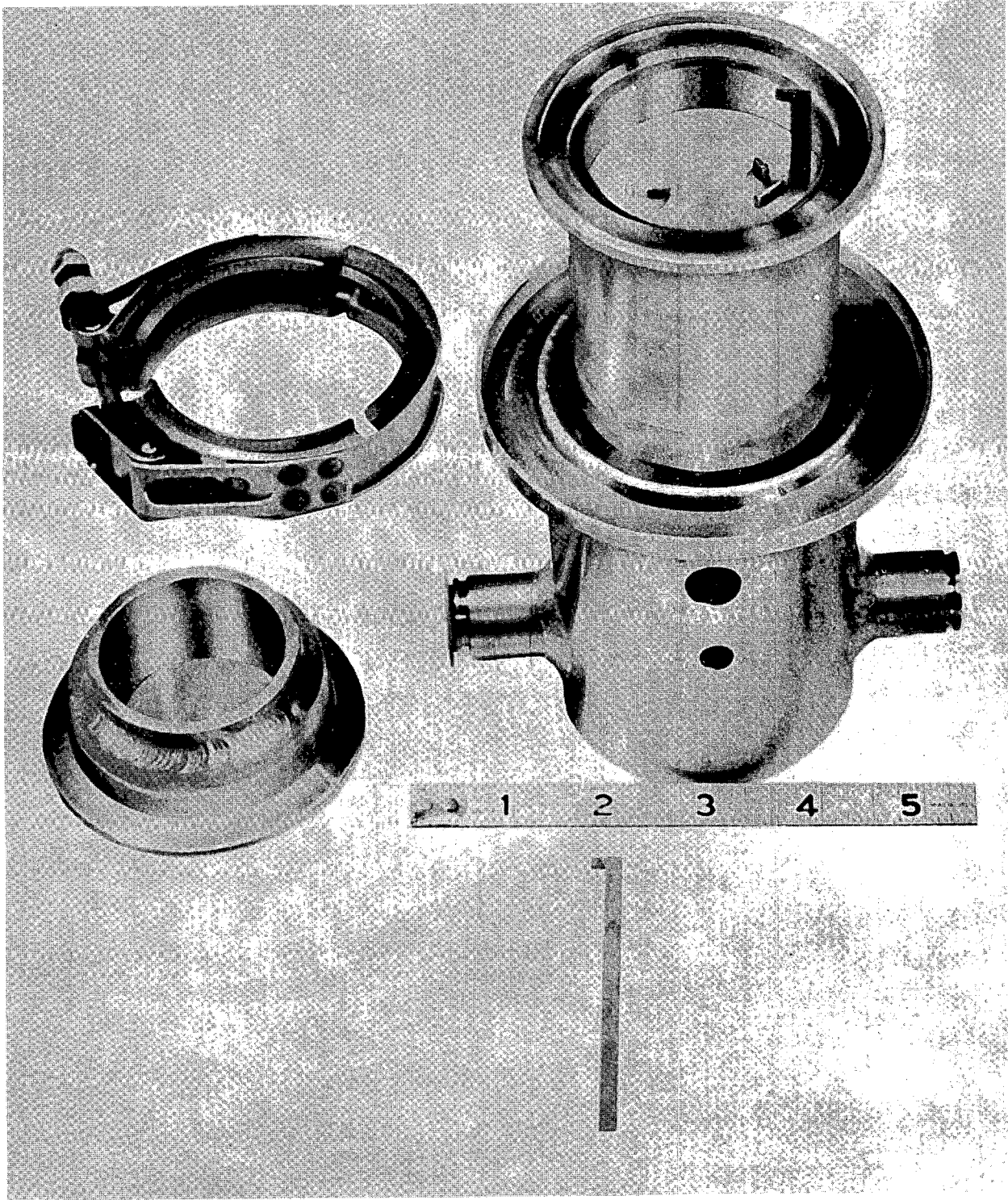


FIGURE III-2 SAMPLE HOLDER

276-74

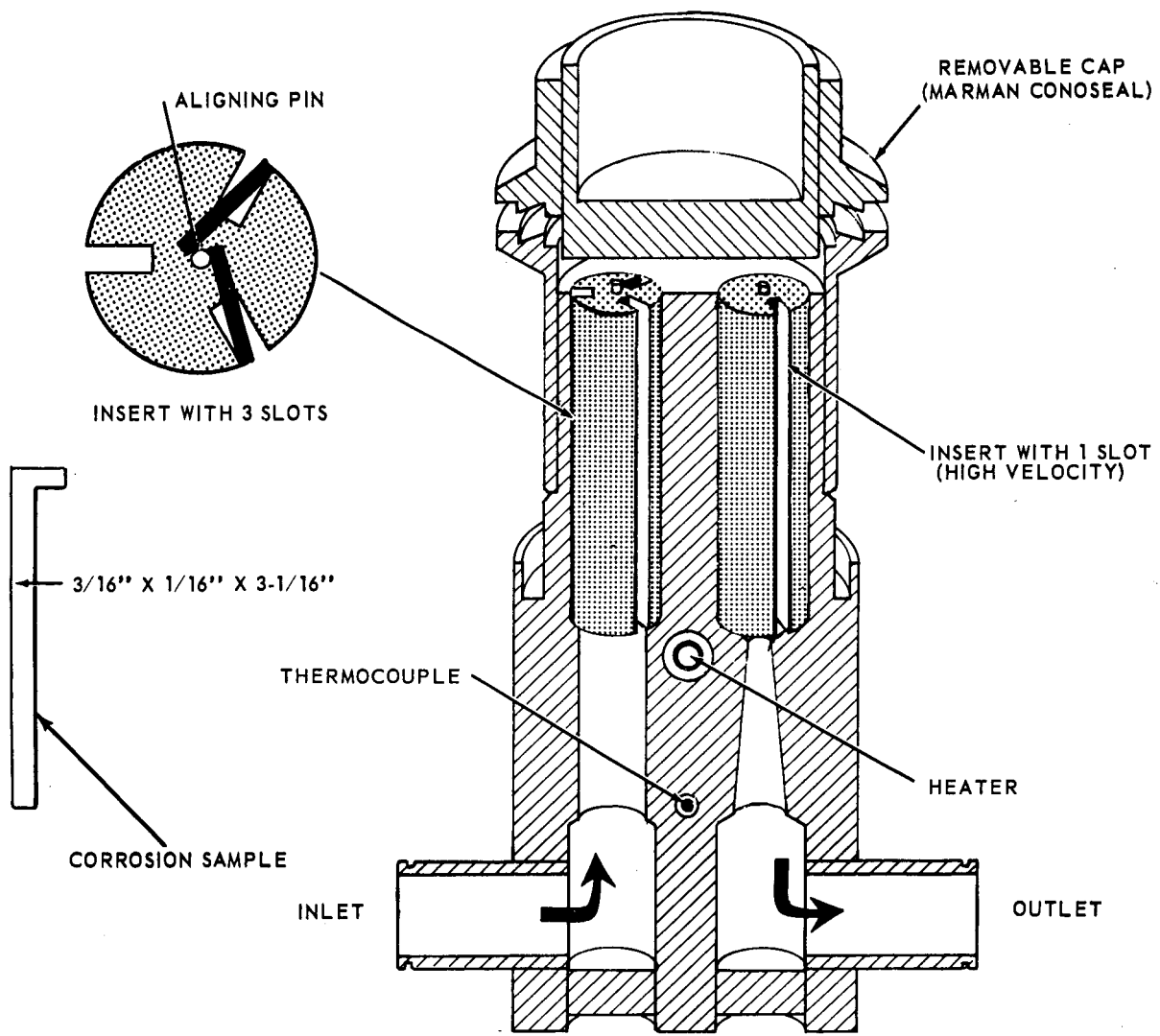
flow with a corrosion sample located within the slot. With the standard corrosion sample inserted, slots 0.115-inches wide by 0.180-inches deep were provided to give 30 fps sodium with one passage per insert, or 10 fps velocity with three parallel passages per insert. With the foil sample located in the outlet passage of sample holder H3R, the insert was provided with a single slot measuring 0.0625-inch wide by 0.1875-inch deep to provide 30 fps velocity past the sample.

A total of seven sample holders are installed in each loop. The sodium flows in series through each sample holder and past each sample as listed below:

<u>Sample Holder</u> <u>Designation</u>	<u>Temperature</u>	<u>Number of Samples</u>	
		<u>10 fps</u>	<u>30 fps</u>
H1	Tmax-200 F	3	1
H2	Tmax-100 F	3	1
H3	Tmax	-	2
H3R	Tmax	3	1
C1	Tmax-100 F	3	-
C2	Tmax-200 F	3	-
C3	Tmax-500 F	6	-

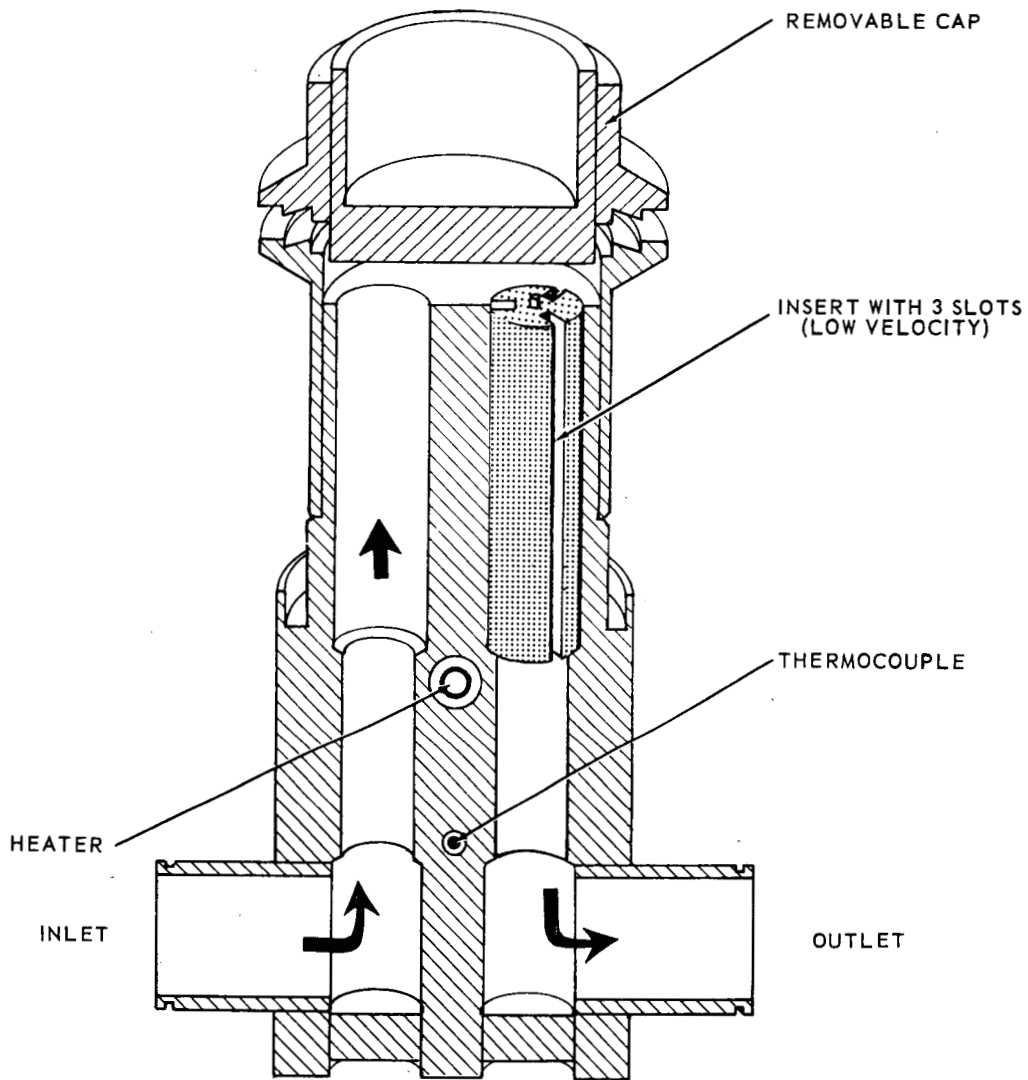
High velocity slots were not installed in the cold leg since the loop pressure drop was high and the velocity in power plant heat exchangers is generally less than 10 fps. At a sodium flow of 1 gpm the loop pressure drop was calculated to be approximately 70 psi with the bulk of this drop in the high velocity flow passages. A diffuser nozzle was incorporated on the discharge side of the high velocity sections in the hot leg sample holder to minimize pressure drop losses. See Appendix E for additional information on pressure drop in the sample holders.

Figures III-3 and III-4 show a cross-section of a typical hot leg and cold leg sample holder for the standard corrosion samples. Note the central location of the preheater and thermocouple in all holders and the series



276-35

FIGURE III-3 TYPICAL HOT LEG SAMPLE HOLDER



276-36

FIGURE III-4 TYPICAL COLD LEG SAMPLE HOLDER



flow of low velocity (3 passages) and high velocity (1 passage) in the hot leg holders. Manufacturing drawings of all sample holders are presented in Appendix F.

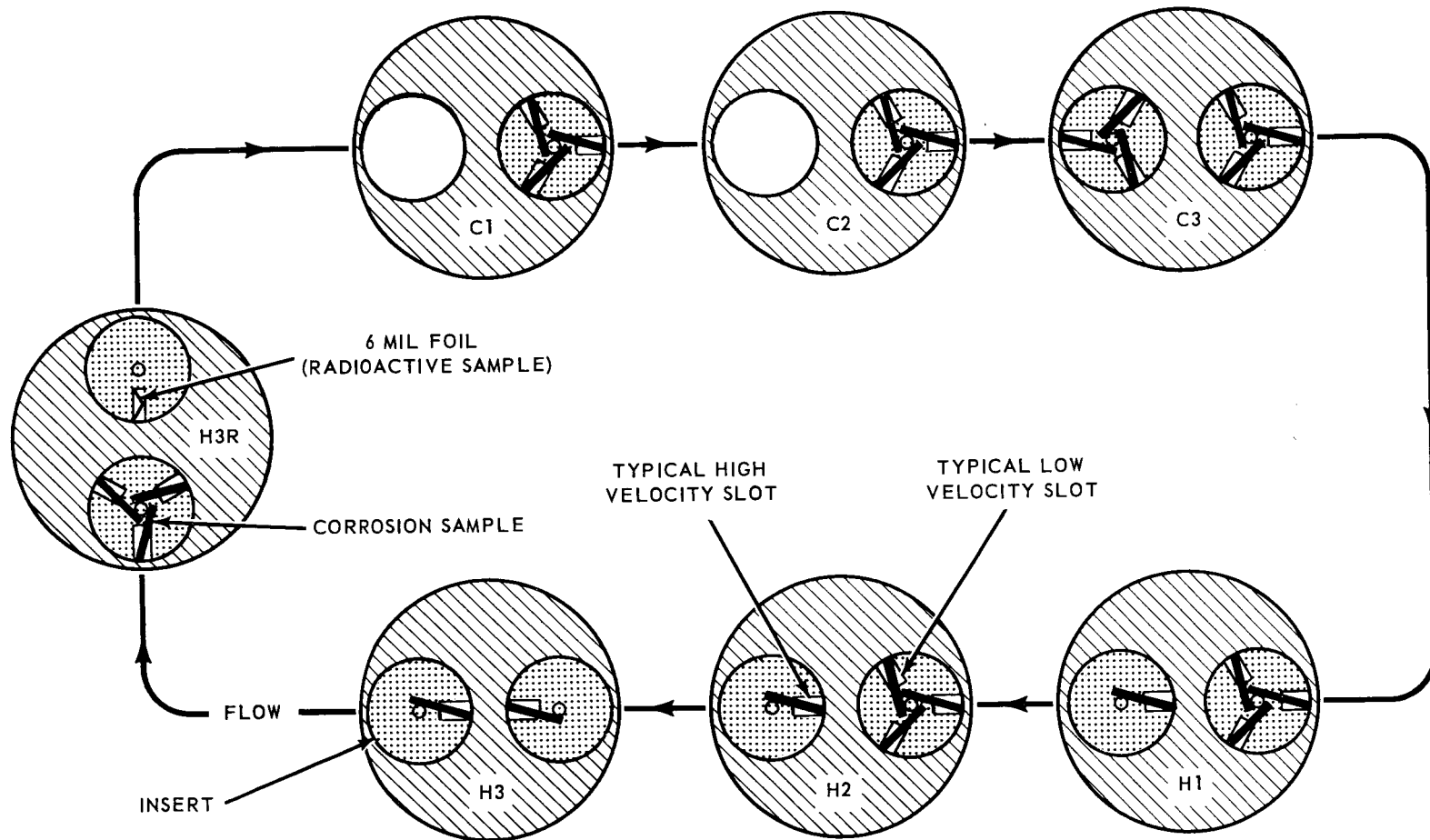
Figure III-5 illustrates the top view of a complete loop sample loading pattern. Additional inserts can be installed in C1 and C2 sample holders. Such action would increase the loop pressure drop or decrease the maximum flow rate.

## B2 Main Heaters

The main heaters are vertical shell and tube type heat exchangers having seven heater thimbles (tubes) projecting upward from the bottom head. Each thimble accommodates a snug fitting,  $\frac{1}{2}$ -inch diameter, cartridge heating element rated for operating continuously for one year at a surface temperature of 1600 F.

Cartridge heating elements (Firerods) furnished by the Watlow Engineering Company are used because they have an operating temperature and heat flux rating high enough to provide the required 1300 F outlet temperature with a one gpm flow and provide a compact monometallic heater unit. Refer to Figure III-6. Note the doughnut baffle tacked to the shell midway through the unit and the material code mark, "B" for the  $2\frac{1}{4}$ -1Mo steel, on the tube-sheet. This particular unit was installed in loop number 5. Manufacturing drawings of both the 300 F  $\Delta T$  heater (No. 1) and the 100 F  $\Delta T$  heaters (No. 2 and No. 3) are presented in Appendix F. The dimensions of each type of heater are the same for all loops but the fabrication material is appropriate for the particular loop.

Tolerances used in fitting the heater elements to the thimbles were at least one mil closer (0.003-inch) than the manufacturer's recommended tolerance for a one years operating life. Refer to Figure III-7 for heater fitting design curves. All heating elements in the highest temperature heater (Number 3) will be operated at the lowest possible voltage to reduce the heat flux ( $\Delta T$ ) and to lengthen the operating life. The design heat flux for the number 1 heater (preceding the H1 sample holder) is approximately 45,000 Btu/hr/ft<sup>2</sup> (93 watts/in<sup>2</sup> or 75% of manufacturer's rating).



SAMPLES: (25) REGULAR + (1) 6 MIL FOIL

FIGURE III-5 SAMPLE LOADING PATTERN

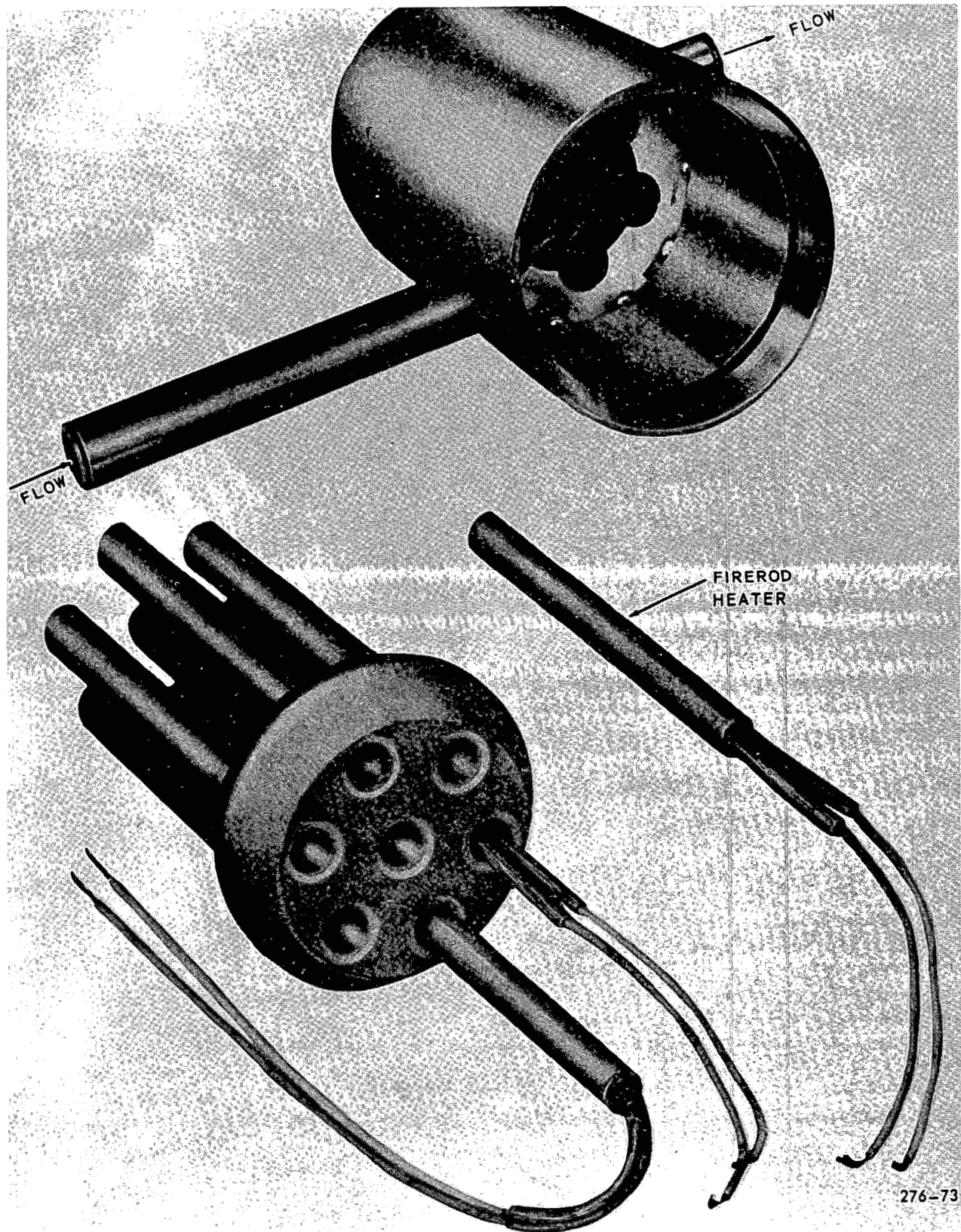


FIGURE III-6 MAIN HEATER

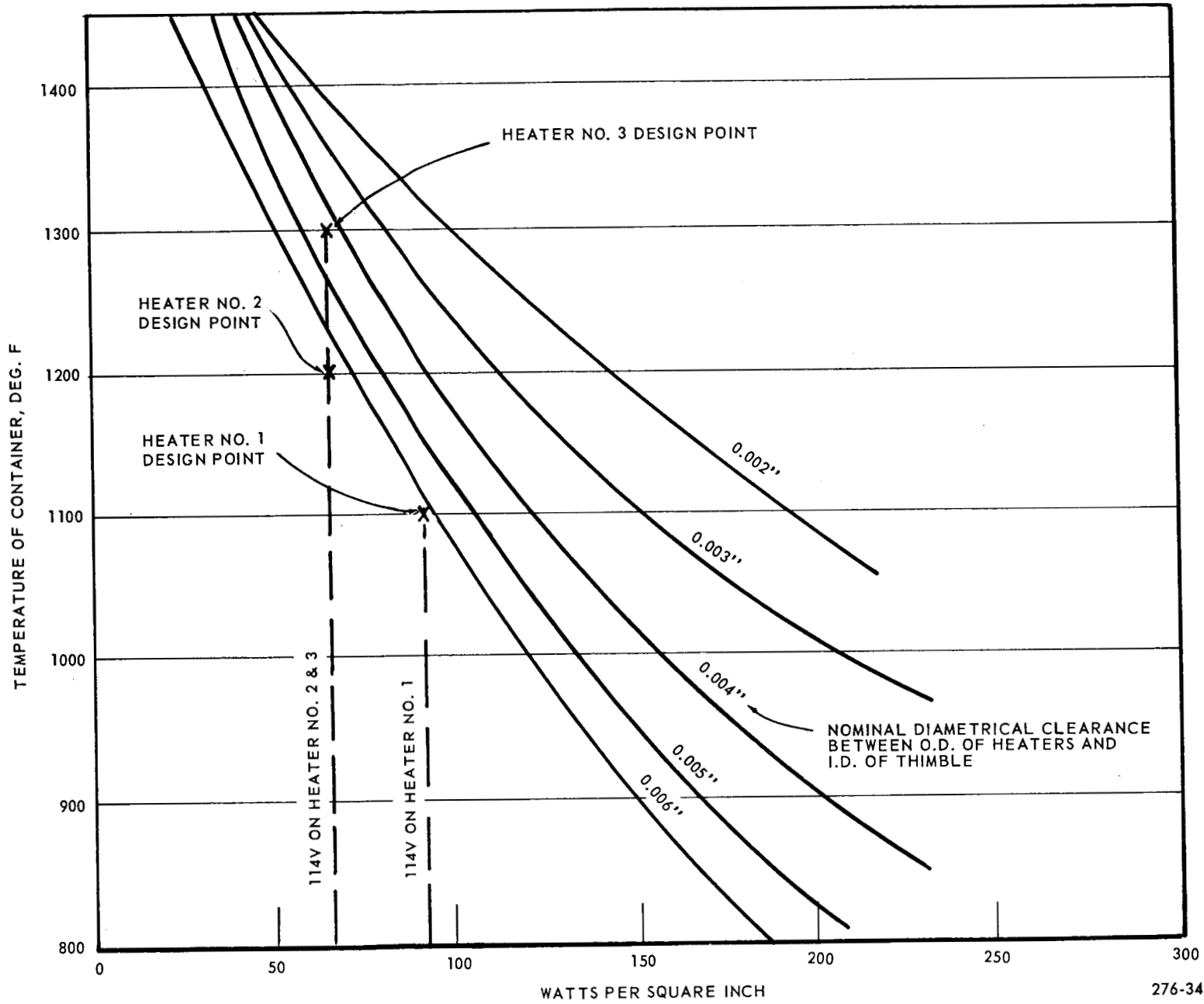


FIGURE III-7 HEATER FITTING CURVES

Heat flux for the number 2 (preceding the H2 sample holder) and number 3 (preceding the H3 sample holder) heater is 33,000 Btu/hr ft<sup>2</sup> (66 watts/in<sup>2</sup> or 79% of manufacturer's rating). Estimating the heat transfer coefficient in the heaters to be about 3,000 Btu/hr ft<sup>2</sup>/F, the estimated temperature film drop is 15 F in the number 1 heater and 11 F in the second and third heaters. This is believed to be satisfactorily low, but it should be kept in mind that the heater tube temperatures are the highest in their respective zone of the loop.

The individual heating elements are not considered removable because of their tight (shrunk) fit but it is anticipated that replacement of an element can be accomplished by drilling out the defective element and replacing it with a new unit. Internal baffles are provided to minimize flow channeling and to achieve more uniform heating.

Each heater has been designed to include one spare heating element (one out of seven) however all elements will be used initially at reduced voltage to minimize the burden and increase the potential life of the units.

Preheating of each of the main heaters is provided by an external tubular resistance heating element operating at one-half of manufacturer's rated voltage. This element can also be used to supplement heaters. Additional design details for the preheaters are found in Subsection B9. Details of the heating element power rating and heat control are presented in the next few paragraphs.

1. Main Heater No. 1 (300 F ΔT)

Seven - 2000 W 250 V Firerods

$$\begin{aligned}
 \text{on 225 volts} &= 14,000 \times \left(\frac{225}{250}\right)^2 = 11,350 \text{ watts} \\
 \text{Heat Required for Na} &= \underline{10,680} \\
 \text{Excess for heat losses} &= 670 \text{ watts}
 \end{aligned}$$

- a) one element is controlled by the 250 volt automatic control variac.
- b) one element is controlled by a manual variac.

- c) one element is on a manual circuit breaker.
- d) the remaining four heaters are connected to two on-off circuits with two heaters on each circuit.

2. Main Heater No. 2 (100 F ΔT)

Seven - 700 W, 125 V Firerods

$$\begin{aligned} \text{on 112.5 V} &= 4900 \left( \frac{112.5}{125} \right)^2 = 3,960 \text{ watts} \\ \text{Heat for Na} &= \underline{3,560} \\ \text{Excess for losses} &= 400 \text{ watts} \end{aligned}$$

- a) one element is controlled by the 125 v auto-control variac.
- b) three elements are controlled by a manual variac.
- c) three elements are controlled by a manual on-off circuit breaker.

3. Main Heater No. 3 (100 F ΔT)

Same elements as for No. 2 Heater

- a) One element is on the 125 v auto-control variac.
- b) Six elements are connected to a manual variac.

4. Automatic Control Variacs

Two variacs (one model W20H and one Model W20) are ganged together and driven by the control motor.

The W20H variac is a 250 volt unit and is used for one of the 2000 watt elements in heater No. 1.

The W20 variac is a 125 volt unit and is used for one 700 watt element each in the No. 2 and in the No. 3 heaters. Control of the autovariacs and resulting power to the heaters is described in Section III E.

B3 Coolers

There are a total of five coolers installed in the piping of each loop to provide forced air sodium cooling. The three main coolers are mounted horizontally in the cold leg portion of the loop and two are incorporated in the bypass purification loop. One of these is mounted horizontally

(preceding the cold trap and plugging indicator) and the other is an integral part of the plugging indicator. One-inch, Schedule 40 piping for the ferritic coolers was supplied to the Brown Fintube Company for attachment of longitudinal fins. The 316 SS cooler finned tubing was purchased to specification. The piping in all cases met material specification for the cold leg side of the loop. The longitudinal fins are 35 mils thick and approximately 18 inches long. The finned section of each cooler is jacketed with 1/16-inch mild steel covering to form the air duct.

The first two coolers in the cold leg preceding sample holders C1 and C2 are designed to reduce the sodium temperature by 100 F or about 12,000 Btu/hr with the sodium temperature as low as 800 F and with the inlet air at room temperature. These units, fabricated from a single pass pipe, are 18" long and were calculated to have a pressure drop on the air side of less than 1-inch of water at an air flow of 100 SCFM each.

The third, and larger capacity main cooler (300 F  $\Delta T$ ), which precedes sample holder C3, is constructed with three passes of 18 inch long finned piping. The finned piping is arranged on a 3-inch triangular pitch. Material from the same stock as the smaller coolers was used to fabricate this unit. The pressure drop on the air side of the larger cooler is also less than 1-inch of water at an air flow of 300 SCFM. The physical location of the coolers are apparent on Figure III-1 and on the loop assembly drawing shown in Appendix F.

Each of the six test loops has a separate forced air cooling system. A Buffalo Forge Company Model No. 22 blower with a capacity of 500 SCFM at 2½-inches of water column directs inlet room air through separate ducts past the individual coolers and discharges the hot air outside the test building through a 6-inch duct system. A single, motor driven damper is used to control some of the air flow in the main supply duct, while manually adjusted dampers in the individual cooler ducts are used to establish proper distribution from each cooler to obtain desired temperature conditions.

The two coolers in the oxide control section are identical to the 100 F  $\Delta T$  units except that they are shorter (15 inches long) and there are no air ducts. Cooling rates are adjusted by removing some of the thermal insulation that is normally in place around these units. Natural convection air flow removes the heat from these coolers. Additional cooling can be obtained by circulating the air with a portable fan or blower.

All cooler sections have preheat tubular heaters fastened between the fins. Preheater information is found in Subsection B9.

#### B4 Surge Tanks

The surge tank for each loop is designed for 1000 F at 50 psi operating conditions and is fabricated from 8 inch schedule 40 pipe, 14-inches in length. Flat plate heads,  $\frac{1}{2}$ -inch thick, close each end. The material used in the construction of the surge tank and level probe is the same as that specified for the cold leg section of the loop. The tank is located at the high point of the piping system and is between the main flow meter and the inlet side of the electromagnetic pump. See Figure III-1 or the loop assembly drawing in Appendix F.

The tank is oriented vertically and is of the flow-through type for potential gas de-entrainment. The sodium inlet nozzle is located 3-inches above the bottom head. A flat plate type, anti-vortex baffle is positioned across the top diameter of the outlet pipe.

A resistance type sodium level probe is located as far as possible from the inlet and is an integral part of the bottom head. It extends into the vessel for a distance of 12-inches and the practical measuring length is about 10-inches. The read out meter scale indicates percent level so the 10% level marks represent inches of level in the tank. Calibration of the units are described in Section V. Normal sodium level during operation is 4-inches to 5-inches above the bottom corresponding to about one gallon of sodium. The surge tank top head contains a vapor trap and inert gas connection identical to that provided on the dump tank.



B5 Electromagnetic Pumps

The following electromagnetic pump specifications were originally established to satisfy system design requirements:

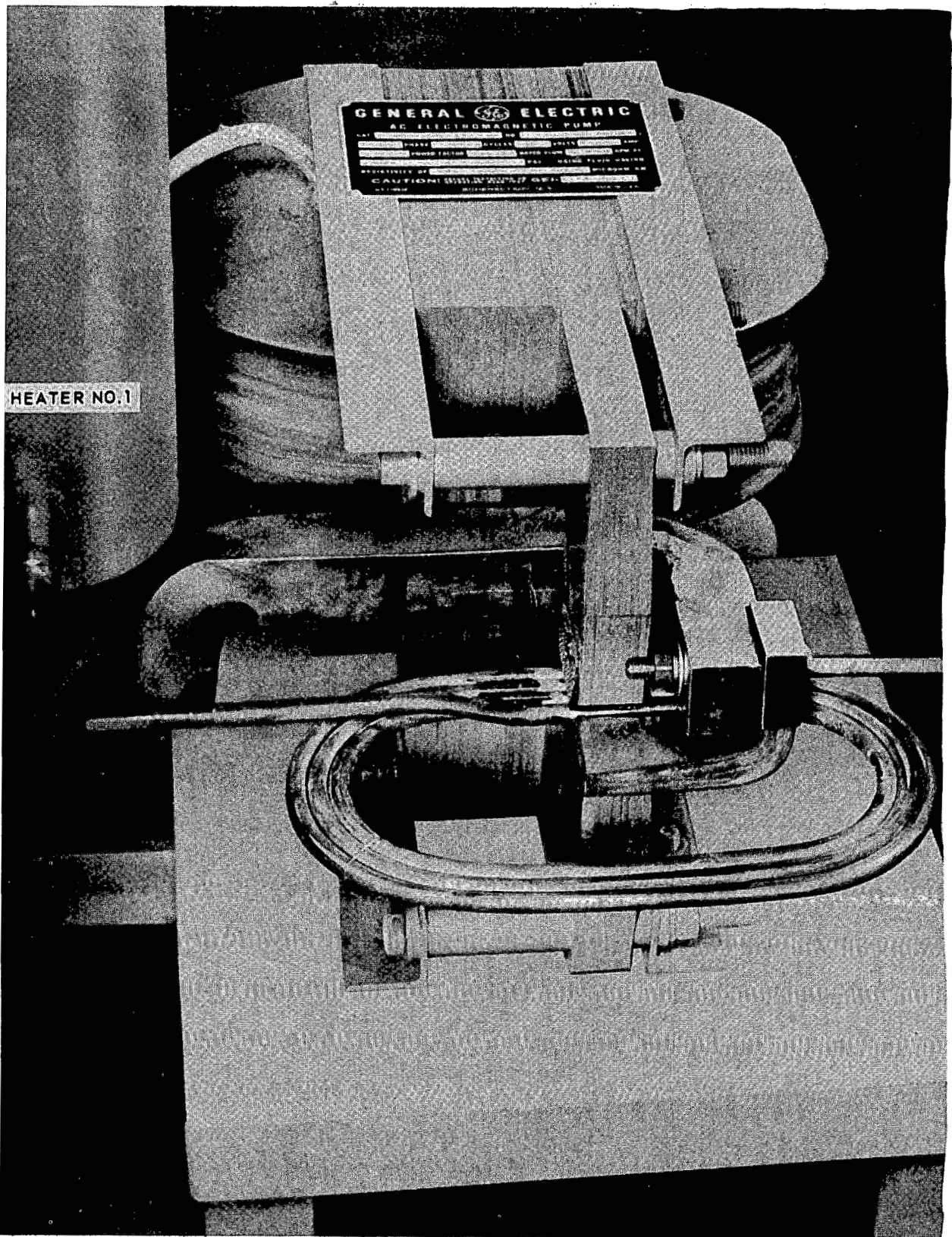
Maximum sodium flow-----	1 gpm
Flow control-----	continuous
Developed head-----	75 psi
Maximum sodium temperature at pump cell-----	1000 F
Quantity and cell material-----	1 of type 316 stainless steel 3 of $2\frac{1}{4}$ Cr-1Mo steel 2 of 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti steel

A General Electric single phase, AC, electromagnetic pump composed of a pump cell, pump transformer, and capacitor unit was supplied for each loop. Figure III-8 illustrates the cell mounted in the pump transformer. The type 316 stainless steel and the  $2\frac{1}{4}$ Cr-1Mo steel pumps were ordered in accordance with G.E. catalog numbers. Modified manufacturing drawings were prepared for the 5Cr pump cells. All pump assemblies are identical except for cell material and brazing procedures. Cell manufacture was in accordance with G.E. drawing 585D158 with -G2, -G4 and -G6 describing the 316 stainless,  $2\frac{1}{4}$ Cr, and 5Cr steel cells respectively. Each pump cell is fabricated of 3/8 inch O.D. tubing, with four pass construction in order to provide the specified developed head. Refer to Figure III-9.

Sodium flow is regulated by manual adjustment of applied 60 cycle voltage over the range from 0 to 270 volts. Line current at full voltage does not exceed 30 amperes. More detailed descriptive information of the electromagnetic pumps with characteristic performance curves are available in General Electric publications GEA-6395A and GEI-56200 (not included herein).

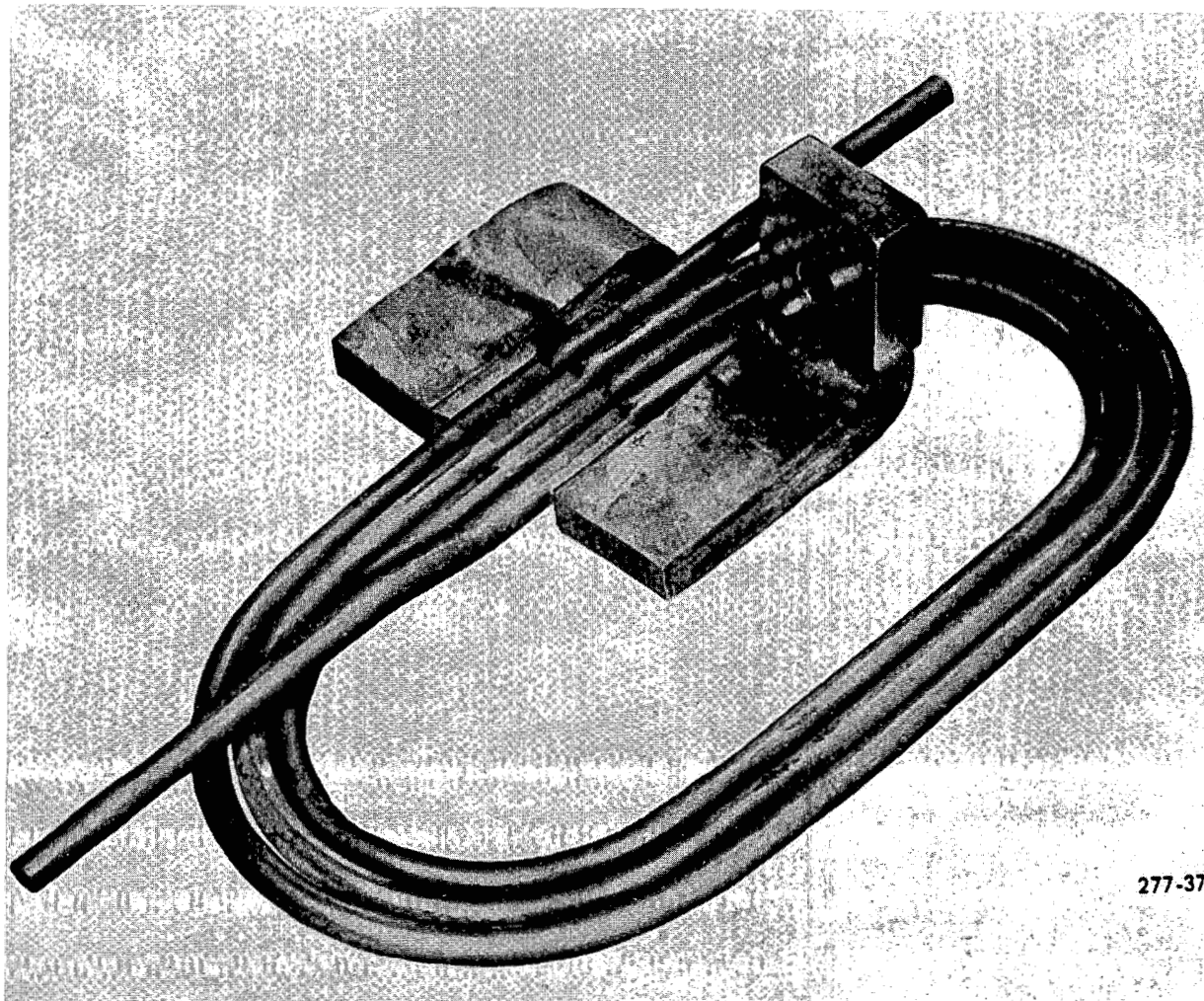
B6 Purification System

The purification system is in parallel with the main corrosion loop but normally is isolated by closed valves. When this system is operating, flow is common through the pump and appropriate parts of the piping between the surge tank and the pump. This system is fabricated of the same material as the cold leg of the loop.



276-14

FIGURE III-8 PUMP ASSEMBLY



**FIGURE III-9 FOUR PASS PUMP CELL**

Sodium flow through the purification system passes the bypass inlet valve, the bypass EM flow meter, the auxiliary finned cooler, and either the cold trap or plugging indicator depending on whether the cold trap or the plugging indicator outlet valve is open and then is returned to the loop through the appropriate valve and piping. See the loop flow sheet, Figure III-1, for component arrangement.

The trap and indicator have separate return lines to the main loop, since only one of the two units is used at a time. Sodium flow through either unit is controlled by altering the pump voltage (which also varies the main loop flow) or by using the  $\frac{1}{2}$ -inch stop valves as throttle valves. Use of the stop valves for throttling is possible but not recommended. All pipe lines and components are preheated with tubular heaters. Extra heating can be supplied to the plugging indicator and cold trap outlet piping and valve by switching to higher voltage. Details are presented in subsequent sections of the report.

The forced convection cold trap is used to control the oxide content of the system sodium. Sufficient oxide to maintain saturation at 450 F is expected to be in the system from normal operation; additional oxide in the form of  $\text{Na}_2\text{O}$  can be introduced through the dump tank charging port if necessary. The oxide level is adjusted by periodically directing flow through the cold trap at a temperature corresponding to the desired oxide saturation temperature. A plugging indicator is used to measure the system oxide content after cold trap operation.

The cold trap temperature can be measured with either the outlet pipe thermocouple or with the thermocouple welded to the middle of the body of the trap. The region of the cold trap above the outlet nozzle forms an inverted cup-type surge tank. Gas trapped during the filling procedure should provide pressure relief for the purification system during temperature changes when the isolation valves are closed. During operation the loop surge tank will provide pressure control.

During cold trap operation, heat is dissipated from the incoming sodium to the surrounding room air by natural convection from the finned auxiliary cooler upstream of the trap (also used during plugging runs) and from the cold trap vessel walls. Appropriate insulation jackets are removed to control the cold trap temperature. Additional cooling can be obtained by forcing the air across the trap with a blower. The system is designed for the following sodium conditions:

Flow	0.2 gpm
Inlet Temperature	800 F maximum
Outlet Temperature	300 F minimum
Residence Time	5 minutes

The cold trap is constructed of 4-inch, schedule 40 pipe, 16-inches long and is packed with knitted stainless steel "York" mesh for all loops. The unit is oriented for vertical flow with the inlet on the bottom and the outlet on top.

The plugging indicator provided in each loop to determine the oxide level in the sodium is a device which detects oxide saturation temperature by measuring the temperature corresponding to a sudden flow decrease resulting from precipitation and deposition of the oxide in multiple flow restricting orifices. The temperature is measured with a thermocouple welded to the edge of the orifice (outside the pipe). The plugging (orifice) plate for the loop is perforated with four 0.0465-inch diameter holes. The plate is welded across the discharge end of the 1-inch finned indicator cooler. The system, including

the auxiliary upstream cooler, the indicator cooler, and the plugging plate, is designed for a flow of 0.1 gpm at a sodium inlet temperature of 800 F and a minimum saturation temperature of 300 F (about 10 ppm oxide). Cooling rate at the orifice plate is held in the region of 10 F/minute or less by removal of appropriate insulation jackets surrounding the finned coolers and cold trap. Insulation closest to the plugging plate is removed first.

#### B7 Valves

Four sodium valves, one to isolate the dump tank from the main loop and three to isolate the purification system, are used in each loop. Except for the loops where the body is made of ferritic material, the valves are similar to those commonly used elsewhere. The valves for the stainless steel loop are Powell model No. 2475 BSF. They are  $\frac{1}{2}$ -inch, stainless steel globe valves rated for 150 lb service or 40 psi at 1000 F. The bellows plate is welded to the bonnet for improved tightness. The valves for the chrome-moly and bimetallic loops are identical to the type 316 stainless steel valves except that the bodies were fabricated of  $2\frac{1}{4}$ Cr-1Mo steel. In all cases the bellows are fabricated of type 316 stainless steel. All actuators are identical. Valve bodies of 5Cr steel for loops #4 and #6 could not be obtained since none of the valve manufacturers contacted were willing to quote on this material for the body.

In order to minimize the risk of valve failure or metallurgical effects resulting from miscellaneous materials exposed in the loops, the following design philosophy was used:

- a. No valves to be located in main circulating loop flow path.
- b. Valves to be maintained at the specified minimum temperature region of the main loop.
- c. Valves to be oriented with bellows away from the main loop sodium stream.

#### B8 Dump Tanks

The dump tanks are fabricated from 8-inch schedule 40 pipe with standard welding cap heads on the ends making each of the tanks 30-inches in overall length. Each tank is mounted in the loop with the major axis in the horizontal plane. Volume of the vessel is adequate to contain the entire loop charge of sodium (about 35 lb) at 1000 F with approximately 30 percent free volume. Materials of construction of each of the tanks are the same as the material in the cold leg of the loop.

A two inch port provided with a Marman Gonoseal flange assembly is attached to the top of the tank. This port is used for charging sodium into the system and access for sampling and impurity additions. A concentric 8-inch flange is attached to this port, similar to the sample holders, to permit the use of a dry box in removing sodium samples or for adding  $\text{Na}_2\text{O}$  if necessary. A  $\frac{1}{2}$ -inch pipe line with a  $\frac{1}{2}$ -inch sodium valve connects the bottom of the dump tank to the main loop and permits transfer of sodium between the loop and tank. This line is used for filling and draining the test loop and is maintained above the oxide saturation temperature for emergency dumping if necessary.

To minimize deposition of sodium vapor in the gas and vent lines a vapor trap is attached to the top of the dump tank and is connected to the gas system. The vapor trap is 12 inches long and is constructed of 1-inch schedule 40 pipe which will perform the following functions:

- a. Permit relatively low gas velocities, compared with the  $\frac{1}{2}$ -inch O.D. tube gas lines and control valve, during venting operations.
- b. Prevent excessive heat losses during normal operation.
- c. Provide temperature below the sodium melting point at the outlet end.

Both vapor traps in the stainless steel loop (#1) only were packed with knitted wire mesh, identical to the cold trap packing. This was done in an attempt to further minimize transfer of sodium particles through the vent system since this loop is designed to be operated at higher temperature than the others.

#### B9 Preheaters

Electric heaters are provided on all loop piping and components to preheat the system to 500 F before filling or during "isothermal" operation of the loop. The heating methods vary with the application and use factor. The amount of electrical heat available for each circuit is shown in Subsection III D on Figure III-16 - Loop Power Distribution.

Below is a description of the various preheater applications.

- a. Each of the sample holders is equipped with a 175 watt heating element (Firerod) 2-inches long inserted into a socket in the main body of the sample holder.
- b. The loop piping normally operating at temperatures exceeding 800 F, such as the hot and cold leg sections and main heaters, are provided with tubular electric heaters operating at  $\frac{1}{2}$  rated voltage ( $\frac{1}{4}$  power).
- c. The loop piping not subject to operating temperatures above 800 F and not requiring energizing above 500 F, such as the drain line and flow meters, is wrapped with heating cable. (Asbestos insulated, armored, nichrome wire.)
- d. The surge tank and dump tank are provided with tubular resistance heaters for use during normal operation as well as during preheating. For long life they are operated on a fraction of the rate voltage.
- e. The purification leg, except for the bypass flow meter, is heated by tubular resistance heaters. A single unit is used for the auxiliary cooler and plugging indicator cooler. This unit has a two heat switch (120/240 V) to provide low heat (approximately 313 watts) for normal preheat use and a high heat, 1250 watts for rapid recovery at the end of a plugging run. The outlet valve and pipe for the plugging indicator is heated by a single 188 watt unit. The cold trap is heated by a single 500 watt tubular resistance heater located under the trap like a hot plate. The tubular heater for the cold trap outlet valve and pipe has a two heat switch (120/240V) to provide 188 watts for normal preheat and 750 watts for reheating during cold trap operation.
- f. On loop #1 the main coolers were provided with 60 cycle induction heating coils which heated the mild steel cooler air ducts. The potential advantage of this method is potential long heater life since the heaters can be energized during loop operation with the coil operating at relatively low temperature. The coil is external to the insulation so modification or maintenance is convenient.

The induction heaters for the loop #1 coolers consist of number 14 AWG quadruple formvar wire wound on the outside of the 1" thermal insulation with approximately ten turns per inch. Heat is generated in the 1/16-inch thick mild steel shell (duct) surrounding the fintube coolers. Heat input can be adjusted by changing the number of turns of the wire between the two points where line voltage is applied. The induced current should be directly proportional to the number of turns and the heating will vary as the square of the induced current.

The use of induction heating for the coolers was abandoned after operation of the first loop because the design and location of neighboring components prohibited the winding of the entire length of the cooler. On subsequent loops tubular resistance heaters were used for main cooler preheating. One heater was inserted between the fins and under each of the single-pass coolers and under two of the three finned sections in the 300 F  $\Delta T$  cooler of each loop.

### III C Assembly and Installation

This subsection covers in general the procedures and methods employed in the fabrication and assembly of the six sodium test loops. Applicable code specifications are included with each operation where required.

- C1 Weld Preparation and Procedure
- C2 Stress Relief
- C3 Quality Control
- C4 Final Assembly and Installation

#### C1 Weld Preparation and Procedure

All loop welding was performed in accordance with Section VIII of the ASME Boiler Code. Materials preparation and welding procedures used in the fabrication of the loops are generally outlined in the following paragraphs.

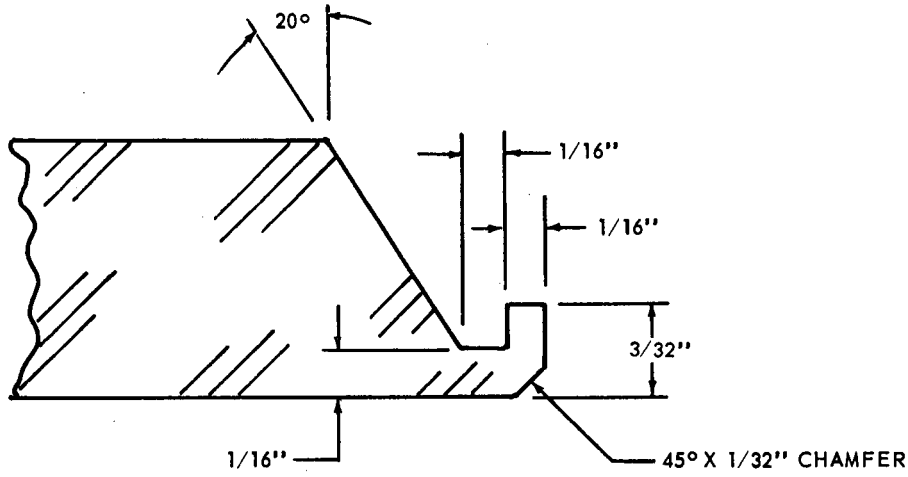
##### Welder Qualification:

All welding operations for the fabrication of the loops were performed by welders properly qualified under the performance qualifications required of Section IX and referenced in Section VIII of the ASME Boiler Code.

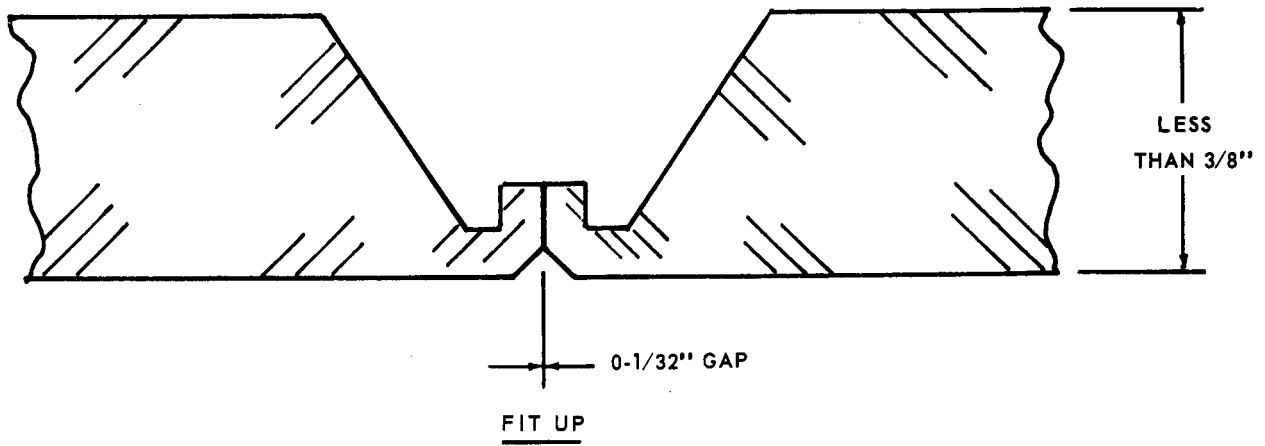
##### Weld Preparation:

The "W" design was used in the preparation of weld joints for piping in order to obtain sound welds with less difficulty. Refer to Figure III-10 for details. Although this type of weld preparation requires additional machining compared to conventional "butt weld" preparation, it provides sound welds while eliminating the requirements for radiographic inspection.





EDGE PREPARATION



276-33

**FIGURE III-10 WELD PREPARATION & FIT UP FOR MASS TRANSFER PIPING**

**Welding Process:**

Welding was performed with the manual, tungsten inert gas-shielded arc, welding process with nonconsumable electrodes.

**Filler Material:**

Weld Filler material conforms with the analysis of A7 weld metal which contains more than 1% ferrite, as set forth in Section IX, Table Q 11.3 of the ASME Boiler Code.

**Shielding:**

Welding grade argon gas was used for shielding during the welding processes.

**Electrodes:**

Thoriated 2% tungsten electrodes were used in the welding processes.

C2 Stress Relief

All welded components and loop assemblies, where required, were stress relieved in accordance with Section VIII of the ASME Boiler Code. All the 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti Steel welds were stressed relieved but only the surge tanks and dump tanks (components larger than 4-inches diameter) of the three 2 $\frac{1}{4}$ Cr-1Mo loops were relieved. None of the stainless steel components were stress relieved.

Furnace heating was used wherever practical. However, due to size limitations of the furnace and loop assemblies, some of the final loop welds in the 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti steel sections were locally treated with cartridge heaters (Firerods) imbedded in fitted, removable stainless steel holders wired to the "field" weld area and surrounded with insulation. Manual voltage adjustment was used to control the rate of temperature increase or decrease.

In accordance with Section VIII of the ASME Boiler Code, the following conditions were maintained during stress relief operations:

- a. Furnace temperature was not greater than 600 F when welded assemblies were inserted.
- b. Above 600 F the heating rate did not exceed 400 F per hour.
- c. At the completion of heating period the temperature was held at 1350 F for 20 minutes.
- d. Cooling rate did not exceed 500 F per hour until temperature reached 600 F.

- e. Argon gas bleed through interior of welded assemblies was maintained at 10 scf/hr during the time the temperature was above 600 F.

C3 Quality Control

In an effort to provide rigid control of materials used in the loop construction, procedures were established to maintain positive identification of all parts at all times in the shop from the receipt of raw materials and vendor items through fabrication and assembly. Certified chemical and physical analyses were required of all construction materials to show compliance with purchase order and applicable ASTM specification. Table III-1 provides a list of procurement specifications. Upon acceptance, the materials were marked with paint and/or electroetch pencil in accordance with the following code:

- a. Identification of Materials:

<u>Material</u>	<u>Electroetch Pencil Mark</u>	<u>Color</u>
316 Stainless steel	A	Green
2 $\frac{1}{4}$ Cr-1Mo steel	B	Black
5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti steel	C	Red

In general, all pipe, fittings, valves, forgings, sheets and other parts purchased from outside were color coded with paint. As the pieces progressed through the shop in machining and cleaning operations the identification was maintained. Machined parts were marked with the electroetch pencil so as to provide a more permanent identity that would not be destroyed upon final cleaning or during welding in assembly. Periodic check analyses of certified materials were made by the GE-APED Chemical and Metallurgical Laboratory or by the Hales Testing Laboratory in San Jose, California. In addition, reserve material from the various lots were stored to permit additional analyses if considered desirable at a later date.

An example of the certified and check analyses that were obtained for all stock is presented below. These analyses were obtained for the corrosion sample stock:

TABLE III-1

MATERIAL SPECIFICATIONS

The following ASTM specifications were used in procurement of the reference materials:

<u>Item</u>	<u>316SS</u>	<u>2<math>\frac{1}{4}</math>Cr-1Mo</u>	<u>5Cr-<math>\frac{1}{2}</math>Mo-<math>\frac{1}{2}</math>Ti</u>	<u>ASTM Specification</u>
Pipe	X			A 312 GR TP 316
		X		A 335 Gr P22
			X	A 335 Gr P5C
Tubing	X			A 269 TP 316
		X		A 213 GR T22
			X	A 213 GR T5C
Fittings	X			A 403 GR WP 316
		X		A 234 GR WP 32
			X	A 234 GR WP 5
Rod or Forging	X			A 276 GR TP 316
		X		A 182 GR F22
			X	A 182 GR F5
Plate	X			A 246 GR TP 316
		X		A 387 GR D
			X	A 357 (5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti)
Filler* Metal	X			TP 316SS
		X		McKay 2Cr Mo
			X	Type 502, McKay 5Cr Mo

\* 1/16 DIA, type 309, McKay 25-12 was specified for austenitic-to-ferritic transition welds.

<u>Material</u>	<u>Composition Wt %</u>							
	<u>Cr</u>	<u>Mn</u>	<u>Mo</u>	<u>Ni</u>	<u>Co</u>	<u>Si</u>	<u>C</u>	<u>Ti</u>
Type 316 stainless steel****	17.5	1.6	3.0	11.5	0.05	--	--	--
	**	16.8	1.6	1.95	12.5	0.05	0.55	0.55
2 $\frac{1}{4}$ Cr-1Mo steel	*	2.28	0.52	0.96	--	--	0.24	0.10
	**	2.45	0.44	0.88	--	--	0.24	0.11
5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti steel	*	4.76	0.48	0.52	--	--	0.40	0.09
	**	4.92	0.40	0.51	--	--	0.39	0.09

All analyses are within specification.

\* Certified analysis

\*\* Hales Testing Laboratory results

\*\*\* G.E. Lab results Lab. No. 598 - Certified results were not available.

b. Cleaning:

Cleanliness throughout the fabrication and assembly of the test loops was emphasized. After fabrication, but prior to loop assembly, all parts contacting sodium or sodium vapor were cleaned with a commercial type (non-halogenated) degreasing solvent. Brushing, grinding, and sandblasting were used as necessary to remove scale. Following cleaning the open ends of each finished subassembly were sealed with tape or suitable caps. Because the chrome moly steel appeared to rust readily following degreasing, finished parts were wrapped with a desiccant paper (Orchard Paper Co. V.P.I. paper, MIL-1-3240A, Typel, Class 2 Style B) prior to final assembly. Precautions were taken during final assembly and installation to insure a clean system.

c. Weld Inspection:

All loop welds were dye penetrant tested. Welds which showed evidence of cracks during this test were repaired and tested again or rejected.

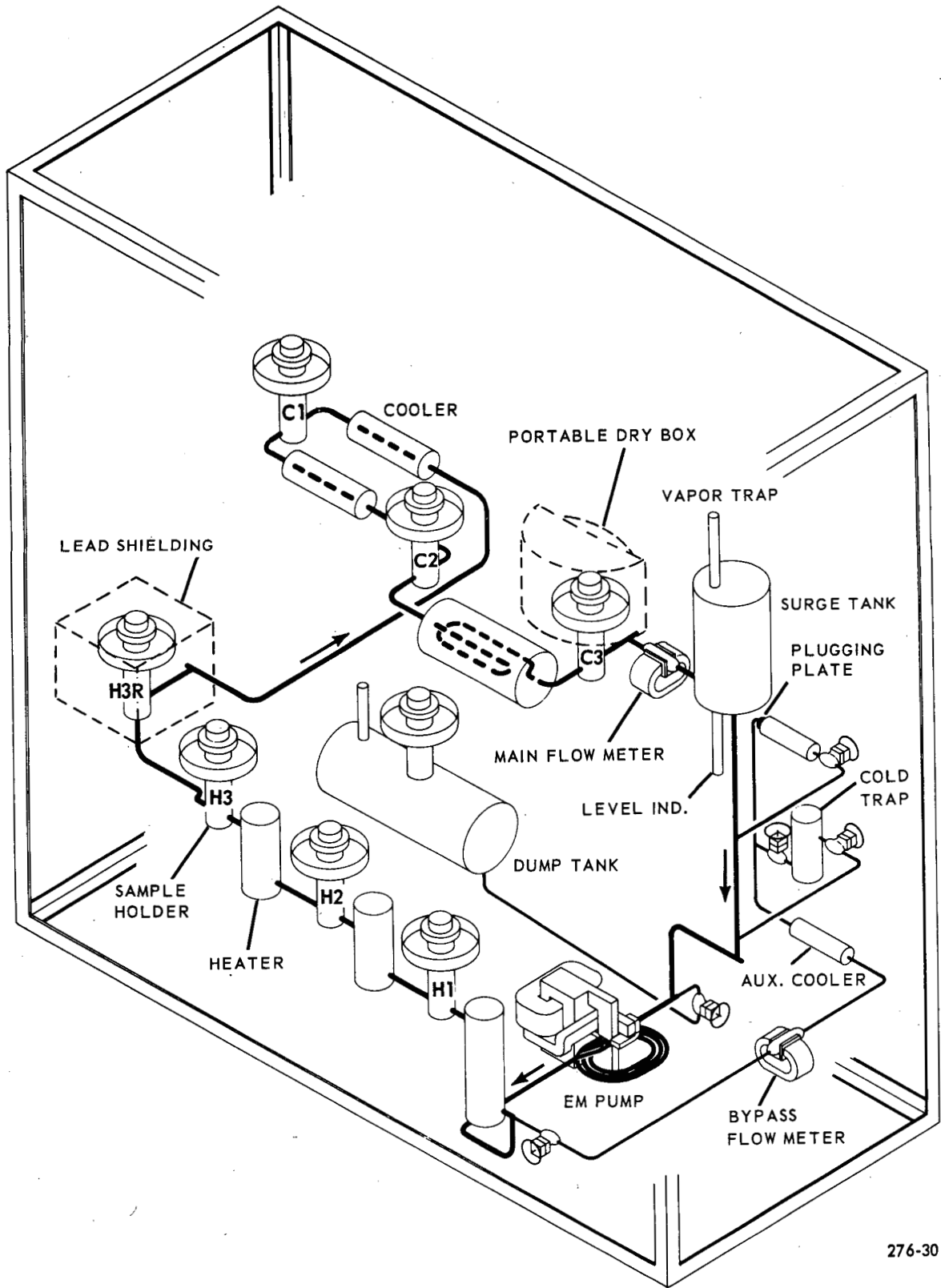
d. Leak Testing:

After assembly the loop was mass spectrometer leak tested at a pressure of 50 microns in the loop and with helium contained in a plastic bag surrounding the system. The maximum allowable leakage rate was 0.01 micron cubic feet per hour. In order to minimize delays in the system leak test some of the inaccessible components and subassemblies (i.e., heater tubes and level probe) were leak tested prior to final assembly. The combined hot and cold leg sections of the loops - including the heaters, sample holders, and main coolers - were pressure tested in accordance with the ASA Piping Code. Compressed nitrogen was used rather than hydrostatic testing in order to prevent subsequent delays in removing moisture prior to the mass spectrometer leak test operation. Gas pressure testing was performed in accordance with the following schedule:

<u>Loop</u>	<u>Test Pressure, psi</u>
All stainless	600
2 $\frac{1}{4}$ Cr-1Mo and bimetallic	950
5Cr- $\frac{1}{2}$ Mo-Ti and bimetallic	1350

C4 Final Assembly and Installation

Components described in the previous subsection were assembled into the test loop. Figure III-11 shows the loop arrangement with component identification and Figure III-12 shows loop #3 during installation. This figure shows the completed electrical thermocouple and air duct installation and partial completion of the thermal insulation. Components not insulated are the three main heaters, sample holders H1 and H2 and the bypass inlet valve. Holder H3 is insulated but the removable insulation cover for access to the flange is removed. On top of the sample holders are aluminum foil dust protectors that obscure the flanges. Steel strip, wired in place over the heater preheat units, and the heater cable wrapped around the bypass inlet valve are visible.



276-30

FIGURE III-11 LOOP ARRANGEMENT

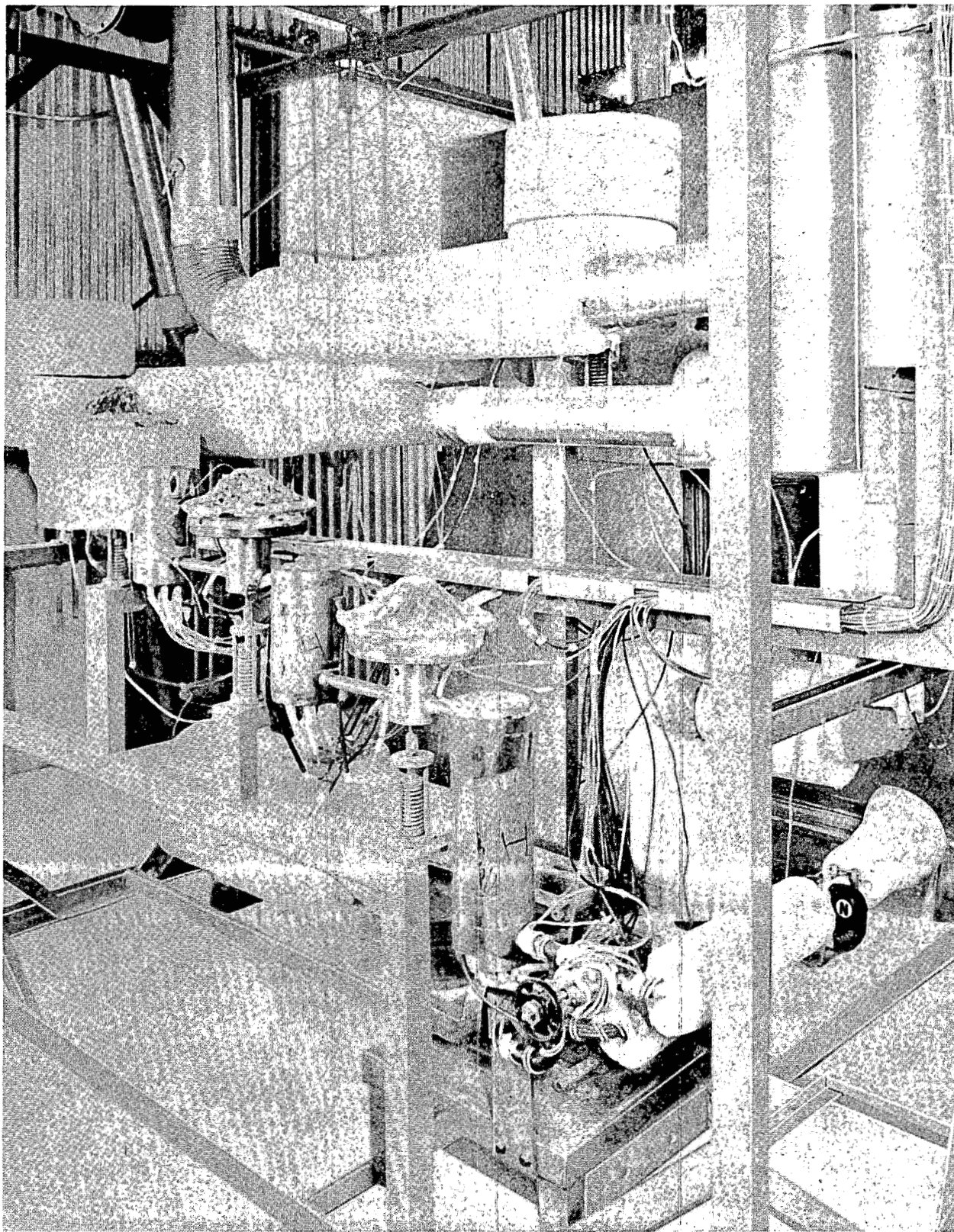


FIGURE III-12 PARTIALLY INSULATED LOOP



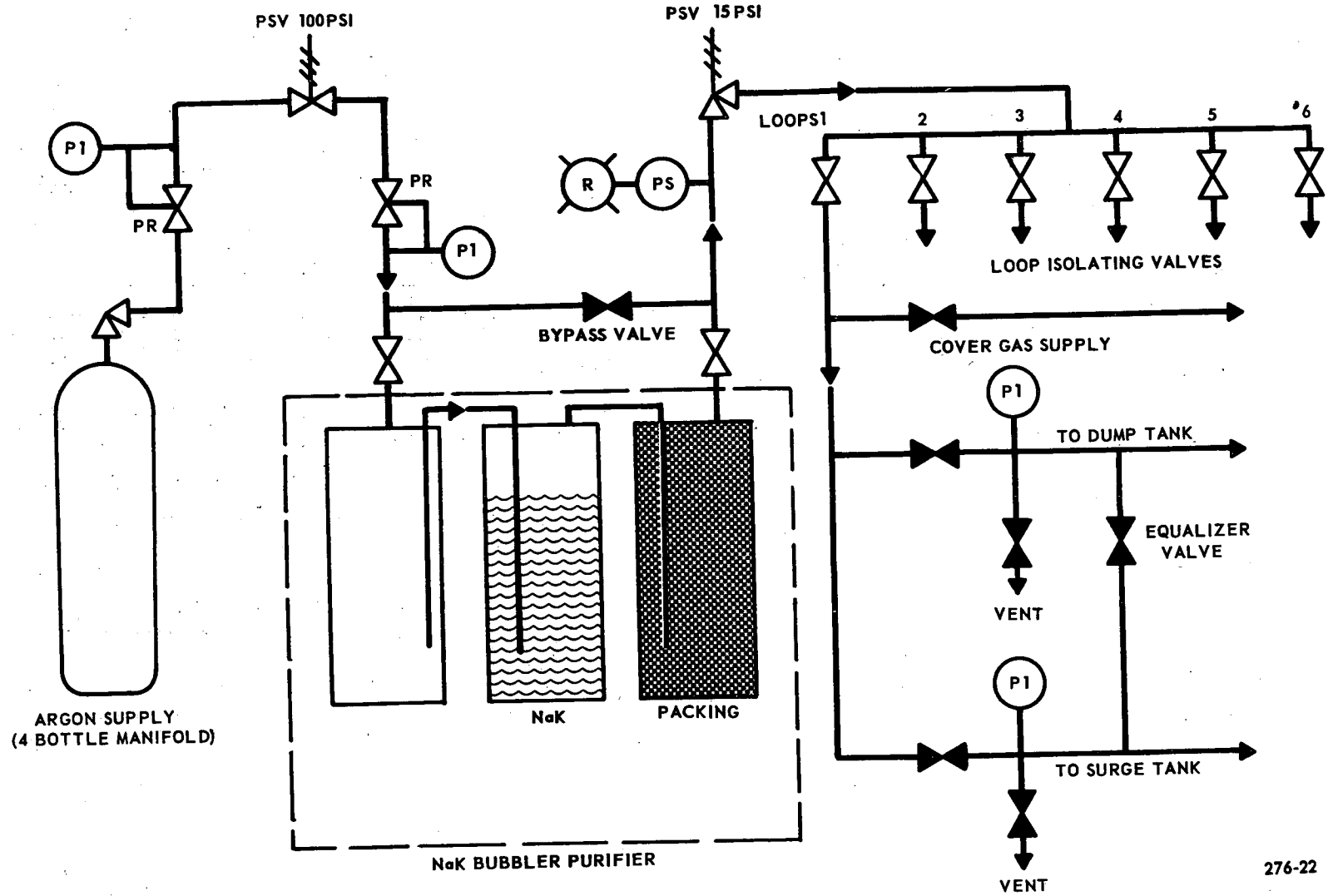
In order to minimize pipe stresses resulting from thermal expansion the sodium system was designed with only one rigid anchor point. To prevent excessive stresses at the pump cell, the inlet side was rigidly anchored to the pump frame while the discharge side was supported to permit motion only along the longitudinal axis of the cell piping. The dump tank was positioned in a pair of "V" supports attached to the loop frame while the remainder of the loop was supported entirely by spring hangers. This provides maximum flexibility during operation from room temperature to 1300 F. Spring hangers were adapted for support beneath sample holders H1, H2, H3, and C3 to facilitate use of a portable dry box during sample removal operations. Overhead spring hangers support the coolers and associated sample holders.

Wherever practical, horizontal piping runs were pitched at a nominal 1/8-inch per foot to allow maximum drainage of sodium from the loop into the dump tank at the completion of testing. The surge tank was located at the high point in the system and the dump tank was placed at the lowest elevation. Pockets of sodium trapped in the lower end of the sample holders, heaters and cold trap are not drainable.

After installation of electrical heaters and thermocouples the entire loop was insulated with Johns-Manville Thermobestos insulation up to 4-inches in thickness. The exhaust cooling air ducts were covered with fiberglass insulation 1-inch thick to prevent excessive heat losses to atmosphere in the test area.

The sodium surface in the dump and surge tanks was protected from the atmosphere by argon cover gas. Commercial grade argon, used as the cover gas, was purified before use in the system by passing through a NaK bubbler to remove potential traces of oxygen and moisture. All gas lines were also flamed prior to loop start up. The cover gas piping in the dump and surge tanks was interconnected to permit free gravity draining of the loop. A gas connection on the supply line was provided for use with the portable dry box to be used during operation of changing and/or removing corrosion samples. Refer to Figure III-13 for the Cover Gas Flow Sheet.

47



276-22

FIGURE III-13 COVER GAS FLOW SHEET

The loops were installed in the test building in the order that they were delivered from the shop. The arrangement of the loops, panels and other equipment in the building is shown on Figure III-14. The material composition of each loop is identified as follows:

<u>Loop</u>	<u>Hot Leg</u>	<u>Cold and Purification Leg</u>
#1	316SS	316SS
#2	316SS	2 $\frac{1}{4}$ Cr
#3	316SS	2 $\frac{1}{4}$ Cr
#4	316SS	5Cr
#5	2 $\frac{1}{4}$ Cr	2 $\frac{1}{4}$ Cr
#6	5Cr	5Cr

The installation of the first loop was completed during June and the sixth loop was completed during August of 1960.

#### III D Electrical Power Supply

The electrical power supply system is designed to provide reliable power for the heaters, controllers, alarms and blower for each of the six sodium test loops. Effectively, the supply for each loop is independent of all other loops and should be comparable in reliability to the utility 60,000 volt supply system. Owing to improved load distribution, and installation of sectionalizing switches to permit washing of insulators outside and within the plant, outages of the power system have been very infrequent recently. Only three short-term outages affecting APED are known to have occurred since September, 1959.

See Figure III-15 for a one line diagram of the power supply system from the plant substation to the individual loop circuit breakers. As shown, the 480 volt, 60 cycle 3 phase supply for the sodium test building is protected by a 600 ampere breaker in the 1000 KVA distribution center which is fed by a 12,000 volt to 480 volt load center transformer. This 1000 KVA transformer is one of two on a 2000 KVA circuit in APED's main switch-gear, feeding from the main 12,000 volt secondary of a 60 KV to 12 KV transformer in Pacific Gas and Electric Company's substation. Each test loop is supplied with single phase power from a 37.5 KVA, 1 phase 480/120-240 volt

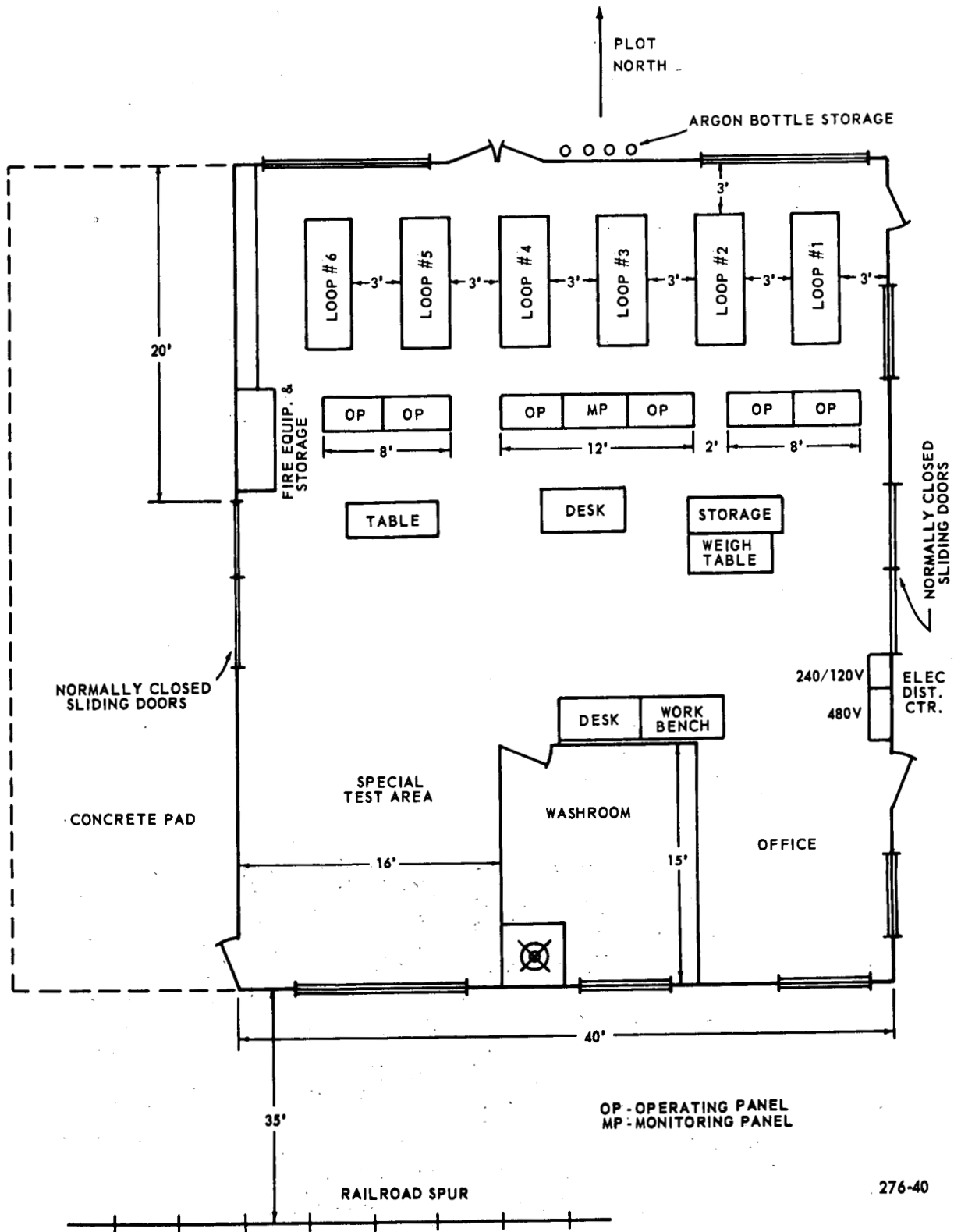
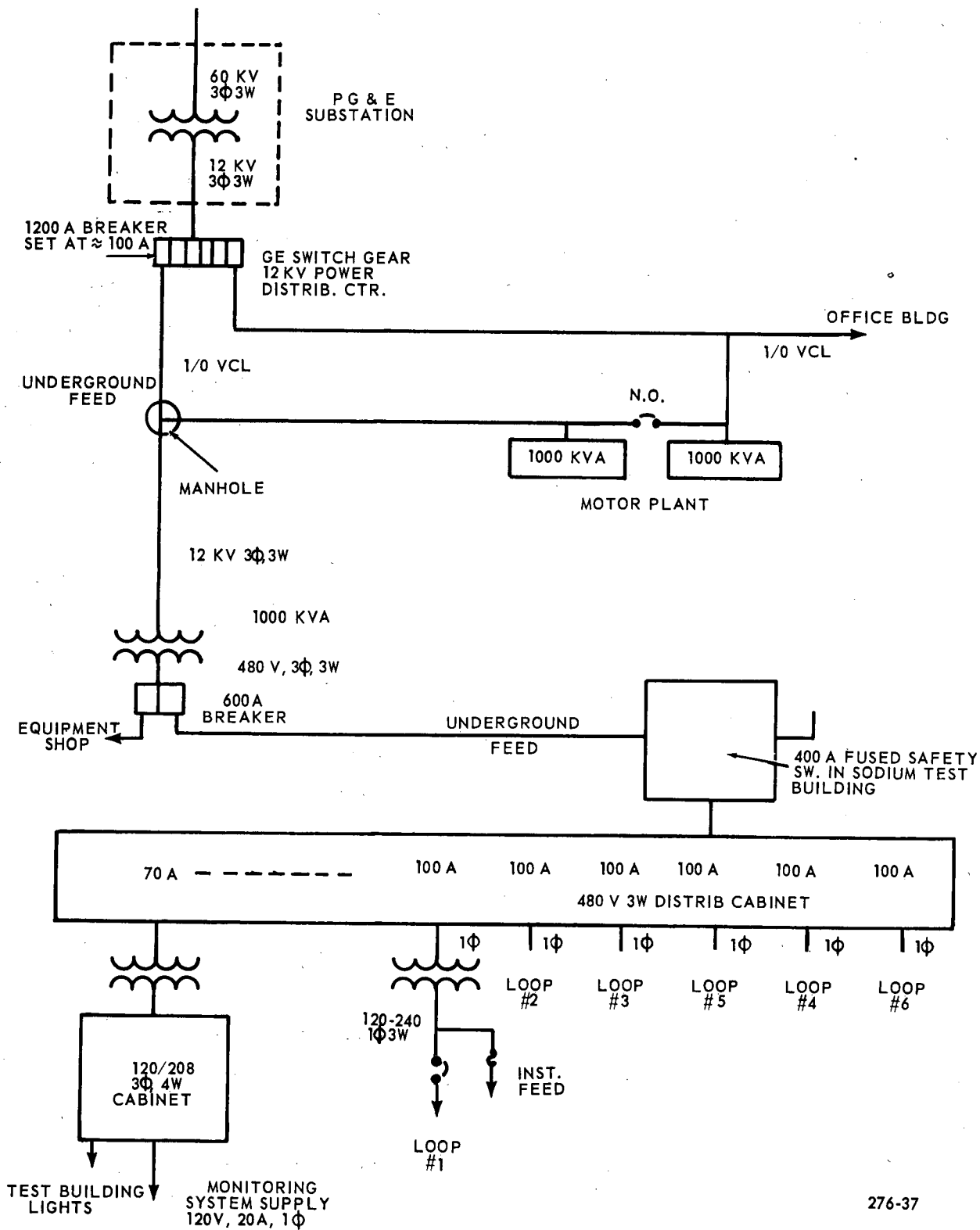


FIGURE III-14 MASS TRANSFER LOOP ARRANGEMENT



276-37

FIGURE III-15 POWER SUPPLY SYSTEM

transformer which is fed from an individual 100 ampere breaker in the test building 480 volt distribution cabinet. These breakers are physically separated from others in the distribution cabinet and are marked to prevent inadvertent opening. They provide the primary overload protection for the individual loop power supply transformers. These transformers are physically located close to their individual loops.

In order to promote long heater life, the transformer low voltage taps on the secondary were connected to the circuit breaker cabinet (loop load distribution). Instead of 240/120 volts, the transformer provides 225/112 volts to the breakers. If additional heat is required in the future, these connections can be changed to higher voltage taps. A magnetic contactor is provided between the transformer and the breaker cabinet as the means for automatic disconnect to each loop. A fused-feed from the line side of each contactor is used for indicating instruments and control for the loop. Shutdown of a loop resulting from a momentary loss of power is guarded against by an adjustable time delay relay which will reclose the main contactor if power is restored within the present delay time (adjustable from 1 to 30-seconds and preset at 7-seconds). A loss of power exceeding 7-seconds duration to any loop will result in loop shutdown with this particular relay adjustment.

A separate common power supply, independent of the individual loop's power supply, is provided for all alarms and recorders to allow continuous monitoring of all loops. This will provide indication and record of shutdown conditions if they occur.

A power supply fault on any one loop will not affect the other operating loops or the monitoring instrumentation. Conversely, power supply failure of the monitoring system will not shut down any operating loop.

A one line diagram of the loop power distribution system is presented in Figure III-16. Each loop has an identical system. This figure also lists the rating of all the installed heater circuits. Additional wiring details are described on the Elementary Wiring Diagrams included in Appendix F.

### III E Control and Instrumentation

- E1 General
- E2 Alarm and Annunciation System
- E3 Temperature Control
- E4 Indicating Instruments
- E5 Operating and Monitoring Panels

#### E1 General

The control and instrumentation systems are designed for continuous reliable operation throughout the expected duration of each run of the Mass Transfer tests. Little or no operator attention is required except for initial attainment of normal operation conditions, minor subsequent adjustments, and data taking. Provisions are made for immediate notification of cognizant personnel at any time of day or night in the event of a loop shutdown so that prompt action can be taken to reactivate the loop.

Automatic control with manual override is provided for the power input to the heaters and cooling air for the coolers to hold loop  $\Delta T$  constant within  $\pm 15$  F and heater outlet temperature constant within  $\pm 5$  F. Sufficient range is provided in the automatic control to accommodate anticipated fluctuations in voltage (approximately  $\pm 5\%$ ) and ambient day to night temperature changes of 50 F. Manual adjustments provide a wide range of flexibility in establishing operating points within the limits specified in the contract. Control of sodium flow and level is strictly manual.

Continuous monitoring is provided for; 1) high and low temperature points in the main loop; 2) flow in the main loops; and 3) sodium level in the surge tank. Alarms (red light and horn) are given whenever system conditions depart from normal limits and a safety trip (with a secondary backup) is provided to trip off the power supply when the maximum temperature exceeds the control set point by a predetermined margin. Additional over-temperature protection is provided for the pump cell where a preset maximum temperature will trip the loop power supply. A tabulation of alarms and shutdown signals with their initial set points is presented in subsection E2.

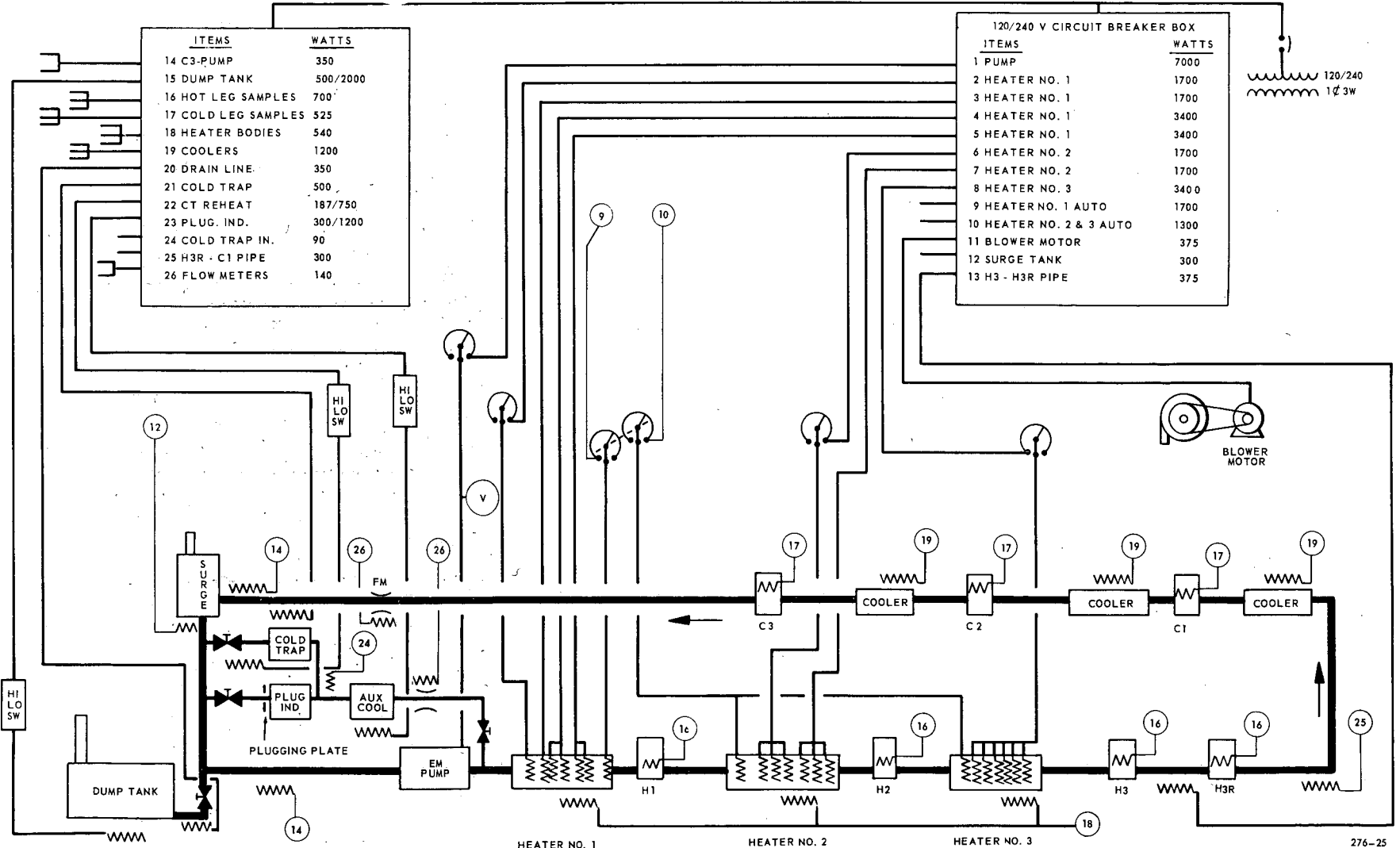


FIGURE III-16 LOOP POWER DISTRIBUTION



The complete control and instrumentation system for each test loop is described in Figure III-17. This diagram depicts the entire test loop with all its primary detectors and readout, control and annunciation equipment. Additional information is found on the two Elementary Wiring Diagrams in Appendix F.

The components of the system shown in Figure III-17 provide the following control and monitoring functions for the operation of the loops:

- a. Manual control of all control elements (sodium pump, heaters, coolers, and gas pressure) over their entire design operating range.
- b. Automatic control of main heater and cooler outlet temperature with control range limited to approximately 10%. This limited range assures that complete failure of the control circuitry will not rapidly affect the sodium system conditions.
- c. Multipoint (printout) recording of the highest and lowest sample holder temperatures for all loops.
- d. Multipoint (printout) recording of main sodium flow and level for all loops.
- e. Continuous recording of bypass flow and plugging plate or cold trap temperature on a two pen recorder for any one of the six loops by appropriate selector switch position.
- f. Indication of temperature at any of the thermocouples located throughout the piping system by appropriate selection.
- g. Continuous indication of sodium flow, surge tank level, pump voltage, control point temperatures, and argon pressure in the surge and dump tanks for each loop.
- h. Overtemperature shutdown, and backup for same, when the main heater outlet temperature exceeds the set point by a predetermined limit.
- i. Audible and visible alarms on high temperature, low temperature, high sodium surge tank level, low sodium surge tank level, low sodium flow (main loop), low argon supply pressure and reduced flow through the bypass flow meter (plugging indicator).

- j. Manual control of argon pressure in dump tank and surge tank.
- k. Manual control of heat input for preheating all piping and loop components.

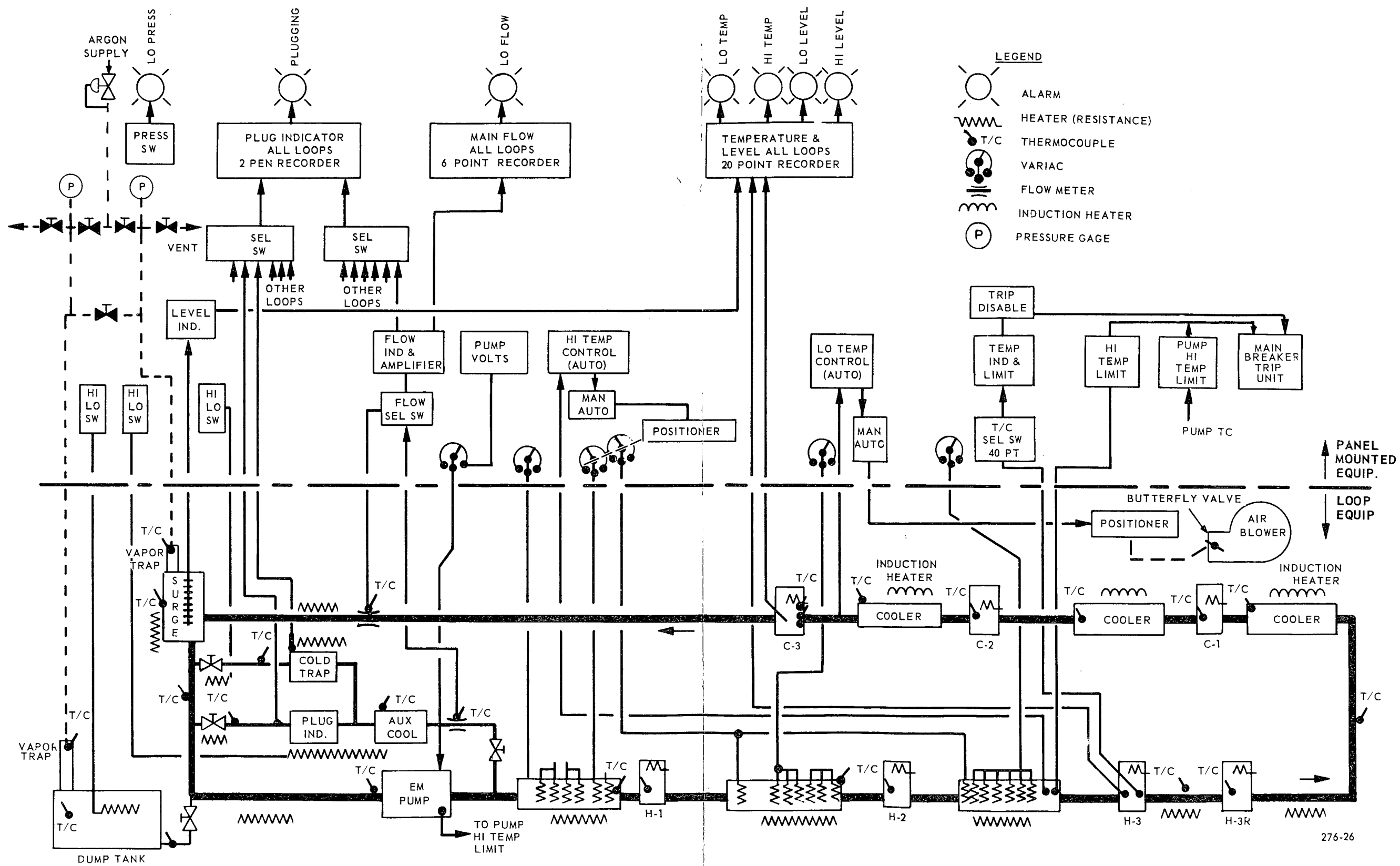
#### E2 Alarm and Shutdown Systems

A special alarm system is activated during off-shift hours while the loops are operating unattended. This system provides a visible and audible alarm in the guard station at the South Gate if any loop shuts down (trips out). Such an alarm will advise the guard to telephone the designated cognizant engineer. He will also be notified if the guards on routine plant patrol report any alarm from the continuous monitoring system. When any particular loop is secured the guard alarm system and the continuous monitoring system for that loop will not be activated.

Loop conditions and resulting action of the alarm and safety systems are itemized in Table III-2. Condition #7 shutdown signal, (high temperature at heater #3 outlet) provided by the indicating temperature pyrometer, can readily be changed to the high temperature signal from any one of the thermocouples connected to the selector switches. This flexibility in selection of the signal overrides the inconvenience of resetting the selector switches to the proper location every time loop temperatures are scanned.

#### E3 Temperature Control

Sodium temperature control is achieved by the operation of heaters and coolers installed in each loop. Refer to Figure III-18 for a diagram of the heaters and coolers temperature control arrangement. Control of heat input holds the  $\Delta T$  constant across the hot leg of the loop and control of the cooling effectively holds the temperature of the heater inlet temperature constant. Thermocouples at the outlet of cooler number 3 (lowest temperature) and heater number 3 (highest temperature) are used for monitoring loop temperature. These measurements are then used for the basis of controlling both the power to the heater and the air to the cooler. The heating elements are connected so that continuous control of heat input from zero to maximum is obtainable on each heater through appropriate use of manual and automatic controls.



276-26

FIGURE III-17 INSTRUMENTATION DIAGRAM

TABLE III-2

ALARMS AND SAFETIES FOR THE SODIUM MASS TRANSFER LOOPS

CONDITION	ACTION			INITIATION	SETTING
	<u>Red Light</u>	<u>Horn</u>	<u>Shut Down</u>		
1. Breaker Open	X		X	Breaker Auxiliary	Type "a" (open when breaker is open).
2. Main Loop Low Flow	X	X		Recorder Contact	80% of Normal
3. Hi Temp at H3 Sample Holder	X	X		Recorder Contact	Normal + 10 F
4. Low Temp at Cooler #3 Outlet	X	X		Recorder Contact	Normal -30 F
5. Low Surge Tank Level	X	X		Recorder Contact	Normal -4-inches
6. High Surge Tank Level	X	X		Recorder Contact	Normal + 4-inches
7. High Temp at Heater #3 Outlet	X		X	Pyrometer Relay	Normal + 50 F (5Cr Loop + 30 F)
8. High Temp at Heater #3 Outlet	X		X	Meter Relay	Normal + 50 F
9. Low Argon Supply Header Pressure	X	X		Pressure Switch	12 - 14 psig
10. Plug Indicator Low Flow	X	X		Recorder Contact	Normal -20%
11. Hi Temp at Pump Cell	X		X	Meter Relay	Normal + 100 F

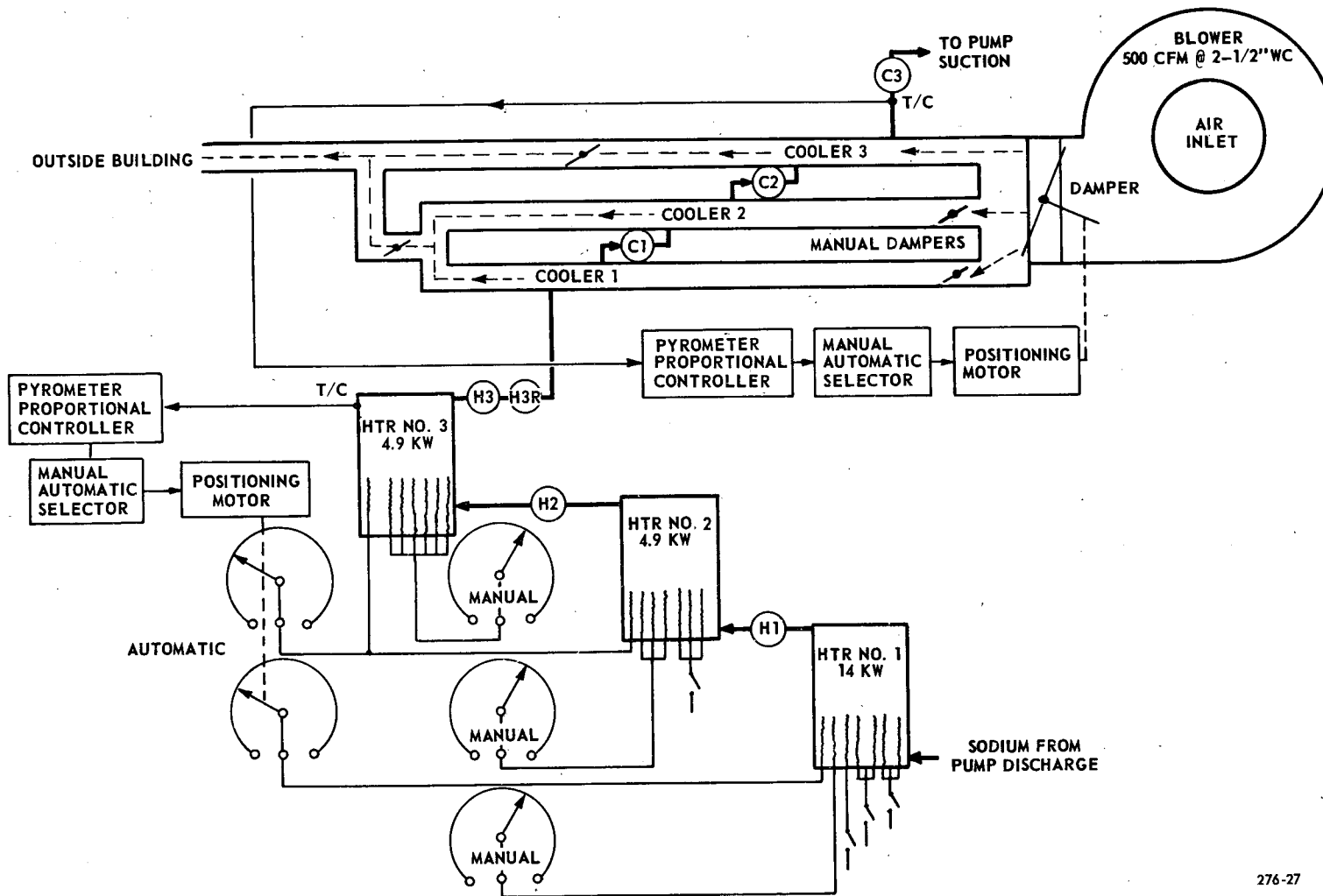


FIGURE III-18 TEMPERATURE CONTROL

In Heater #3 the voltage of all manually controlled units (6) is controlled with a single variac. This allows all heating units to operate at reduced voltage which should increase their life. As indicated the other two heaters have some units on off-on circuits and some connected to variacs.

The automatic heater control is a proportional-position pyrometer-controller operating an industrial control motor coupled to two "variacs" (one feeding a 2000 watt, 240 volt element in Heater #1 and the other feeding a 700 watt, 120 volt element in both Heater #2 and #3). The stroke of the control motor is limited to a value that will give approximately  $\pm 5\%$  control of the heat input to compensate for anticipated line voltage changes of  $2\frac{1}{2}\%$ . With one element out of seven being controlled at heat variation of approximately 70% on that element is required. At the upper end of the voltage range this requires approximately 43% reduction in voltage or a variac travel of 140 degrees. Limit switches on the control motor are set to avoid over travel.

The proportioning control (without reset action) is set to give full corrective action for a temperature error of approximately 10 F. An automatic-manual control switch gives manual override of the automatic control over its entire range.

The automatic air damper control utilizes the same type controller and control motor as the heater control described above. The motor drives a single 5-inch butterfly valve between the blower and the cooling air manifold. Appropriate division of air flow among the three coolers is accomplished by individual dampers which are adjusted manually and then clamped in place. Over the desired control range ( $\pm 10\%$ ) the division of air flow to each cooler is considered to be nearly constant. Manual override of the automatic system is available as in the heater control.

#### E4 Indicating Instruments

Equipment provided for visual indication of loop temperature, level and flow is described in the following paragraphs.

a. Temperature Indication

Chromel-alumel thermocouples are used as the temperature sensing elements throughout the installation. These thermocouples are asbestos-and-glass insulated with ceramic beads over the first few inches in the high temperature zones. The junction is formed by connecting the bare thermocouple leads to an adjustable voltage power supply and arcing the twisted end of the thermocouple in a grounded pool of mercury. Arc welding was used to attach thermocouples to the pipe walls and the sample holder thermocouples are installed by inserting the couple into a horizontal well in the center of the sample holder body.

Two thermocouples are installed in the hottest sample holder, H3, and two in the coldest sample holder, C3. One of the thermocouples in each holder provides the signal to the multipoint recorder and the other to the indicating pyrometer. This feature permits periodic calibration of the pyrometer for each loop by visual comparison with the multipoint recorder which is common to all six loops.

Thirty-two thermocouples from each loop are connected to two interconnected 20-point selector switches. The selected temperature is indicated on the central pyrometer of the appropriate operating panel. Table III-3 illustrates the position of the switch as related to a specific thermocouple on both the operating and the preheat-auxiliary selector switches. The operating switch must be in position 20 when observing temperature readings on any of the preheat-auxiliary thermocouples.

Because millivolt pyrometers are used for measuring the system temperatures, a large diameter thermocouple extension wire (size 14 AWG) was used to minimize voltage drop (lead length) errors. A voltage drop error of approximately 2 F exists in these loops because of the difference in lengths of the thermocouple wires.

TABLE III-3

SELECTOR SWITCH NUMBER IDENTIFICATION

<u>Operating Switch</u>	<u>TC No.</u>	<u>Preheat-Auxiliary Switch</u>
H3 Sample Holder *	1	Plugging Plate
C3 Sample Holder	2	Auxiliary Cooler
H1 Sample Holder	3	Cold Trap Outlet Valve
H2 Sample Holder	4	Plugging Indicator Valve
H3 Sample Holder	5	Cold Trap Body
H3R Sample Holder	6	Cold Trap Outlet
C1 Sample Holder	7	Pump Cell
C2 Sample Holder	8	Pump Inlet
Surge Tank	9	Pump Cell Loop
C3 to Pump	10	Heater No. 1
Drain Line	11	Heater No. 2
Dump Tank	12	H3 to H3R
Cooling Air Outlet	13	H3R to C1
Bypass Inlet Valve	14	Cooler No. 1
Dump Valve	15	Cooler No. 2
Spare	16	Cold Trap Inlet
Spare	17	Dump Tank Vapor Trap
Spare	18	Spare
Spare	19	Spare
Preheat-Auxiliary Selector Switch	20	Spare

\* Normal position



b. Flow Indication

Magnetic flowmeters are used in both the main and purification loops to detect sodium flow. In order to obtain a usable signal from the meters with Cr-Mo alloy steel cells, a special magnet design was used to provide an air gap flux of about 2000 gauss. These flow sensing elements are designed to provide an output of 0.5 millivolt per gpm so that with rated flow of 1 gpm in the main loop, the signal level will be 0.5 mv which is suitable for indication and direct input to the multipoint recorder. The purification loop flow will be approximately 0.1 gpm with a signal level of 0.05 mv. This signal is indicated by a galvanometer amplifier whose output is fed into the plugging indicator recorder through an appropriate voltage divider.

Each flowmeter is equipped with a thermocouple to measure the magnet temperature. This thermocouple is not permanently connected to the panel mounted selector switch due to anticipated infrequent use.

c. Level Indication

The level of sodium in the surge tank is indicated by a bottom mounted resistance probe made of the same material as the surge tank in which it is mounted. Refer to Figure III-19 for the measuring circuit diagram and arrangement.

Level indication is achieved by measuring the voltage drop of the current transmitted through the probe and sodium. The current is transmitted from the probe to a millivoltmeter located on each of the loop control panels and to the 20-point recorder located on the monitoring panel. The scale on the millivoltmeter was reversed since zero level provides maximum voltage (resistance).

Error caused by the cold junction compensation in the 20-point recorder produces an acceptable maximum error of 5% of true sodium

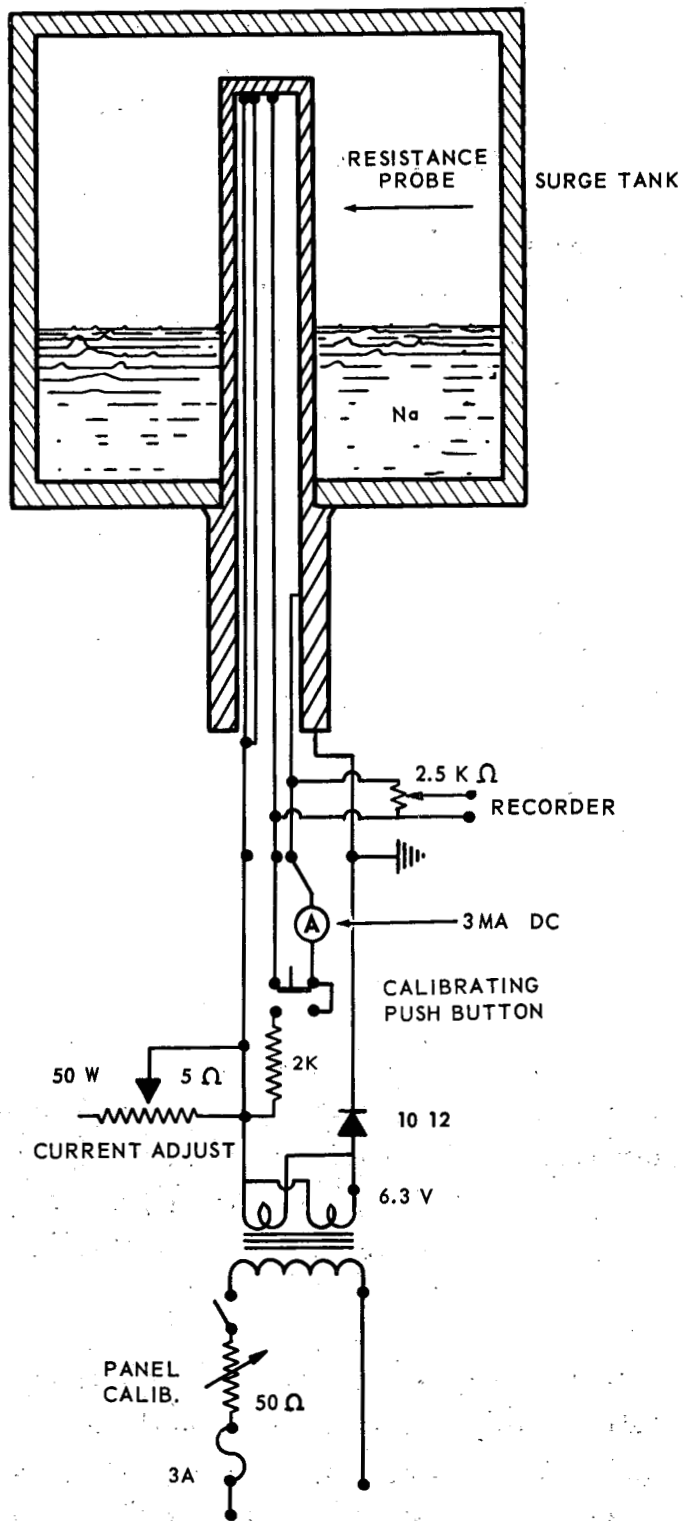


FIGURE III-19 LEVEL INDICATOR

276-31

level. Indicators for all six loops are calibrated to give the same percent level reading so that a common alarm point on the recorder operates for all six loops.

#### E5 Operating and Monitoring Panels

All of the indicating and recording instruments and the automatic and manual operators are located on the 7 instrument panels. A photograph of the installed panels appears on Figure III-20. As shown, there is an individual operating panel for each loop, plus the centrally located monitoring panel. The arrangement of these panels in the test building was shown on Figure III-14.

The arrangement of the instruments and operators mounted on the six identical operating panels is described on Figure III-21. As you will note the alarm lights, pressure gauges and inert gas control valves are in the upper area of each panel. The temperature control and indicating pyrometers are located at eye level; below this are the flow, level and pump voltage indicators. At hand height are the various selector and control switches. In the lower level are the variac knobs for controlling the pump and heater voltages. On the right hand side of the panel is a 34 circuit, column type, circuit breaker cabinet. These breakers are used as on-off switches as well as for overload protection. As indicated there are three high or low heat (voltage) selector switches for the heaters on the plugging indicator, dump tank and cold trap. The bypass switch at the left side of the panel is used for deactivating the over-temperature safety switch on the temperature indicator pyrometer during normal data logging of the thermocouple readings. This arrangement minimizes the possibility of an inadvertent trip of loop power.

The centrally located monitoring panel contains the three recorders which provide multi-point print out recording of the temperatures at the hottest and coldest sample holders, surge tank sodium level, and main flow for all 6 loops. The recorders are used to initiate visible (red lights) and audible alarm for each of the recorded conditions that change by more than a

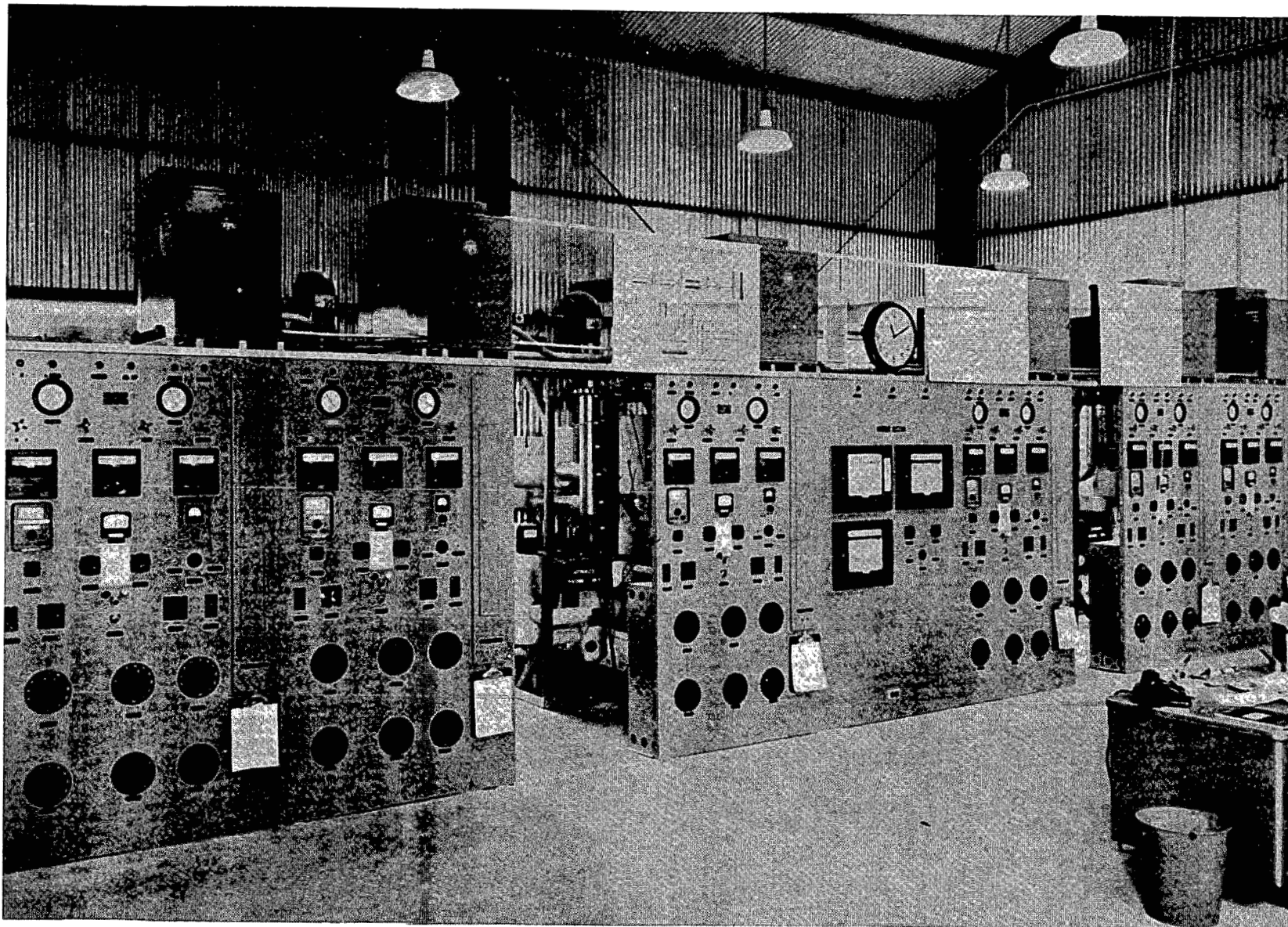
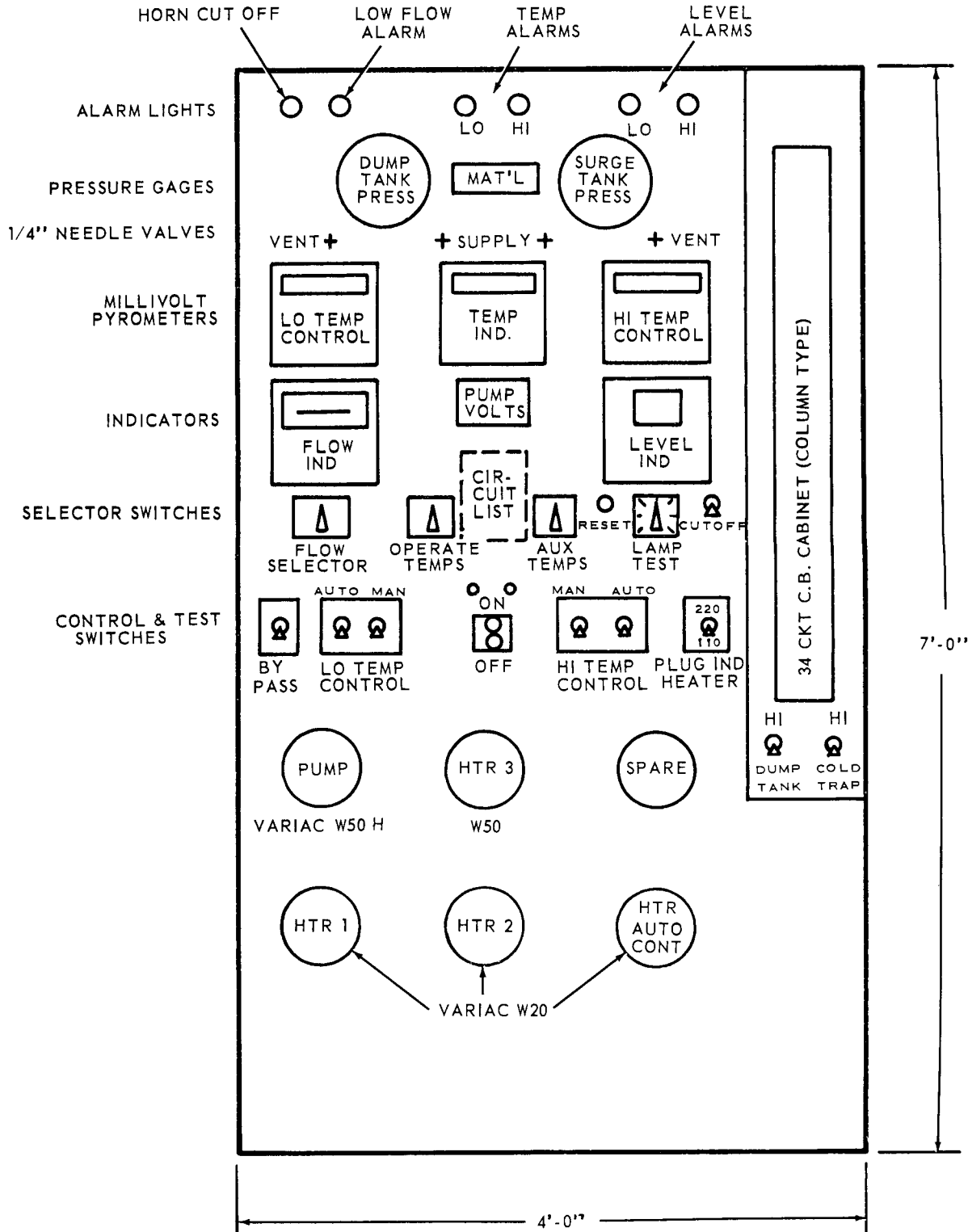


FIGURE III - 20 INSTRUMENT PANEL INSTALLATION



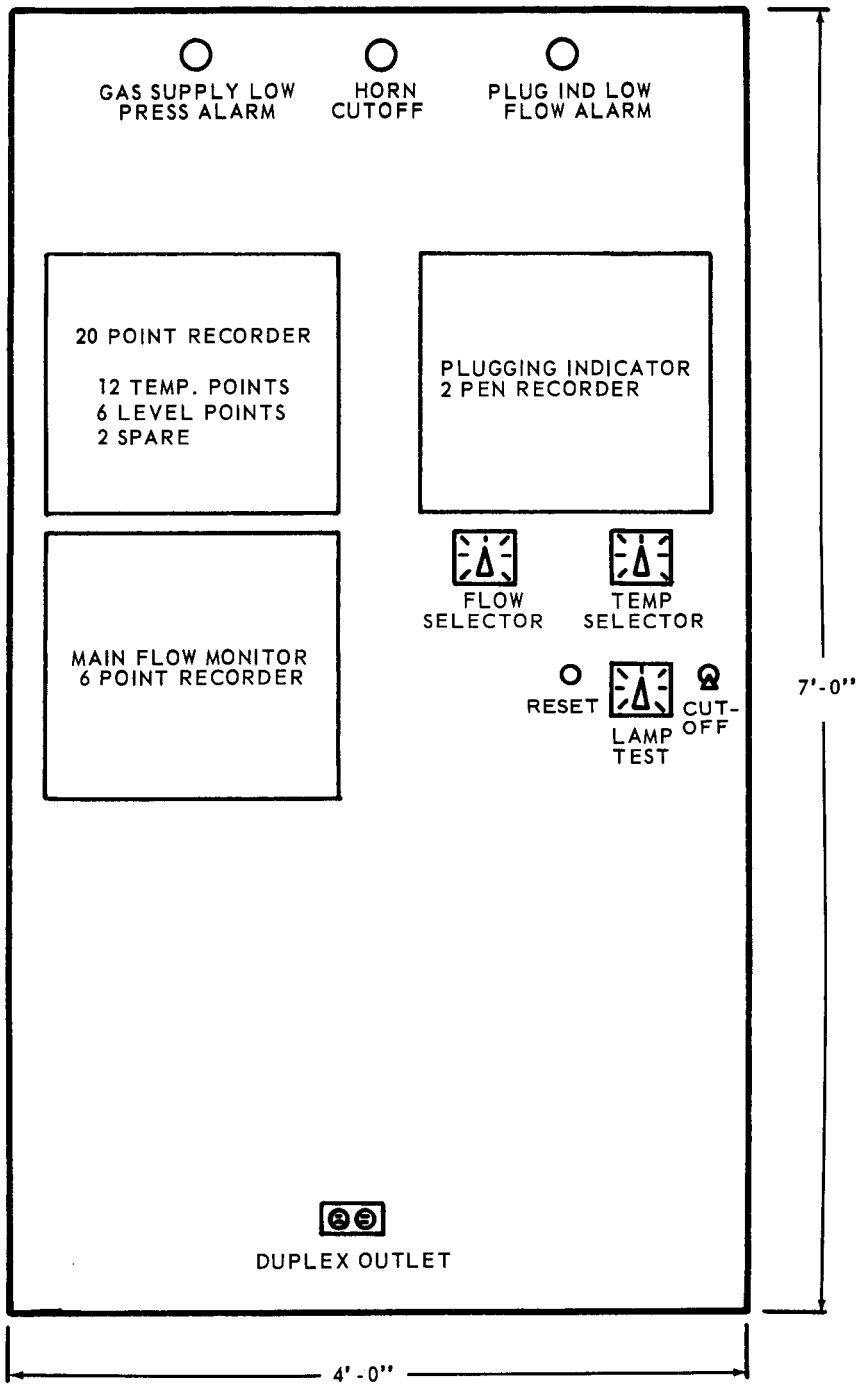
276-23

FIGURE III-21 OPERATING PANEL ARRANGEMENT

preset value. Each alarm seals in when initiated. The audible alarm can be silenced from the individual operating panel so that any further alarms pertaining to that particular panel will be visible only. In addition to the alarm light, a "horn cutoff" light is energized on the operating panel when the circuit is turned off. See Figure III-22.

The monitoring panel has a two pen continuous recorder to facilitate obtaining plugging indicator data. Plugging plate or cold trap temperatures and bypass flow from any of the six purification loops can be selected manually and are recorded when desired. A low flow alarm signal is provided by this recorder which gives a warning to terminate the plugging run when a preset loss in flow is obtained. The plugging indicator recorder can also be used to record cold trap temperature and flow for any of the six loops.

The monitoring panel also contains two alarm and horn cutoff lights as indicated on the arrangement figure. These lights warn the operator of low inert gas supply in the main header (less than 35 psi) and low flow on the two pen recorder. Similar to the operating panels, the audible alarm can be silenced by manual action. The "horn cutoff" light is activated if this action is taken.



276-24

FIGURE III-22 MONITORING PANEL ARRANGEMENT

#### IV. PROCEDURES FOR INITIAL OPERATION AND SAMPLE PREPARATION

- IV A Sodium Charging and Filling
- IV B Loop Start-up
- IV C Steel Sample Preparation
- IV D Sample Loading and Removal
- IV E Steel Sample Analysis
- IV F Sodium Sampling and Analysis

##### IV A Sodium Charging and Filling

Commercial grade sodium, supplied by the National Distillers and Chemical Corporation was used for initial operation. The sodium was furnished in five gallon containers, in cast solid form. An adapter for a 2-inch Marman Conoseal flange and a 65 micron stainless steel filter was attached to the bottom of the container before transferring the sodium. Each loop was charged with approximately five gallons of sodium, transferred to the dump tank under vacuum (subatmospheric pressure argon) at 250 to 300 F. Reduced pressure was created in the dump tank by attaching a vacuum pump line to the vapor trap gas nozzle. An argon gas bleed was maintained over the charging container during the transfer process. A blank flange was installed after the charging container was removed.

The loop was filled with sodium after the dump tank had been heated to 300 F and the main loop preheated to approximately 500 F by pressurizing the dump tank to 2 or 3 psi above the surge tank pressure. During the initial loop filling operation, the liquid level indicator in the surge tank was calibrated by checking with a portable (spark plug) level probe inserted through the vapor trap. Upon completion of the level check, the level was adjusted to approximately 4-inches in the surge tank (equivalent to 30% on the indicator) and then the drain valve was closed. Since the pump is operated at low voltage for preheating, flow is established as soon as there is sodium in the pump cell.

##### IV B Loop Start-Up

The sodium was heated to 600 F for initial circulation at approximately one gpm to promote wetting of the electromagnetic pump and the magnetic flowmeter. After determining the plugging temperature, the loop was cold



trapped at 300 F in order to establish a low oxide level during continued shakedown testing. A temporary reference flow meter and pressure gage was attached to the H1 and H2 sample holder flanges after the H1 outlet and H2 inlet passages were plugged with metal wedges. Flow and pressure data at isothermal loop temperatures of 600 F to 700 F were obtained for pump voltages ranging from 0 to 225 volts to accomplish the following:

- a) Measure pump performance
- b) Check system pressure drop
- c) Calibrate main loop chrome-moly flow meters with a standard (reference) stainless steel flow meter.

Corrosion samples were loaded into the cold leg sample holders (C1, C2, and C3) and pressure drop data obtained; after loading the H3 and H3R samples the pressure drop was again measured as a function of pump voltage and flow. Finally, the H1 and H2 samples were loaded after removal of the metal flow plugs and the temporary flowmeter-pressure gage assembly. Meter attachment details and results of these tests are reported in Section V.

#### IV C Steel Sample Preparation

The steel corrosion sample material was purchased to commercial specifications. Certified analysis of the material composition was reported in Subsection III-C. The 316 stainless steel samples were initially machined from 1/8-inch sheet that had been rolled to the sample thickness of 1/16-inch. The ferritic sample material was obtained by cutting, and flattening pieces of 1-inch or 2-inch pipe and then "hot" rolling to 1/16-inch thick and then machining to size. The 0.006-inches thick foil sample material was obtained by additional rolling of the 1/16-inch sheet and cutting to size.

After shop fabrication is completed the prepared samples are stored until needed. The samples are then stamped with a 4-digit identification number, cleaned, dried, weighed and stored in a desiccator until loaded into the loop. In the identification system, the first digit is the loop number (material combination). The second digit identifies sample location as follows:

<u>Second Digit No.</u>	<u>Position In Loop</u>	<u>Second Digit No.</u>	<u>Position In Loop</u>
1	H1	6	C1
2	H2	7	C2
3	$\frac{1}{2}$ -inch pipe at H3 outlet	8	1-inch pipe adjacent to C3
4	H3	9	C3
5	H3R		

The last two digits from 00 - 99 are used in sequence to chronologically identify samples in a given location.

The procedures used in cleaning and weighing samples are outlined below:

Cleaning and Weighing - Prior to Test

A. Low Alloy Steels

1. Vapor blast each sample.
2. Obtain desired physical measurements and macrophotographs.
3. Etch in 10%  $H_2SO_4$  for 30 seconds.
4. Rinse in distilled water.
5. Acetone rinse and dry. In subsequent operations use forceps or lint free gloves for handling.
6. Store within desiccator in 50 ml beakers.

B. Stainless Steel

1. Acetone rinse.
2. Passivate (standard procedure).
3. Obtain desired physical measurements and macrophotographs.
4. Degrease with detergent and water. (Do not use halogenated solvents).
5. Rinse in demineralized or distilled water.
6. Acetone rinse and dry. In subsequent operations use forceps or lint free gloves for handling.
7. Store within desiccator in 50 ml beakers.

C. All Samples After Cleaning

1. Remove from desiccator and bake in oven at 105 C for 30 minutes.
2. Return to desiccator and cool to room temperature.
3. Weight to nearest 0.1 mg and return to desiccator for transfer to test loop.

Cleaning and Weighing - After Test

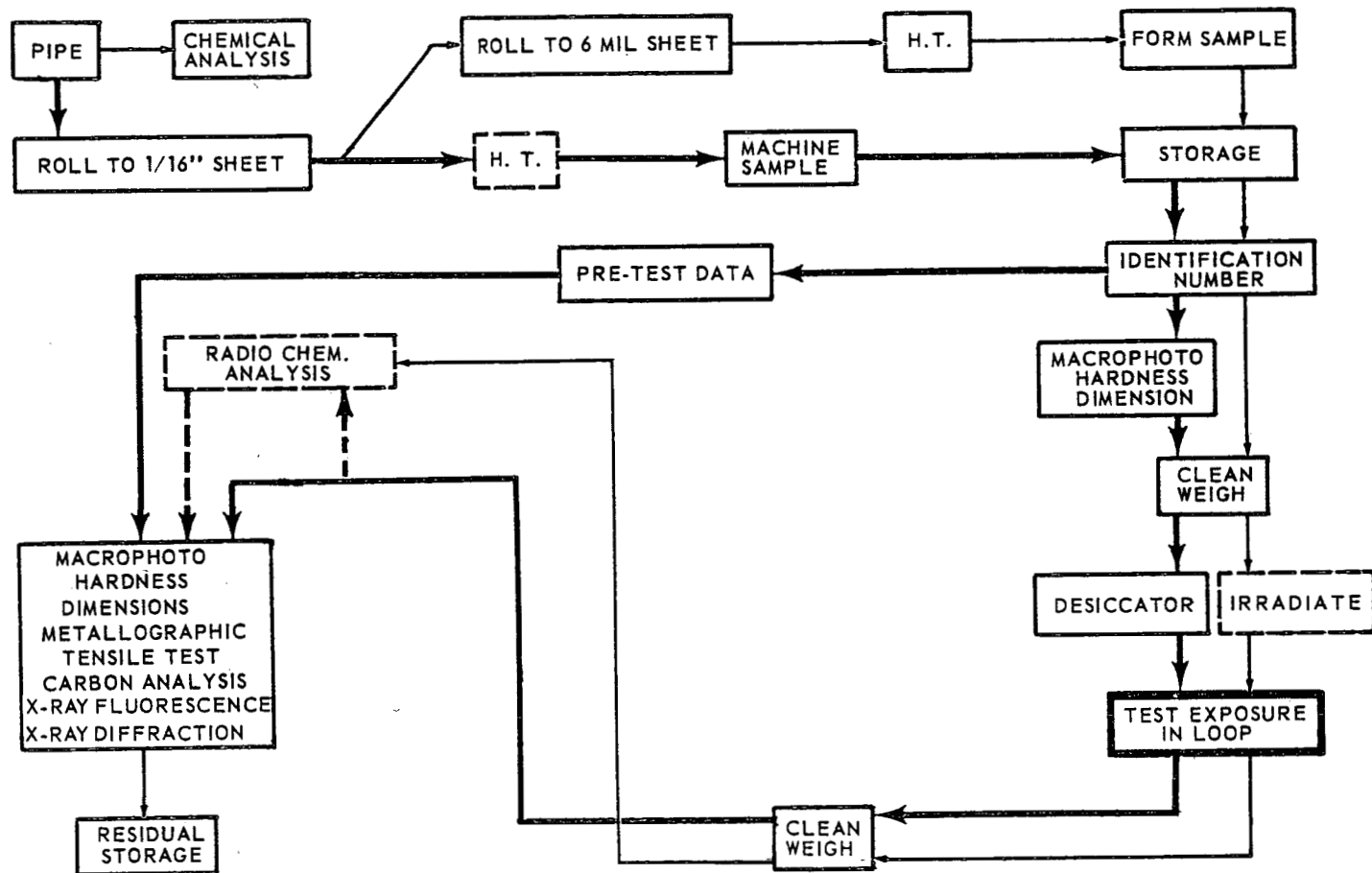
A. All Samples

1. Transfer exposed sample to laboratory in stoppered test tube under argon.
2. React with ethyl alcohol for 30 minutes at room temperature (If suspended corrosion products appear in liquid, collect on a filter, dry and weight products separately for each sample).
3. Rinse in demineralized or distilled water.
4. React with 130 F ethyl alcohol for several minutes, or if evidence of reaction appears, until reaction ceases.
5. Rinse in distilled water until pH of rinse equals pH of water supply.
6. Acetone rinse and dry.
7. Bake in oven at 105 C for 30 minutes.
8. Place in desiccator and cool to room temperature.
9. Weigh to nearest 0.1 mg and return to desiccator for storage until subsequent metallographic examination.

Graphically the proposed pre and post exposure events for each sample are outlined on Figure IV-1. Boxes that are dashed indicate that function is applied to only a select number of samples. Most samples will receive only a few of the metallographic examinations listed. Additional description of the proposed post exposure examination is found in Subsection IV E.

IV D Sample Loading and Removal

In order to minimize system contamination from the atmosphere it was originally planned to use a dry box over the sample holder during transfer of corrosion samples into or from the loop. A vacuum dry box (see Allied Engineering Drawing 5534-1, Rev. C and included in Appendix F) obtained for



73

276-32

FIGURE IV-1 SAMPLE FLOW SHEET

this purpose was designed for attachment to the 8-inch Marman flange which was connected to the body of the sample holder. In principle the dry box was to serve as a means of providing clean argon cover gas over the open loop when samples were either loaded or removed.

During shakedown operations it was found necessary to thoroughly clean the sealing surfaces of the two-inch Marman Conoseal assemblies on the sample holders before remaking the joint. The alcohol and water used in the flange cleaning procedure obviously contaminated the cover gas atmosphere. In addition, because the sample transfer operations and flange uncoupling and clamping could be considerably simplified and expedited with no dry box, subsequent access to the samples was accomplished without using the dry box. The unit is being maintained for special tests.

To minimize sodium contamination during sample transfer operations without a dry box, the following steps were incorporated:

1. An argon gas purge is maintained over the sample holder at all times when the top was uncovered.
2. While cleaning the Marman Conoseal joint, a temporary, close fitting inner cap is positioned inside the Conoseal assembly below the level of the gasket seating surface.

A typical cycle for loading and removing corrosion samples includes the following sequence of operations:

1. Drain sodium into dump tank.
2. Cool sample holders to 250 F - 300 F.
3. Remove Conoseal clamp.
4. Remove sample holder cap under a stream of argon, withdraw corrosion samples and replace cap.
5. Place exposed sample in a test tube under an argon atmosphere, stopper and send to laboratory for cleaning and weighing.
6. Remove sample holder cap under a stream of argon and cover with a temporary, inner cap.

7. Clean sodium film from gasket seating surface of female flange using cotton swabs saturated with propyl alcohol, then rinse with water and wipe dry with clean swab. During this interval the male flange cap is removed from the area of the loop, cleaned with water and dried and returned.
8. Install new gasket.
9. Remove temporary inner cap under a stream of argon, then load new corrosion samples in empty passages and cover with cleaned male flange cover.
10. Attach conoseal clamp in accordance with vendor instructions.
11. Reheat sample holders and loop to about 500 F, then fill with 450 F sodium from the dump tank and re-establish normal test conditions.

These procedures were modified somewhat when the radioactive tracer foil was used in run 1-1. Prior to loading the foil, the H3R sample holder was surrounded with lead brick shielding 2-inches thick, which was adequate to reduce dose rates to less than 2 mr/hr in the surrounding operating area. 24-inch long tongs were used in transferring the foil between the shipping cask and the sample holder. Of the 4 samples normally used in H3R, the radioactive foil was the last sample loaded and the first removed. Other samples were handled in the normal fashion.

Three  $\frac{1}{2}$ - by  $\frac{1}{16}$ - by 2-inches metallic samples, prepared in a similar manner to the corrosion samples, were spaced, wired together and placed in the piping near the H3 ( $T_{\max}$ ) sample holder. Three additional samples were similarly placed near the C3 ( $T_{\min}$ ) holder. In order to remove these samples the piping near them will have to be cut. The potential availability of additional data justified their insertion.

#### IV E Steel Sample Analysis

The basic data for the mass transfer evaluations are supplied by removable steel samples which will be exposed to sodium under controlled conditions of time, temperature, temperature difference, velocity and oxide content. Each sample will be analyzed in a comprehensive manner in order to obtain

pertinent information for correlating effects of specified sodium environment conditions and materials. The previously shown sample flowsheet, Figure IV-1 and Appendix B itemize or describe most of the procedures used in the sample analysis.

Changes in weight of the steel samples after exposure to the sodium provides an important measure of the combined effect of controlled variables. The development of an appropriate statistical program to supply mass transfer correlations is based largely upon the analysis of weight change data.

Radiochemical analysis of steel samples is used to supplement the weight change measurements and metallographical data. Details of this analysis are provided in Appendix C. In each loop containing a radioactive foil, the foil serves as a source for transferring activated isotopes to loop components and to the remaining steel samples throughout the system. After exposure and removal from the loop each corrosion sample and the foil will be analyzed for activated Cr 51, Mn 54, Fe 59 and Co 60. Because some of the deposited activity may be removed in the cleaning operation, the alcohol and water washes used in cleaning the sample also will be analyzed for the same isotopes. After correcting for the activity contained in the sodium which also will be counted, the residual activity in the wash will be attributed to coming from the steel sample.

All specimens after test will be examined metallographically and compared with representative pre-test samples in order to evaluate changes attributed to the sodium exposure. Metallographic examination of a sample from each order (size) of steel used to fabricate the loops has been made and will be reported in future reports. All materials met specifications and the sample material is representative of the loop material.

#### IV F Sodium Sampling and Analysis

As part of the experimental program to control and/or monitor impurity levels, sodium samples for each loop will be analyzed (according to procedures in Appendix B) at periodic intervals corresponding to the following schedule:

1. Sodium, prior to loading in loop. These samples were obtained by boring out cylinders from the 5 gallon containers of cast sodium at room temperature.
2. Loop sodium samples, taken at 500 F from the dump tank, using the thief tube technique. Samples will be taken at the following periods:
  - a. At beginning of each test run.
  - b. Each time loop is dumped for removing corrosion samples during a run (700 and 1400 hours).
  - c. At end of test run (2800 hours).

Impurities which are analyzed by wet chemistry and/or emission spectrographic methods include those elements which are normally present in relatively high concentrations in commercial sodium (Ca and K)\* plus other elements normally found in the materials of construction (Fe, Cr, Ni, Mn, Mo). Lithium was also included in this list because of its tendency to attack stainless steel and decarburized ferritic steels.\*\*

Activated isotopes - Cr 51, Mn 54, Fe 59, and Co 60 - in sodium samples from loops containing radioactive foils will be analyzed to assist in providing material balances for mass transfer correlations.

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\* Liquid-Metals Handbook, Sodium-NaK Supplement TID 5277, P. 4, July, 1955.

\*\* Liquid-Metals Handbook, NAVEXOX, P-733 (Rev.) P. 106, June 1952.



V. PRE-OPERATIONAL TESTS

- V A Level Gage Calibration
- B Main Flow Meter Calibration
- V C Observed Pressure Drop Data

V A Level Gage Calibration

Level indication is accomplished by measuring the voltage drop across a resistance probe which extends through the bottom of the surge tank to a height of 12-inches. A panel mounted voltmeter, calibrated to % level, is used for level measurement.

Calibration data, using a portable spark plug probe, was obtained to check the assumption of linear voltage change with level. The spark plug probe consisted of a 12 volt source with one lead fed through an indicator light and the probe element while the second lead was grounded to the loop. For a specific measurement, the portable probe was inserted through the vapor trap in the top of the surge tank to a known distance. Then sodium was added to the loop until the electrical circuit was completed, as detected by the indicator light. Simultaneously the reading on the panel level indicator was recorded. A tabulation of the data obtained during the initial operation of Loop #1 are shown below:

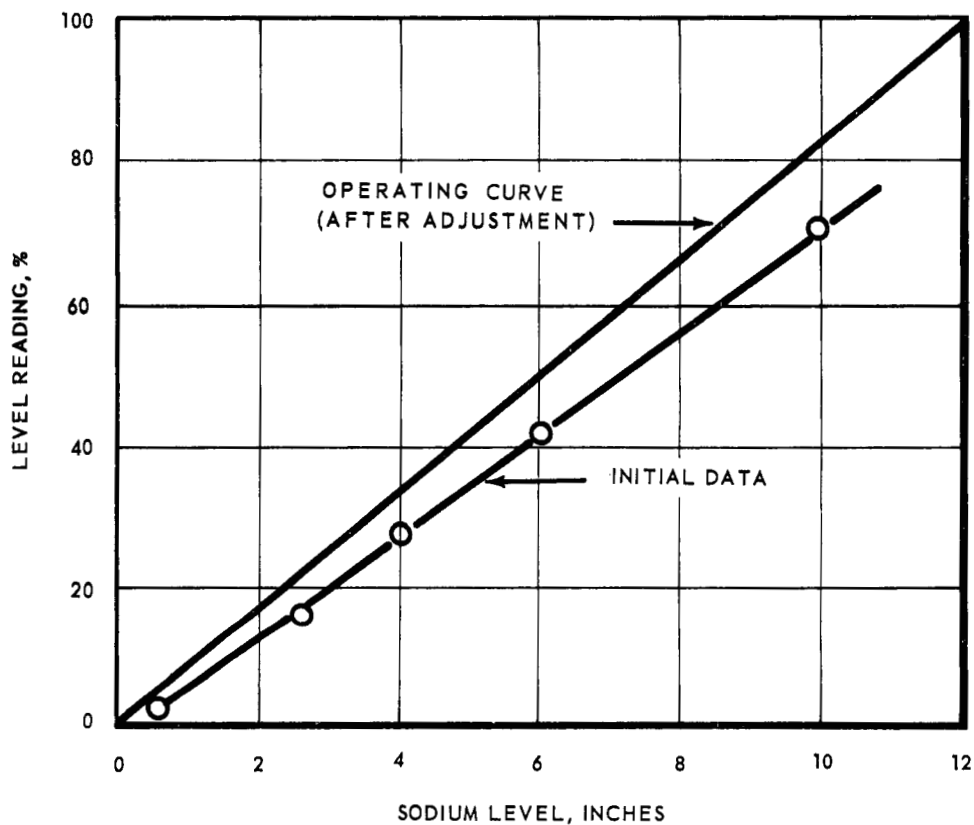
TABLE V-1

Surge Tank Temperature - 530 F

<u>Sodium level, inches</u>	<u>Indicator reading, %</u>
$\frac{1}{2}$	2
$2\frac{1}{2}$	15
4	26
6	42
$9\text{-}7/8$	71

A review of the data shows good agreement with the assumed linear variation of signal with sodium level.

Final calibration consisted of checking the end point conditions and adjusting the indicator to read 0% at zero level and 100% at 12-inches or maximum level. The full tank condition (12-inches) was simulated by shorting the resistance probe. The empty tank condition was obtained with the surge tank drained but at operating temperature. Subsequent checks with an adjustable probe in the region of 25% to 50% showed agreement within  $\frac{1}{2}$ -inch for the simultaneous measurements on the portable and fixed probes. See Figure V-1 for a plot of the operating and initial level gage calibration.



276-19

FIGURE V-1 SURGE TANK LEVEL INDICATOR CALIBRATION

V B Main Flowmeter Calibration

Sodium flow is detected by a permanent magnet flowmeter and indicated on a panel-mounted, Kintel electronic galvanometer. Each loop contains two flowmeters - one in the main flow stream in the minimum temperature region

and one in the bypass line preceding the plugging indicator and cold trap. A selector switch determines which meter is providing the signal to the galvanometer.

In order to satisfy loop design and operating test conditions the main flowmeter was to cover the range of 0.2 to 1.0 gpm and the bypass meter 0.1 to 0.2 gpm. Pipe materials used in flowmeter cell construction were as follows:

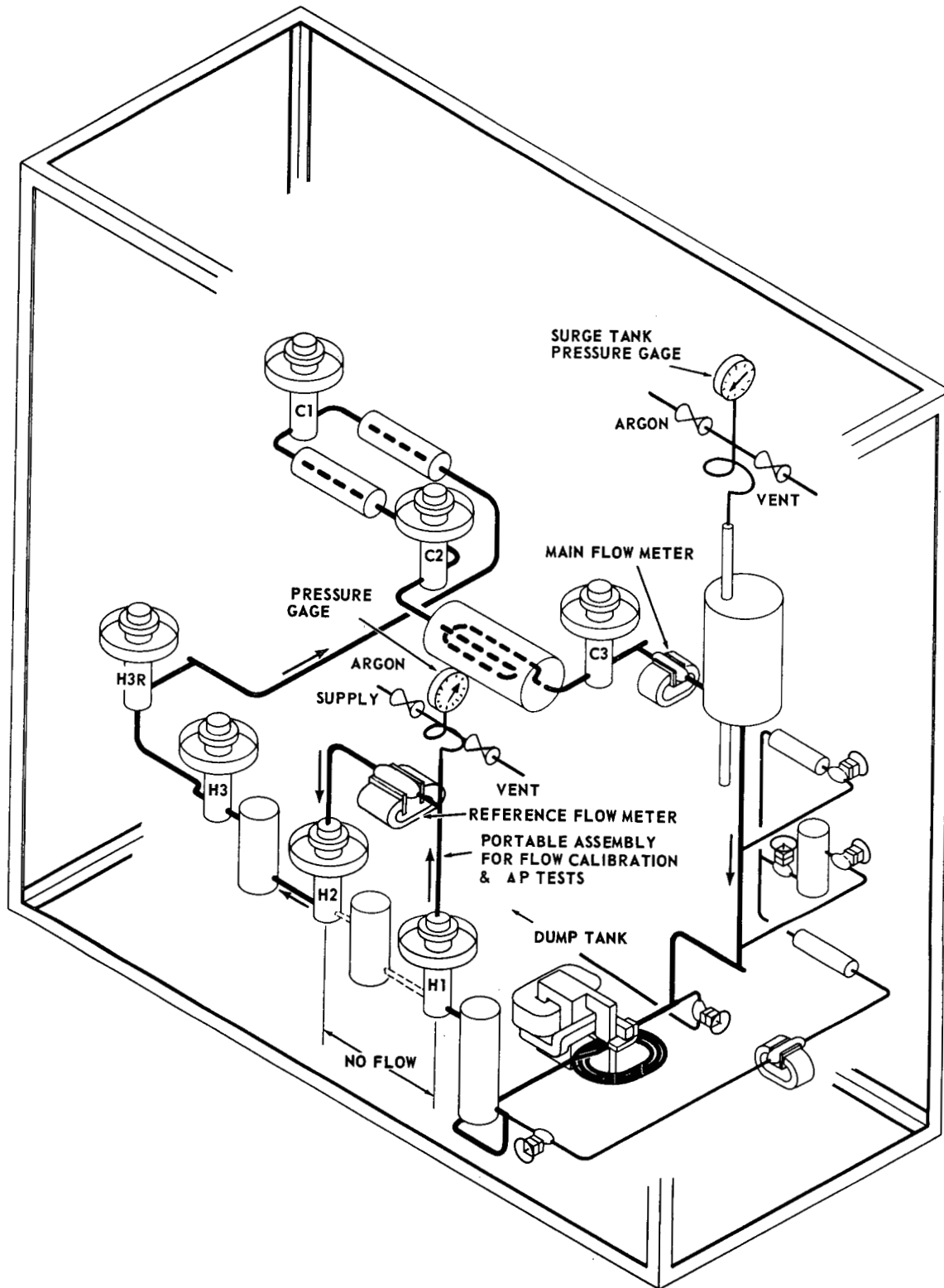
- Type 316 stainless steel - Loop #1
- $2\frac{1}{4}$ Cr-1Mo alloy steel - Loops #2, #3 and #5
- 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti alloy steel - Loops #4 and #6

Previous experience with magnetic flowmeters of austenitic stainless steel indicated excellent agreement between predicted and measured millivolt signal/gpm flow. With the use of ferritic flow pipes, however, the signal is considerably smaller and therefore more subject to error from thermal EMF effects. In order to provide more accurate flow measurements all ferritic, main flow meters were calibrated against a stainless steel flow meter.

For these calibration tests the reference flowmeter was installed in a temporary bypass line between the H1 and H2 sample holders as shown in Figure V-2. In order to insure that all of the flow stream was directed through the reference flowmeter, metal plugs were inserted in the H1 outlet passage and in the H2 inlet passages. In this manner the sodium was forced to flow in series through the ferritic and the stainless steel flow meters.

Separate indicating instruments were used to provide simultaneous readings for the two flow meter signals. Initial flow calibration runs were conducted before loop samples were added. The range of flows covered by the tests varied from zero to about 1.5 gpm, corresponding to applied pump voltages from zero to 225 volts in 25 volt increments. All test runs were at essentially isothermal loop conditions.

Flow tests at 500 F, 600 F and 750 F showed negligible temperature effect. Most of the subsequent calibration data was obtained with sodium in the region of 600 F to 700 F, approximating the normal temperature conditions of the flowmeter pipe during standard test runs.



276-18

FIGURE V-2 FLOW- $\Delta P$  TEST EQUIPMENT

Because of the tendency of the Kintel electronic galvanometer to drift, it was found necessary to zero the instrument immediately preceding each flow measurement. In order to compensate for reading and instrument errors, two sets of data were obtained over the pump voltage range, and the arithmetic average values used for each point. One set of readings was obtained during the period of increasing pump voltage and the second set during the decreasing voltage period.

Loop #2 was down, awaiting a new pump cell during the flow calibration tests for Loops #3 through #6. Because this loop (#2) was a bimetallic system already containing type 316 stainless steel and due to the small and variable flow signal experienced with the ferritic flowmeters, the two chrome moly pipes in Loop #2 flow meters were exchanged for stainless steel ones prior to the initiation of the standard test runs.

The flow calibration data for Loop #3 shown in Table V-2 and plotted in Figure V-3 are typical for data from the ferritic flow meters. A summary of the flow calibration factors used for each of the test loops is shown in Table V-3.

TABLE V-2

LOOP #3 FLOW DATA  
SODIUM TEMPERATURE - 600 F

<u>Pump Voltage</u>	<u>Main Flow Meter Signal, Mv</u>	<u>Reference Flow*</u>	
		<u>Mv.</u>	<u>gpm</u>
75	0.006	0.11	0.177
100	0.024	0.22	0.354
150	0.085	0.46	0.74
175	0.13	0.57	0.92
200	0.15	0.69	1.11
225	0.20	0.82	1.32

\* Calculated calibration factor for reference stainless flowmeter is 0.621 mv/gpm based upon measured magnetic flux and dimension data.

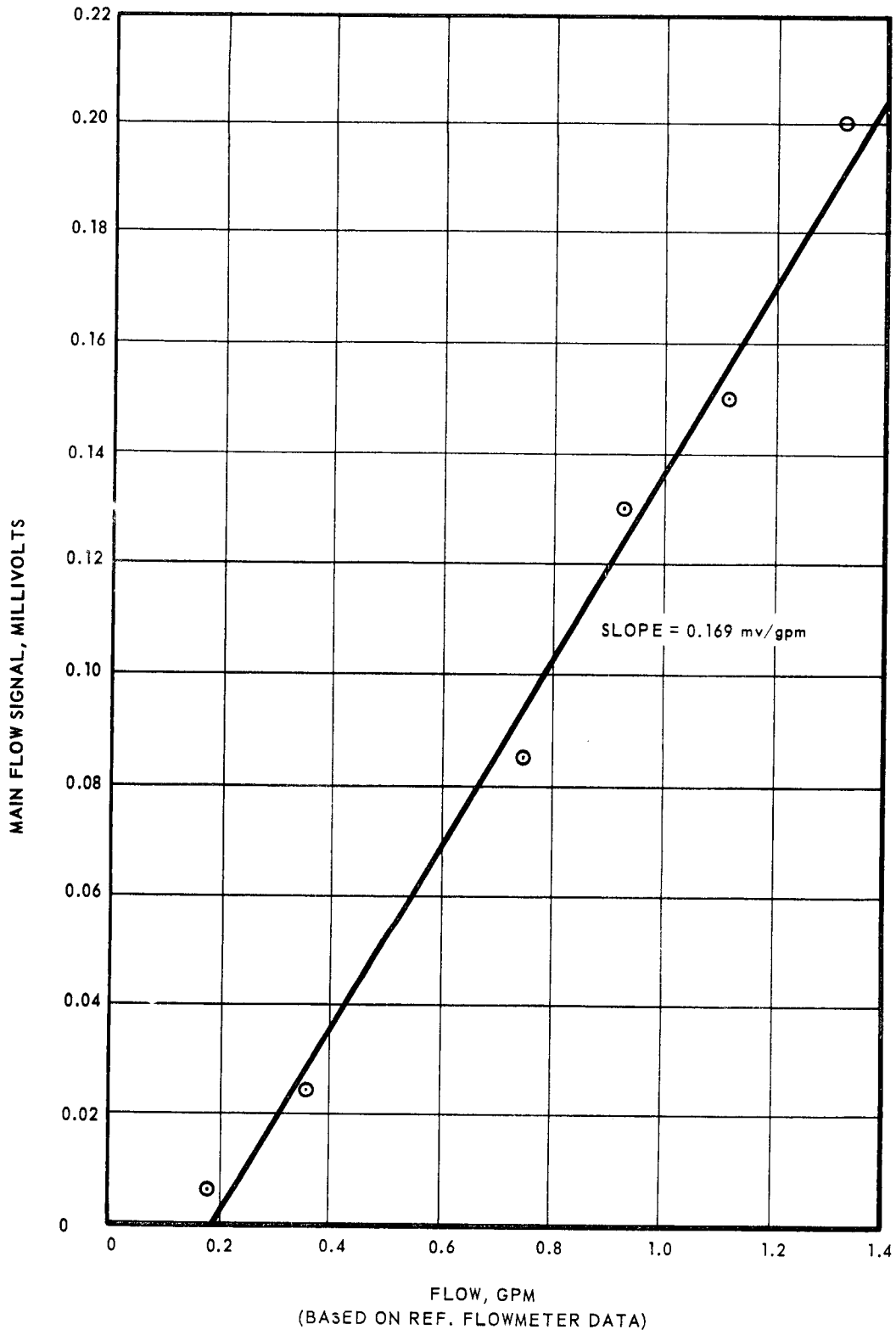


FIGURE V-3 LOOP #3 FLOW CALIBRATION CURVE

276-28

TABLE V-3

FLOW METER CALIBRATION DATA FOR MASS TRANSFER LOOPS

<u>Loop</u>	<u>Location</u>	<u>Material</u>	<u>Calibration Factor, mv/gpm</u>
1	Bypass*	316SS	0.618
	Main	316SS	0.641
2	Bypass	316SS	2.32
	Main	316SS	2.02
3	Bypass	316SS	2.27
	Main	2 $\frac{1}{4}$ Cr	0.169
4	Bypass	316SS	2.49
	Main	5Cr	0.279
5	Bypass	316SS	2.53
	Main	2 $\frac{1}{4}$ Cr	0.126
6	Bypass	316SS	2.49
	Main	5Cr	0.215
—	Reference F.M.	316SS	0.621

\* Bypass refers to the bypass purification portion of loop. Ferritic flowpipes originally in bypass were changed to 316 stainless steel prior to Run 2 and calibration factors are tabulated for the modified flowmeter. The stainless bypass data postdates preparation of the text but is included to report current conditions.

A review of the calibration curve for Loop #3 indicates that a negative flow signal on the main flow would be obtained for zero flow. Subsequent data verified this observation so that in normal test operation two flow readings are always logged for each point. One reading was taken at the operating pump voltage and the other at zero pump voltage. For normal test operation the flowmeter leads were oriented to give a positive signal. Therefore, to obtain a net flow signal, the value at zero pump voltage was added, if negative, and subtracted, if positive, to the measured reading at the operating pump voltage.

#### V C Observed Pressure Drop Data

Initial operation of loop #1, with all samples in place, indicated greater system pressure drop and/or lower capacity pump performance than originally anticipated. With 200 volts applied to the primary of the EM pump, loop flow was only 0.6 gpm instead of the anticipated 1.0 gpm. Pump voltage was limited to ~210 volts in order to avoid excessive heating at the nickel foil laminations of the pump cell electrodes.

As a result of the flow experience with loop #1, pressure drop data were obtained for all other loops prior to start-up of the planned test runs. Since the normal test loop and its associated instrumentation did not include provisions for measuring pump outlet pressure, a temporary line containing a Bourden tube pressure gage was installed at the H1 sample holder. This installation, shown on Figure V-2, was part of the temporary assembly for flowmeter calibration which was described in the preceding section.

Periodically during test, argon was admitted to the loop through the supply valve at the temporary pressure gage in order to maintain the interconnecting line open between H1 and the gage (free from sodium). With the system essentially isothermal in the region of 600 F to 800 F, data were accumulated at pump voltages ranging from 0 to 200 volts in 25 to 50 volt increments. Pressure readings at the surge tank and at the H1 sample holder locations were recorded for each voltage setting; at the same time loop flow plus C3 and H3 temperatures were noted. Data were obtained for three different conditions of sample loading in the flanged holders: (1) no samples, (2) all cold leg holders loaded with samples, and (3) all samples in position except the H1 and H2 sample holders.



To help compensate for reading and instrument errors, for each series of tests one set of readings were obtained for increasing pump voltage and another set for decreasing voltage, and the mean values were used in plotting the data. The  $\Delta P$  between the H1 sample holder (with no samples in the inlet passages) and the surge tank was taken as the system pressure drop or pump developed head. Over the flow range of primary interest, 0 to 1 gpm, the pressure drop between the surge tank and the pump plus the drop between the pump and H1 sample holder was calculated to be less than 1 psi and was partially compensated for by the lower static head ( $< 1$  psi) at the surge tank.

Pressure drop data listed in Table V-4 for loop #3 are shown as representative of these tests. Curves based upon data from all the loops (covering loops #2 through #6) are shown in Figure V-4. These results indicate that the system pressure drop is greater than was calculated for the initial design ( $< 70$  psi at 1 gpm). However, later pressure drop calculations for the final loop design (which included more samples than when the earlier calculations were made) is shown in Appendix E and indicates closer agreement with the experimental data.

TABLE V-4

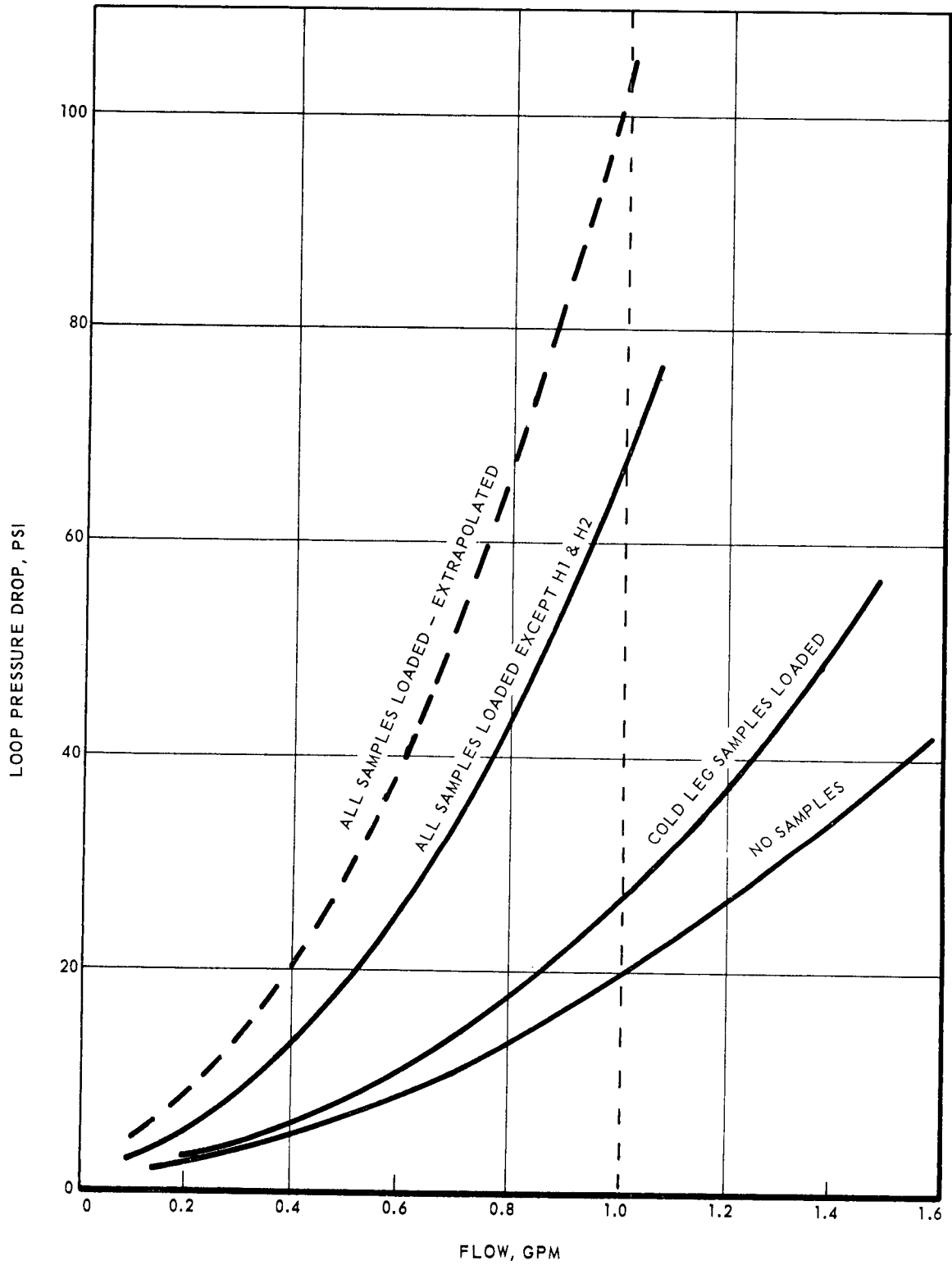
LOOP #3 PRESSURE DROP DATA

Sodium Temperature - 600 F

Samples Status - All loaded except H1 & H2 holders

<u>Pump Volts</u>	<u>Flow, gpm</u>	<u><math>\Delta P</math>, psi</u>
0	0	-
50	0	1.3
100	0.18	4.3
125	0.28	7.8
150	0.40	13.9
175	0.53	20.0
200	0.65	30.9
225	0.78	42.1

In order to facilitate attaining the design flow of 1.0 gpm for subsequent tests the pump cell design can be modified to provide increased developed head.



276-29

FIGURE V-4 LOOP PRESSURE DROP

VI. PROJECT STATUS (DECEMBER, 1960)

This report has described the design details of the Mass Transfer Test Loops and has indicated the results of the pre-operational tests and described the initial operating procedures. To the best of our knowledge these test loops have been designed, fabricated, checked out and now are ready to provide contracted test data. The only shortcoming of these loops appears to be involved with the loop pressure drop (or the pump specifications) and removal of samples at room temperature and with brief exposure to air instead of at operating conditions.

The initial test runs on each loop have been operated according to test specifications except for the maximum velocity (or flow rate). The pumps are operated at maximum developed head conditions and with corrosion samples in all sample holder slots. Experimental data indicates that the loops with stainless steel pump cells will operate at about 0.6 to 0.7 of rated (one gpm) flow. While the loops with ferritic pump cells will be limited to less than 0.5 gpm flow.

The flow meter calibrations have indicated somewhat lower than anticipated signal from the ferritic flow meters. This indicates that there will be difficulty due to thermal electric or other low voltage pickup in these systems.

The results of the first series of test runs will be reported in the forthcoming Topical Report: GEAP-3726-Sodium Mass Transfer: II-Screening Test Data and Analysis.

## APPENDICES

Each individual appendix contained herein and listed below is preceded by a colored divider page.

- A. Literature Survey on Mass Transfer in Liquid Metal Systems
- B. Analytical Procedures
- C. Steel Activation
- D. Steel Sample Removal Under Operating Conditions
- E. Pressure Drop Calculations
- F. Reference Drawings

## APPENDIX A

### LITERATURE SURVEY ON MASS TRANSFER IN LIQUID METAL SYSTEMS

A limited literature survey was undertaken in the fall of 1959, during the initial phase of the Mass Transfer Program, to provide an up-to-date bibliography for alkali metal corrosion and mass transfer. One of the objectives of this survey was to accumulate additional background information for use in defining the loop design, operating procedures and the experimental test program.

An abstract or list of pertinent information contained in each report is presented in this Appendix.

LITERATURE SURVEY ON MASS TRANSFER IN LIQUID METAL SYSTEMS

October 27, 1959

Prepared By T. J. Powell

The main purpose of this literature survey is to provide a current bibliography, included herein, on the subject of mass transfer and corrosion in liquid metal systems together with any information pertinent to our present study of mass transfer in molten sodium.

In this bibliography most of the references are followed by a list of the report highlights which were considered of primary interest to the Mass Transfer Program. The following points were given special consideration in the survey, and are summarized in the following text.

- a. The effect of continuous or non-continuous operation of the loop.
- b. The pro and con of a monometallic system.
- c. The use of an economizer. (Figure 8 loop)
- d. Special sample handling techniques.
- e. The effect of velocity.
- f. Diffusion bonding and removable parts.
- g. Radioactive tracer techniques.
- h. The results of tests on 316SS, 2 $\frac{1}{4}$ Cr-1Mo alloy steel, and 5Cr- $\frac{1}{2}$ Mo-Ti alloy steel.

In considering the following remarks, it must be kept in mind that all deductions are based on what is, at most, a limited literature survey. Certain trends are indicated but no definite conclusions can be made with respect to the state of the art without a further search of the literature, and, if possible, a determination of the extent of currently unpublished work. The following remarks correspond alphabetically to the points listed above.

- a. In the literature reviewed, no reference is made to the desirability of continuous operation of the loop, nor to the ill effects on the validity of experimental results obtained by a shutdown-startup method. However, it is implied that discontinuous operation is not very desirable in that no experimental procedures were found

which involved intentional shutdown before the end of the run on all samples in the loop. Literature (McKee (29)) reviewed after the original literature survey was issued, indicates that his loops were cooled to room temperature for removing selected corrosion samples prior to the end of a test run.

- b. Although the additional expense must be considered, the desirability of a monometallic system seems to be little questioned, especially at temperatures greater than 1000 F, Epstein, (12) Kelman, Wilkinson, and Yaggee(20) and Koeing and Brush.(22)
- c. The use of an economizer seems to have been almost universal in forced circulation thermal gradient loops. Generally no comment has been made in the literature regarding limitations associated with use of an economizer. However, Markert(28) describes an economizer which did not perform according to design in that it had a lower capacity for heat transfer than was predicted.

No mention is made of experimental data from the economizer region and/or its validity.

- d. The sample handling techniques of those tests reviewed were fairly standard. In general, all consisted of dumping and/or cooling the loop before removal of the sample. A somewhat original approach, proposed by Prados and Scott, (30) is based upon obtaining weight data for hot zone samples and cold zone deposits after the first plugging occurs in initial operation. Then, data would be obtained for progressively shorter periods of time in subsequent operations. This method however, could be prohibitively time consuming.
- e. No tests were found which endeavored to measure velocity effects at velocities as high as 30 ft/sec; however, "...flow rates are said to be important up to 30 ft/sec," Draycott and Rich(6).

Haag(15) indicated the transport rate to be invariant with flow velocity in the range 0.1 to 15 ft/sec for sodium temperatures up to 900 F.

- f. "There is one peculiarity of the sodium. ...pieces of similar metal in contact under sodium generally weld if the temperature is high enough. Self-welding has never been observed to occur below about 600 C," Epstein.(12) And again, "The scavenging action of sodium catalyzes diffusion welding," Koeing and Brush.(22) These observations would seem to indicate that movable parts could not be used in high temperature sodium systems if the system is to be monometallic.
- g. The use of tracers in the study of corrosion was foreseen by Stanley(33) as early as 1947. Today tracer techniques have been used together with weight measurement, in the study of mass transfer and preferential leading, Davis and Draycott,(4) Draycott and Rich(6) and Eding and Carr.(9) Tracers are particularly recommended in the range where the mass transfer cannot be measured by weighing, Markert.(28)

Using tracer techniques, Haag(15) indicates that buildup of activity is uniform with respect to system geometry.

- h. Data is presented with the outline abstracts indicating the rate of mass transfer of particular alloys under various test conditions.



## BIBLIOGRAPHY

1. Basham, Stang, and Simons, Corrosion Screening of Component Materials for NaK Heat Exchange Systems, Nuclear Engineering & Science Conference, XXIII-24, March 1958.
  - a. Test conditions:
    1. Tilting furnace
    2. 1200-1600 F
    3. 80 sec/cycle
  - b. Summary of results on some refractory metals, alloys, cements, and ceramics.
  - c. "High temperature valves must have sealing surfaces which show little or no tendency to self-weld when pressed together in a strongly reducing liquid metal environment."
2. Brasunas, A. de S., Liquid Metal Corrosion, Corrosion 9, 78, March 1953.

A good general background on the subject.
3. Crocker, et al, Design and Operation of a Sodium to Lithium to Air Heat Transfer System, APEX-327, 1957.
  - a. Work carried out during the period from September 1949 to June 1953.
  - b. 1000-1400 F system mainly of 316SS.
  - c. No corrosion data except that it was negligible.
4. Davis and Draycott, Proceedings of the Second International Conference on the Peaceful uses of Atomic Energy, Vol. 9, p.25, United Nations, Geneva, 1958.
  - a. "The value of static tests is extremely limited and their main purpose lies in eliminating materials which are completely unsuitable."
  - b. Test conditions:
    1. 300-650 C
    2. Up to 30 ft/sec
    3. Figure-of-eight loops of 18-8-1 stainless steel

- c. The design of a loop for radioactive mass transfer is essentially the same as that for other corrosion tests except that it is fitted so that samples of the liquid metal can be withdrawn at any time during the test. (Both activity and weight transfer are given in test results.)
- d. "...corrosion rates were insensitive to flow rates over the range of 2 to 5.5 ft/sec" at temperatures up to 550 C for the ferritic steels tested.
- e. 18-8-1 SS in cold trapped loops:

<u>T(°C)</u>	<u>ΔT(°C)</u>	<u>v(ft/sec.)</u>	<u>R(g/dm<sup>2</sup> mo)</u>
400 (752F)	100	~ 4	+0.0028
400	100	~ 8	+0.0003
400	100	10	-0.0032
600	300	55	+0.0022
600(1112)F	300	0.1	-0.0138

Cold trap temperature was 180 C except in the last case where it was 100 C. Duration of tests 350-500 hours. "No systematic variations with temperature or velocity were observed."

- f. 2½Cr-1Mo corrosion in cold trapped loops (18-8-1 loops):

<u>T(°C)</u>	<u>v(ft/sec.)</u>	<u>R(gm/dm<sup>2</sup> mo)</u>
550 (1022 F)	5.5	-0.00532
550	5.5	-0.03974

- g. Economizer used in loops.

5. De Van, J. H. and West, J. B., A Brief Review of Thermal Gradient Mass Transfer in Sodium and NaK Systems, ORNL 57-2-146, February, 1957.

Available corrosion information is assembled to help formulate an experimental program for studying the mechanisms of mass transfer in Inconel-NaK or Inconel-sodium systems.

Solution, chemical reaction, and nucleation processes are discussed. The effects of temperature, temperature gradient, purity and other pertinent operating parameters on corrosion are reviewed.

6. Draycott and Rich, Mass Transfer in Liquid Metal Containers: A Review of Progress in Initial Studies, IGR-TN/W-454.
  - a. Test conditions:
    1. 120-450 C
    2. 2.1-10.5 ft/sec
  - b. "A detailed examination of the effect of flow variations upon mass transfer has not been made as flow rates are said to be unimportant up to 30 ft/sec."
  - c. Tracer techniques are used in conjunction with weight measurements.
  
7. Dudek and Ferguson, Investigation of the Corrosion and Mass Transport Resistance of Structural Materials to Liquid Sodium, BW-3988, 1957.
  - a. Test conditions:
    1. Monometallic figure-of-eight loops (pump sections of different metal)
    2.  $T_{max} = 1150$  F,  $\Delta T = 350$  F
    3. 10 ft/sec
  - b. Materials:
    1. Croloy loops and specimens decarburized above 850 F.
    2. Croloy lot zone specimens generally lost weight while cold zone specimens gained weight.
  
8. Dudek and Ferguson, The Corrosion Testing of Various Materials in Sodium, BW-7020, 1957.
  - a. Test conditions:
    1. 1100 F, Cold trap at 400 F or less.
    2. 0-55 ft/sec. (Specimens rotated to attain velocity effect.)
  - b. Carbon steel, Croloy  $\frac{1}{2}$ ,  $1\frac{1}{4}$ , and  $2\frac{1}{4}$  decarburized almost completely. No other attack was noticed.
  - c. 316SS was carburized when any carbon was present but was not otherwise attacked regardless of structural condition.

d. Corrosion data:

<u>Materials</u>	<u>Specimen Condition</u>	<u>R, mg/dm<sup>2</sup>-mo</u>
Croloy 2 $\frac{1}{4}$	Rotating	-15, -13
Lukens Clad 316SS	Stationary	-12, -6, -27, -25, -25
Lukens Clad 316SS	Rotating	-12, -6

9. Eding and Carr, Mechanism of Surface Absorption in Homogenous Reactor Loops, AECU-4050, 1958.
- Mass transfer using tracer techniques
  - Rocking auto-clave apparatus, 250-300 C
  - Experimental techniques explained
10. Eldred, V. W., Interactions Between Solid and Liquid Metals and Alloys, AERE-XIR-1806, 1955.
- Extensive data on many metals and alloys in static tests with some introductory theory.
11. Epstein, L. F., Static and Dynamic Corrosion and Mass Transport in Liquid Metal Systems, KAPL-P-1714.
- A general paper devoted, mainly, to theory.
  - Dynamic corrosion rates of Armco iron, S.A.E. 1020 and 1010 in Na and NaK.
  - Diffusion rates in liquid metals (not much on Na).
12. Epstein, L. F., Proceedings of the First International Conference on the Peaceful Uses of Atomic Energy, Vol. 7, p.119, United Nations, New York, 1956.
- General theory.
  - "In many cases, if two dissimilar metals are immersed simultaneously in a liquid metal, the corrosion rates may be accelerated compared with the rates observed when only a single metal is in contact with the solvent."
  - Welding and diffusion bonding are considered.
  - Carburization and nitridification are considered.

- e. Two principal methods of liquid metal attack are indicated:
  - 1. By solution - of the major constituent of the alloy or by leaching out a minor alloying element or by solution of grain boundary constituents.
  - 2. By chemical reaction, either of the liquid metal or of some impurity which it contains (such as oxide).
  
- 13. Epstein and Weber, Corrosion and Other Factors in the Application of Sodium to the KAPL Reactor, KAPL-M-LFE-4, 1948.
  - a. A general discussion of sodium technology with reference to KAPL reactors.
  - b. "There is one peculiarity of sodium... Pieces of similar metal in contact under sodium generally weld if the temperature is high enough. Self welding has never been observed to occur below about 600 C."
  - c. "For Metals whose oxides are reduced by Na, corrosion is nearly independent of flow velocity."
  - d. In oxygen free systems (>0.01 w/o) no effect of flow velocity on corrosion is expected.
  - e. There is no effect of the oxide film on SS on the purity of the sodium.
  - f. The diffusion of oxygen from SS into sodium is negligible.
  
- 14. Fleishman and Konig, Selection of Materials, Design, and Specialized Fabrication Procedures for Sodium and NaK cooled Heat Transfer Systems, Nuclear Science & Engineering Conference, Preprint 49, December 1955.
  - a. General discussion per title.
  - b. Inspection requirements.
  
- 15. Haag, F. G., Activity Transport in Sodium Cooled Systems, KAPL-P-1707, 1957.
  - a. Test conditions:
    - 1. 600-900 F
    - 2. 0.1-15 ft/sec
    - 3. Monometallic Construction

b. Transfer rates for isotopes in 347 SS:

Isotope	R, mg/cm <sup>2</sup> -mo		Relative Transfer Rate (Co = 1)
	Stainless Steel	Individual Element	
Co - 60	0.0032	2.7x10 <sup>-6</sup>	1
Ta - 182	0.20	2.0x10 <sup>-4</sup>	75
Mn - 54	0.13	-	-
Fe - 59	0.012	8.3x10 <sup>-3</sup>	3,100
Cr - 51	0.0006	1.0x10 <sup>-4</sup>	40

c. Migration inhibitors (Ba is best to date).

d. "The following assumptions are justified by data:"

1. Rate of transport is invariant with flow velocity.
2. Rate of transport is invariant with disposition area.
3. Buildup of activity is uniform with respect to system geometry.

16. Hayes and Shepard, Corrosion and Decarburization of Ferric Chromium-Molybdenum Steels in Sodium Coolant Systems, NAA-SR-2973, 1958.

a. Test equipment:

1. High-velocity isothermal pump loop: 1025 F, 13.5 ft/sec, Na<sub>2</sub>O at 36 ppm.
2. Low-velocity anisothermal pump loop: 550-1050 F, 0.13 ft/sec, Na<sub>2</sub>O at 10 ppm.

b. All of steels tested showed low corrosion rates.

c. Decarburization encountered in low-chromium samples.

d. Steels with 5% Cr or more were carburized.

17. Hoffman and Manly, Corrosion Resistance of Metals and Alloys to Sodium and Lithium, Advances in Chemistry Series #19, Am. Chem. So., 1957.

a. General discussion of corrosion.

b. Several example loops shown.

18. Holman, W. R., Mass Transfer by High Temperature Liquid Sodium, AECU-4071 and 4072.

a. Test conditions:

1. Thermal convection loops
2. 1500 F maximum, 1330 F minimum
3. Material: TP 316 stainless steel

4. Calculated average velocity:  $<3$  ft/min
  5.  $\sim 50$  ppm  $\text{Na}_2\text{O}$  by diffusion cold trap
  - b. "Mass transfer rates are not necessarily the same for both solution and deposition, but, --- they appear to be of the same order of magnitude."
  - c. Corrosion samples located in chain fashion throughout circulating loop.
  - d. Corrosion rates ranged from  $+0.76$  to  $-0.64$   $\text{mg}/\text{cm}^2\text{-mo.}$
  - e. Samples gained weight at colder end of hot leg and lost weight at higher temperature region. Zero weight change noted near center of hot leg.
19. Jackson, C. B., Editor. Liquid Metals Handbook, Sodium-NaK Supplement, Third Edition, U.S. Government Printing Office, 1955.
- a. Techniques and facilities for radioactive material investigations are described including:
    1. A figure-of-eight sodium test loop, at 890 F  $T_{\text{max}}$ ,  $\Delta T = 250$  F, flow = 0.6 gpm, oxygen = 35 ppm, with an irradiated stainless steel source specimen.
    2. An in-pile sodium test loop using a non-radioactive NaK circuit for cooling and an economizer to minimize heat load, operated at 900 F  $T_{\text{max}}$  and  $2\frac{1}{2}$  gpm flow.
  - b. The effects of oxygen, temperature,  $\Delta T$ , velocity, time and selected inhibitors on radioactive material transfer are discussed. Most of the results are based upon a maximum temperature of 1000 F. However, thermal convection capsules at 1300 F and 700 F  $\Delta T$  showed transport rates  $\sim 300$  times those obtained with the in-pile loop (referred to above).
20. Kelman, Wilkinson, and Yaggee, Resistance of Materials to Attack by Liquid Metals, ANL-4417, 1950.
- a. General background and testing methods.
  - b. Extensive consideration of work on Na and NaK corrosion prior to 1950. Less on a large number of other liquid metals.

- c. "...because of this mass transfer of copper (and to a less extent of iron) multimetallic systems containing these elements yield data that are often difficult to interpret."
- d. "..., when exposed to sodium, metals high in chromium, such as stainless steel and stellite, acquire oxide films which prevent intimate contact and thereby inhibit diffusion welding" if enough oxygen is present initially to form the film.

21. Keyes, J. J., Some Calculations of Diffusion Controlled Thermal Gradient Mass Transfer, ORNL 57-7-115, July, 1957.

Presents calculations on the amount of mass transfer to be expected in liquid alkali metal systems for two assumed diffusion mechanisms:

- a. Boundary layer assumed saturated and rate of transfer limited by rate of diffusion of solute into liquid.
- b. A simplified general expression for effects of variation in temperature and composition is given by:

$$W(\theta) = k_L S(t_H - t_C)\theta$$

Where  $W(\theta)$  is mass transferred in time  $\theta$ ,  $k_L$  is mass transfer coefficient,  $S$  is solubility of solute in liquid Na or NaK,  $t_H$  and  $t_C$  are hot and cold zone temperatures, respectively.

- c. Rate of transfer limited by rate of diffusion of a component of a solid alloy element up to the solid surface.

A simplified general relationship is given by:

$$W(\theta) \approx 2 \rho_w (D_w \theta / \pi)^{\frac{1}{2}} x (t_H - t_C) / t_H$$

Where  $D_w$  is diffusivity of alloy element in the solid wall;  $x$ , the bulk composition;  $\rho_w$ , solid density.

22. Koeing and Brush, Selecting Materials for Liquid Sodium Systems, KAPL-P-1484, 1955.

- a. Tests:
  - 1. Dynamic up to 1000 F
  - b. Static up to 1650 F



- b. At temperatures above 1000 F, stainless steel is prohibitively attacked if the sodium oxide content is more than a few hundredths percent.
- c. Effects of cover gases.
- d. Non-monometallic systems of ordinary structural materials show no tendency to transfer at temperatures up to 1000 F.
- e. "The scavenging action of sodium catalyzes diffusion welding."

	<u>T(Deg. F)</u>	<u>R(mg/dm<sup>2</sup>-mo)</u>
2 $\frac{1}{4}$ Cr-1Mo	950	10
5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti	950	10

23. Lyashenko, et al, Proceedings of the Second International Conference on the Peaceful Uses of Atomic Energy, Vol. 7, p.2194, United Nations, Geneva, 1958.

- a. A USSR paper on the corrosion resistance of various alloys in sodium and lithium.
- b. Test conditions:
  1. 600 C (bypassed at 120-150 C)
  2. About 5 mm/sec
  3. Economizer used in loops
- c. Mechanical properties considered after exposure.

24. Lyon, R. N., Editor. Liquid Metals Handbook, Second Edition, U.S. Government Printing Office, 1954.

- a. Describes the design and construction of a typical test loops for dynamic corrosion studies. The test loops were of type 347SS, designed for 1 gpm flow at ~1000 F Tmax and included in economizer. Corrosion specimens were contained in flanged sections in the Tmax and Tmin regions.
- b. Types and mechanisms of liquid metal corrosion are discussed. Summary results, based principally upon static corrosion tests, for the corrosion resistance of various construction materials are included.

25. M.S.A., Final Report on Liquid Metal Technology, Project No. NRO31-364, March, 1955.
- a. General introduction to research and development work over 5 year contract period.
  - b. Presents summary of solubility studies of iron and nickel in sodium.  
$$\text{ppm iron} = 0.5 + 0.0122 t_F$$
$$\text{ppm nickel} = 1.3 + 0.0199 t_F$$
where  $t_F$  = temperature in  $^{\circ}\text{F}$
  - c. Abstracts of all liquid metal technology reports issued by Mine Safety Appliance Co. over contract period are included.
26. Manly, W. D., Fundamentals of Liquid Metal Corrosion, Corrosion, 12 46-52, July, 1956.
- a. Types of attack:
    1. Simple solution
    2. Alloying between liquid and solid metal
    3. Intergranular penetration
    4. Impurity reactions
    5. Temperature gradient mass transfer
    6. Concentration gradient mass transfer, or dissimilar metal mass transfer
  - b. Test variables:
    1. Temperature
    2. Temperature gradient
    3. Cyclic temperature fluctuation
    4. Surface area to volume ratio
    5. Purity of liquid metal
    6. Flow velocity
    7. Surface conditions
    8. Materials in contact with liquid metal
    9. Condition of container material
  - c. Discussion of the above with some examples.

27. Markert, W., The Corrosion Testing of Various Materials in Sodium, BW-3792, 1954.
- a. Work preliminary to BW-7020, Corrosion Testing of Various Materials in Sodium, by Dudek and Ferguson.
  - b. Test conditions: same as BW-7020.
  - c. The 300 series stainless steels generally had low positive weight changes.
  - d. Croloys had low weight changes: negative in those with less than 5 percent chromium and positive in those with more than 5 percent chromium.
28. Markert, W., Investigation of Corrosion and Mass Transfer With Sodium in a Figure-of-Eight Circuit, BW-3794, 1954.
- a. Test conditions:
    1.  $T_{max} = 1067 \text{ F}$ ,  $\Delta T = 617 \text{ F}$
    2.  $v = 30 \text{ ft/sec}$  at 3 gpm
  - b. Economizer did not fulfill design conditions.
  - c. Transfer rates were smaller than those obtained using spinning samples (BW-7020).
  - d. Detailed description of loop and its operation.
  - e. Mass transfer in some cases was so small that weight measuring techniques failed and tracer techniques are recommended.
29. McKee, John M., Sodium Corrosion As a Function of Time, Presented at 5th Nuclear Congress, April, 1959.
- a. Test conditions:
    1. Two thermal convection loops
    2. 1575 F maximum, 475 F system  $\Delta T$
    3. Material: TP 316 stainless steel
    4. Removable corrosion samples located in 1575 F region
    5. Calculated average sodium velocity: 2-inches/second
  - b. Samples removed at 1000 hour intervals throughout completion of run at 5000 hours. Loops were cooled to room temperature prior to removal "to minimize the possibilities for oxygen contamination of the sodium".

- c. Diffusion cold trapping with frozen sodium was used to maintain system oxide content. Oxygen content was obtained by chemical analysis from a sodium sample withdrawn at end of test run. Reported oxygen content:
    - 1. Loop A: 100, 130 ppm
    - 2. Loop B: 40, 80 ppm
  - d. Only reference found which reported sodium corrosion as a function of time corrosion rate data (at 1575 F and velocity of ~2-inches/sec) for:
    - 1. 500 hours: 1.5 mg/cm<sup>2</sup>-mo
    - 2. 4000 hours: 0.5 mg/cm<sup>2</sup>-mo
  - e. Ferritic boundary layer found on corrosion samples.
30. Prados and Scott, Proposal for an Experimental Study of Thermal Gradient Mass Transfer in a Thermal Convection Loop, ORNL-CF 58-2-124, 1958.
- a. General introduction with present state of art.
  - b. Theory with equation of transfer between wall and liquid.
  - c. Pyrex and Quartz loops suggested.
  - d. Criteria of experiment:
    - 1. Loop material must be inert to liquid metal.
    - 2. The rates of transfer must be high enough that significant quantities are transferred in a reasonable amount of time.
  - e. Mode of operation: the loop is operated until plugging occurs in the cold zone, then is shutdown and hot zone sample and cold zone deposits weighed. Process is repeated with shutdown in shorter and shorter times before plugging...a curve is obtained.
  - f. Data are included and method of analysis discussed.
31. Smith, A. A., The Interaction of Sodium and Ferrous Materials at High Temperatures, NP6649, August, 1955.
- Discusses decarburization of ferritic steel in sodium.

32. Smith, F. A., Proceedings of the Second International Conference on the Peaceful Uses of Atomic Energy, Vol. 7, p.2291, United Nations, Geneva, 1958.
- a. General discussion of sodium reactors.
  - b. 1. "Sodium is the best corrosive of liquid metals, in both static and dynamic systems."  
2. "...the mechanism of liquid metal corrosion..., simply depends on the solution rate of the solid metal in the liquid metal."
  - c. Fabrication practices are discussed.
33. Stanley, J. K., Tracer Isotopes in Metallurgy, Nucleonics, 1:70-7, October, 1947.

APPENDIX B

ANALYTICAL PROCEDURES

This document was submitted to the SAN Office of the USAEC prior to fabrication of the six test loops. It contains analytical procedures and other information pertinent to determining the mass transfer of steel exposed to liquid sodium. A description of initial test conditions and the experimental equipment is presented for background information. Some design details and the number of sample slots changed during fabrication, installation and start-up of the test equipment. These procedures are presented as reference material and project status as of February, 1960.

GENERAL ELECTRIC COMPANY  
ATOMIC POWER EQUIPMENT DEPARTMENT  
SAN JOSE, CALIFORNIA

ANALYTICAL PROCEDURES TO BE USED TO DETERMINE  
MASS TRANSFER RATES AND SODIUM PURITY

February 10, 1960

Prepared for the USAEC  
San Francisco Operations Office

under

Contract AT(04-3)-189; P.A. 15  
Mass Transfer Investigations

ANALYTICAL PROCEDURES TO BE USED TO DETERMINE

MASS TRANSFER RATES AND SODIUM PURITY

(Contract AT(04-3)-189; PA #15)

This memorandum contains a copy of analytical procedures and other information pertinent to determining the mass transfer and/or corrosion of selected materials exposed to liquid sodium and the impurities in the sodium. A brief description and operating conditions of the experimental equipment that will be used to provide the corrosion samples (coupons) and sodium samples is presented for background information. The equipment provided by this contract consists of six forced convection test loops fabricated from various structural materials: one each of type 316 stainless steel,  $2\frac{1}{4}$  chromium - 1 molybdenum steel, and 5 chromium -  $\frac{1}{2}$  molybdenum -  $\frac{1}{2}$  titanium steel monometallic loops, two bimetallic loops of type 316 in the hot leg,  $2\frac{1}{4}$  chromium - 1 molybdenum steel in the cold leg and one bimetallic loop of type 316 and 5 chromium -  $\frac{1}{2}$  molybdenum -  $\frac{1}{2}$  titanium steel.

The test loops have sample holders in the hot leg at the  $T_{max}$ ,  $T_{max}$ -100 F and  $T_{max}$ -200 F locations plus a special holder at  $T_{max}$  that can be biologically shielded for tests that require a radioactive source sample; and three holders in the cold leg at  $T_{max}$ -100 F,  $T_{max}$ -200 F and at  $T_{min}$  locations. Each sample holder has a 2-inch Conoseal flange for access to the removable samples and a concentric 10-inch flange for attachment of a portable inert gas (dry) box to minimize contamination of the loop and samples during sample removal, at about 450 F. An EM pump is used to circulate the sodium in the loop. Other loop components include: a surge tank, a dump tank, main and start-up electric heaters, air coolers, and system for heat removal from the coolers, sodium purification, electrical distribution, instrumentation and controls.

Initial operation of the loops will provide material screening tests to determine mass transfer and/or corrosion of materials under the following conditions: sodium velocity 30 ft/sec, maximum temperature 1200 F for all but the type 316 stainless steel loop where the upper temperature will be 1300 F, temperature differential will be 500 F and the sodium purity will



be 50 ppm of oxygen (450 F plugging indicator temperature). The conditions for the second bimetallic 316 - 2 $\frac{1}{4}$  loop will vary by changing one of these variables.

For initial operation there will be one sample of the same material as the sample holder in the -100 F and -200 F of maximum temperature locations and three samples in the hottest and in the coldest zones. The sample removal schedule is presented in the following table:

TEMP. ZONE LOCATION	HOT LEG SAMPLES (v = 30 Feet per Second)			COLD LEG SAMPLES (v = 10 Feet per Second)		
	1 Month	2 Months	4 Months	1 Month	2 Months	4 Months
T <sub>max</sub>	X	X	X	-	-	-
T <sub>max</sub> -100 F	-	-	X	-	-	X
T <sub>max</sub> -200 F	-	-	X	-	-	X
T <sub>max</sub> -500 F	-	-	-	X	X	X

This schedule incorporates minimum disturbance to the loop during operation. Additional low velocity samples may be placed in any of the temperature zones but will only be removed for analysis after the test is completed. At present, pressure drop or heater capacity limits the addition of more high velocity specimens.

The analytical procedures described in subsequent paragraphs are currently planned for use in determining the mass transfer and/or corrosion of materials exposed to sodium and the sodium purity. These procedures will serve as a guide in conducting the experimental program. However, as the program progresses and in the initial test data become available it is likely that some changes in the number and frequency of removing samples and some of the analytical procedures will be desirable for effective data compilation.

## NON-RADIOACTIVE METHODS AND PROCEDURES

### Weight Change Measurements

It is assumed that the principal data for evaluating mass transfer will be supplied by weight measurements of removable specimens withdrawn from the test loops at periodic intervals. All samples will be weighed to  $\pm 0.1$  mg before and after test to determine the change in weight.

From extrapolation and interpolation of hot leg corrosion data found in the literature, and shown on Figure B-1, a corrosion rate of  $10 \text{ mg/dm}^2\text{-mo}$  was selected as a reasonable value for use as a reference design condition in sizing the samples and establishing time intervals for initial test runs. On the basis of the above conditions and a minimum test interval of one month, the selected sample size (1/16-inch x 7/32-inch x 3-inch) will provide a sensitivity of less than  $10 \text{ mg/dm}^2\text{-mo}$  which is equivalent to 0.06 mils/yr of uniform surface change.

Multiple tabs (three) located at the maximum and minimum temperature regions of the loop will be withdrawn at varying time intervals to provide weight change data as a function of time. Single samples at intermediate temperature locations will be analyzed at the end of the test for comparison of temperature effects. If the structural material dissolves in the hot leg and deposits in the cold leg, at the same rate, then the weight change data for a single test run as a function of time may resemble the curves shown in Figure B-2. However, it is recognized that the actual data may be considerably different. If, for example, the mass transfer process is limited by solution rate, then the equilibrium concentration of solute materials in the sodium will correspond to saturation near the minimum system temperature. Under these conditions, since precipitation does not occur until the sodium temperature is at or below saturation, deposition will occur only in the vicinity of the minimum temperature region. Thus samples H<sub>1</sub>, H<sub>2</sub>, H<sub>3</sub>, C<sub>1</sub> and C<sub>2</sub> may all lose weight while C<sub>3</sub> (and the main cooler) may gain weight.

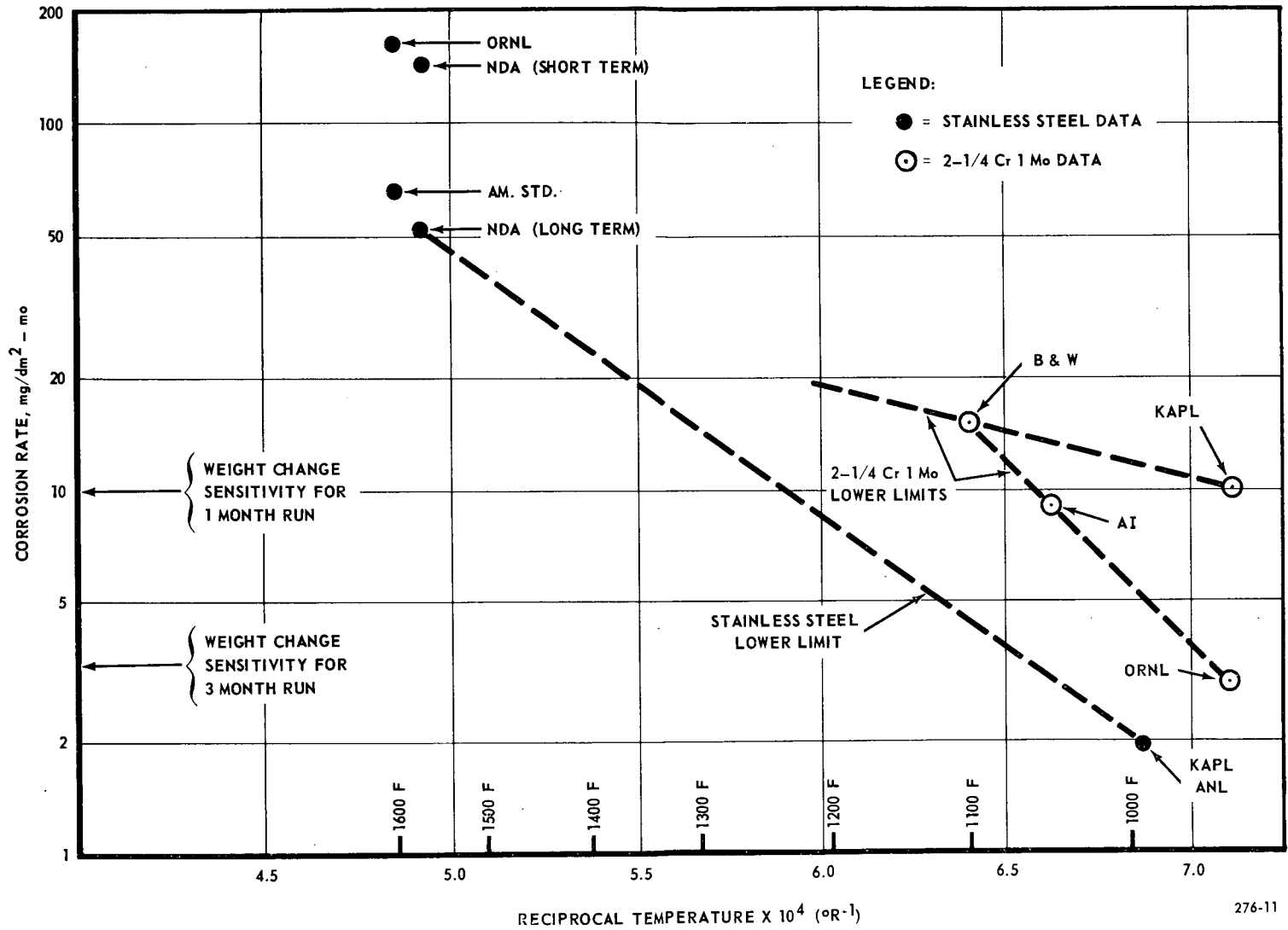


FIGURE B-1 SODIUM CORROSION DATA - HOT ZONE SPECIMENS

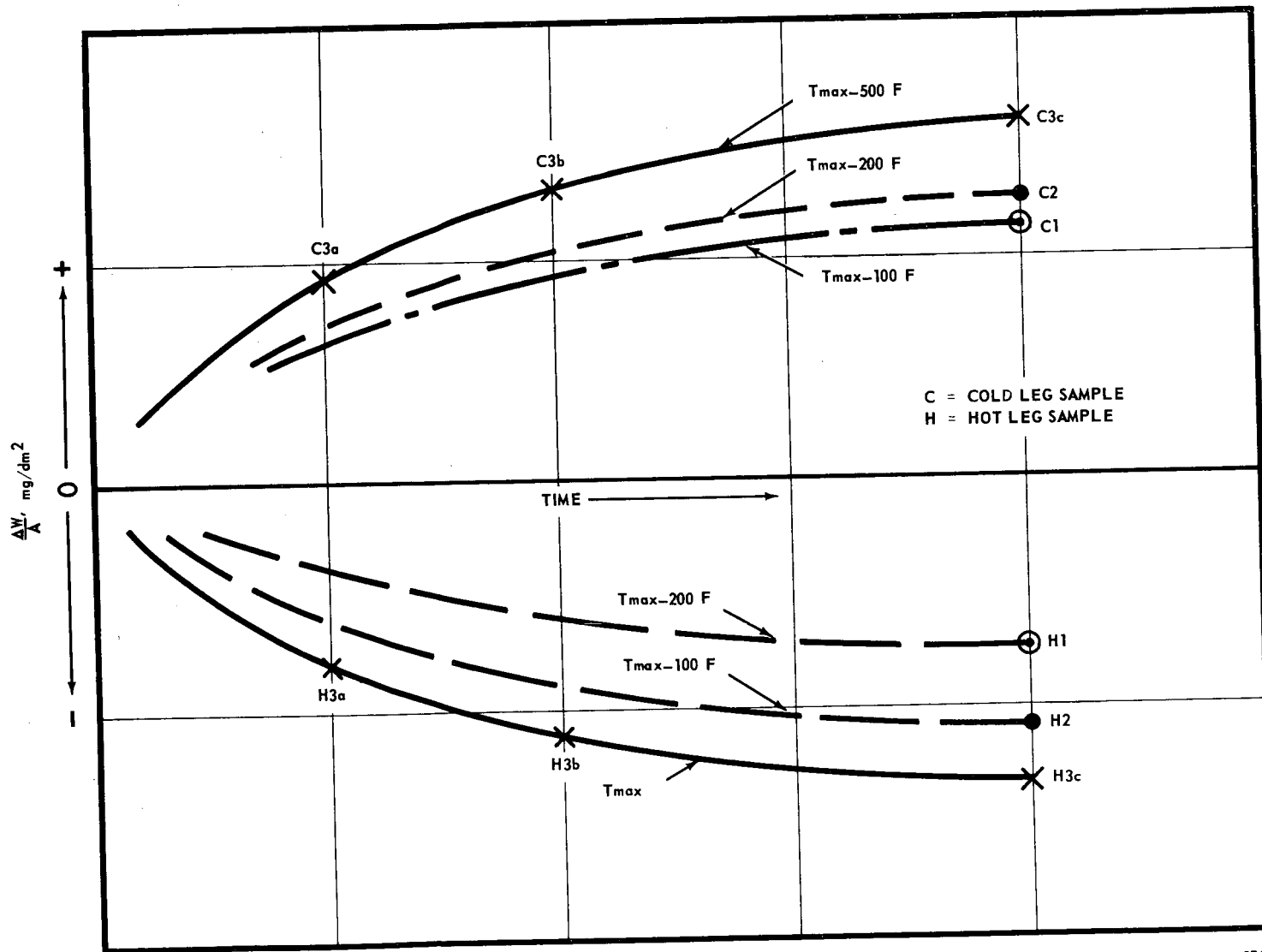


FIGURE B-2 POSSIBLE FORM OF MASS TRANSFER DATA WEIGHT CHANGE VS TIME

### Metallographic Examination

Because it is probably the best single tool for determining the nature and extent of liquid metal corrosion, microscopic examination of metallographically prepared specimens will be used for all samples. The removable samples will be studied metallographically after weight measurements; representative pipe sections, if considered necessary for study, will be obtained by removing pieces from the loop upon completion of the test run and examined metallographically. The latest edition of ASTM Standards, Part 3, "Metals Test Methods" will be used as a reference in preparing the specimens. Samples or pipe sections that exhibit significant build-up of deposition products or change in composition will be chemically analyzed.

### Loop and Sample Analysis

All materials used in constructing the test loop and corrosion samples will be purchased in accordance with applicable ASTM specifications and certified chemical analyses will be obtained. In addition, representative samples will be spot checked by chemical and/or spectrographic analyses using the latest edition of the ASTM "Methods of Chemical Analysis of Metals" and the ASTM "Methods of Emission Spectrochemical Analysis".

### Sodium Analysis

Oxide content of the sodium will be determined with an in-loop plugging indicator. This indicator detects the oxide saturation temperature by indicating the temperature at which a sudden flow decrease through the instrument occurs due to deposition of the oxide at a flow restricting orifice. Additional information on the use of this device as a method for oxide measurements is described in the Liquid Metals Handbook Sodium NaK Supplement (TID 5277). This method of oxide detection is known to provide reliable and reproducible results for the range of sodium impurities anticipated for these tests. The methods of analysis for Ca, Co, Cr, Fe, K, Li, Mn, Mo, Ni, Si, and Ti impurities in the sodium will be in accordance with the chemical or spectrographic analyses described in Attachment A.

## RADIOACTIVE METHODS AND PROCEDURES

To assist in the evaluation of corrosion and/or mass transfer, radioactive material transfer (tracer) techniques similar to those described in the Liquid Metals Handbook (TID 5277) will be used for the initial run with the type 316 stainless steel (monometallic) loop and for subsequent runs in the other test loops if weight change measurements do not provide sufficient information. The isotopic transport rates may be calculated by comparing the radioactivity of the source samples with the deposited activity will be removed from the loops according to the aforementioned schedule and in addition to non-radioactive examinations will be analyzed radiochemically to determine the transfer rate for each detectable radioactive species.

### Irradiated Sample and Control Specimen

Each loop using the radioactive material transfer technique will contain an irradiated sample (6 mls x 7/32-inch x 3-inch) with an initial activity of approximately 0.10 curies. Each irradiated sample will have a comparison control specimen which will be available for comparison of properties and will be monitored for activity decay. After corrosion testing the irradiated sample will be weighed, metallographically examined and radiochemically analyzed as described in Attachment B.

### Radiation Scanning of Loop

The irradiated sample in the loop will be biologically shielded so that the rest of the loop can be periodically scanned with a portable counter to determine qualitatively the rate and location of transferred activity.

### Radiochemical Analysis of Corrosion Samples

The radioactive deposits on the corrosion samples will be counted with a sodium iodide crystal whose output is coupled to a 256 channel analyzer. It is possible that some of the analyses can be obtained from the collector samples directly, without chemical separation, by counting the  $\text{Co}^{60}$  at the 2.5 MEV summation peak,  $\text{Fe}^{59}$  at 1.3 MEV (corrected for  $\text{Co}^{60}$  content),  $\text{Mn}^{54}$

at 0.8 MEV and Cr<sup>51</sup> at 0.32 MEV. If separation is required, the procedures for the radiochemical analysis of Fe<sup>59</sup>, Co<sup>58, 60</sup>, Cr<sup>51</sup>, Mn<sup>54, 56</sup> are presented in Attachment B. Similar procedures will be used for other species that may be present and are detected by the multichannel analyzer examination of the sample.

#### Radiochemical Analysis of Impurities in Sodium

Concentrations of the activated species of Fe, Cr, Co, and Mn will be determined in sodium samples from loops using the radioactive tracer technique to help provide a material balance for the individual isotopes which are involved in the transfer process. After reacting the samples with alcohol and water, radiochemical analysis for the radioactive isotopes will be in accordance with the procedures described in Attachment B.

ATTACHMENT A

ANALYTICAL PROCEDURES FOR NON-RADIOACTIVE

IMPURITIES IN SODIUM SAMPLES

CHEMICAL PROCEDURES

Calcium  
Chromium  
Iron  
Lithium  
Nickel  
Potassium

SPECTROGRAPHIC PROCEDURES

Calcium  
Cobalt  
Iron  
Manganese  
Molybdenum  
Nickel  
Silicon  
Titanium



## CALCIUM

### Abstract

Calcium is titrated to a visible end point with an EDTA salt (Disodium ethylene-diaminetetraacetate dihydrate). Calcein indicator is employed to make the method applicable in the presence of magnesium.

### Reagents and Apparatus

Absolute Methanol

Hydroxide-Cyanide solution, (1N sodium hydroxide and 0.7N potassium cyanide)

Calcein Indicator, 2% solution; keep in foil covered container, G. Fredrick Smith Company, Columbus, Ohio

Iceland Spar (designated for standardizing), J. T. Baker Chemical Co.

0.002M Standard EDTA solution

Microscope illuminator

Lamp, G.E., Type 16 $\frac{1}{2}$  - 29 (100 watts) 120 V

Three Kodak Wratten Filters #78AA

Magnetic stirrer

Microburet

### Procedure

1. Dissolve the sample in an inert atmosphere by the addition of small pieces to methanol. Add an equal volume of deionized water and heat carefully to evaporate the methanol. Cool.
2. To a sample aliquot, add 1 ml hydroxide-cyanide solution and 1 drop of 2% calcein indicator solution.
3. Prepare blank in same manner, substitute deionized water for the sample. (Note 1)
4. Illuminate samples with the triple filtered microscope illuminator -- exclude other light. The blank appears pink and the sample greenish-yellow.
5. Titrate sample with standard EDTA (Note 2) while stirring continuously. (Note 3)

6. The end point is reached when the last trace of yellow is gone and the sample matches the blank in color.

#### Notes

1. A given blank should not be used with samples prepared appreciably later because the color deteriorates with time.
2. A 0.001M solution of Iceland Spar in hydrochloric acid is used to standardize the EDTA solution.
3. Due to the high pH ( $>11$ ), the calcium salts tend to precipitate. To prevent false end points, vigorous stirring is necessary.
4. Magnesium does not interfere in a 100 molar excess. It only interferes when present in large enough quantities to precipitate visibly with the hydroxide-cyanide solution.

#### Reference

Socular, S. J., and Salach, J. I., Analytical Chem. **31**, 473 (1959).

## CHROMIUM

### Abstract

Chromium is determined colorimetrically with s-Diphenylcarbazide reagent.

### Reagents

Absolute Methanol

0.1N Silver Nitrate solution

1N Ammonium Persulfate

6N Sulfuric Acid

1:10 (~1N) Hydrochloric Acid

1:100 (~0.1N) Hydrochloric Acid

0.1% Methyl Orange indicator

2N Sodium Hydroxide

s-Diphenylcarbazide solution, 4 grams of phthalic anhydride and 0.25 gm of s-Diphenylcarbazide in 100 ml of 95% ethanol (stable 1 month).

### Procedure

1. Dissolve the sample in an inert atmosphere by the addition of small pieces to methanol. Add an equal volume of deionized water and heat carefully to evaporate the methanol. Acidify with nitric acid.
2. Take an aliquot to contain 0.005-0.015 mg of chromium. Add 5 ml of 0.1N silver nitrate. Mix and add 10 ml of 1N ammonium persulfate. Boil for 30 minutes and while still hot add 20 ml of 1:10 hydrochloric acid. Filter and wash the silver chloride on the filter with 1:100 hydrochloric acid.
3. Dilute to about 60 ml and make approximately neutral to methyl orange with 2N NaOH without adding indicator (approximately pH 4). Adjust the acidity of the approximately neutral solution by adding 3.3 ml of 6N sulfuric acid and transfer to a 100 ml volumetric flask. Add 1 ml of the reagent and dilute to volume.
4. Determine optical density against a reagent blank at 540 mu in a 10 cm Beckman cell within 5 minutes. Read ppm from a calibration curve obtained with standard solutions.

### Reference

Snell, F. D., and Snell, C. T., Colorimetric Methods of Analysis, New York, D. Van Nostrand Co., Inc. (1957).

## IRON

### Abstract

Iron is determined colorimetrically with o-phenanthroline reagent.

### Reagents

Absolute Methanol

Hydrazine Hydrate, 85%

o-phenanthroline reagent, dissolve 0.5 gm in 5 drops concentrated hydrochloric acid and 1 ml deionized water. Add 100 ml water. Add 200 gm sodium acetate and 200 ml glacial acetic acid. Warm, stir until dissolved and dilute to 500 ml.

### Procedures

1. Dissolve the sample in an inert atmosphere by the addition of small pieces to methanol. Add an equal volume of deionized water and heat carefully to evaporate the methanol. Acidify with hydrochloric acid by adding 1 ml of 1:9 HCl excess for each 50 ml of solution. Boil 5 minutes to dissolve iron.
2. Take an aliquot to contain 0.005 - 0.12 mg of iron. Evaporate if necessary. Add sample aliquot to a 50 ml flask containing 2.5 ml of concentrated hydrochloric acid, 2 ml of o-phenanthroline reagent, and 5 drops of hydrazine hydrate. If pH is not 3 - 5, adjust with concentrated hydrochloric acid or concentrated ammonium hydroxide.
3. Wait 30 minutes for complete reduction of iron and determine optical density with a 520 mu filter in a 4 cm Klett cell or a 10 cm Beckman cell at 510 mu. Read against the original solution instead of a reagent blank since other colors from material may cause interference in true iron reading.
4. Read ppm from calibration curve obtained with standard solutions.

### References

1. Sandress, Ernest, Colorimetric Determination of Traces of Metals, 2nd Ed. New York, Interscience Publishers, (1950).
2. ASTM Standards, American Society for Testing Materials, (1955).

## LITHIUM

### Abstract

Lithium is determined by flame photometry.

### Reagents and Apparatus

Standard lithium solutions

Beckman Model DU Flame Spectrophotometer

Didymium Filter

Hydrochloric Acid

### Procedure

1. Dissolve the sample by dropwise addition of deionized water in an inert atmosphere. Acidify with hydrochloric acid.
2. Determine the approximate sample composition by reading trial samples. Prepare standard lithium solutions containing anions and cations to as closely match the sample composition as possible. Usually a set of standards must be made for each sample since compositions vary.
3. Determine luminosity (% transmittance scale) of the sample against a deionized water blank using a Didymium Filter. Use the following settings:

<u>Wave Length</u>	<u>Slit Width</u>	<u>Sensitivity</u>
670 mu	0.1 - 0.2 mm	Two to four turns from clockwise limit

4. Read ppm lithium from a calibration curve prepared from standard solutions.

### References

1. Farquhar, Analysis of the Alkali Metals, American Potash & Chemical Corporation, Trona, California, (Meeting of the American Chemical Society, September, 1958, Chicago, Illinois).
2. F. Burriel - Marti', J. Ramirez - Munoz, Flame Photometry A Manual of Methods and Applications, New York, Elsevier Publishing Company (1957).

## NICKEL

### Abstract

Nickel is determined colorimetrically with dimethylglyoxime reagent.

### Reagents

Absolute Methanol

Ammonium Citrate Solution, 540 gm per liter, filter if necessary.

Dimethylglyoxime, 1.0 gm in 500 ml of ammonium hydroxide, dilute to one liter, make fresh, good for one month.

Iodine Solution, dissolve 8 gm of potassium iodide and 2.6 gm of iodine in a minimum of water and dilute to one liter.

### Procedure

1. Dissolve the sample in an inert atmosphere by the addition of small pieces to methanol. Add an equal volume of deionized water and heat carefully to evaporate the methanol. Acidify with hydrochloric acid.
2. To an aliquot containing not more than 0.3 mg nickel in a 50 ml volumetric flask add in the following order:
  - 5 ml ammonium citrate solution
  - 5 ml iodine solution
  - 10 ml dimethylglyoxime
3. Dilute to mark and mix. Determine optical density after 10 minutes against a reagent blank at 540 m $\mu$  in a 10 cm Beckman cell.
4. Read ppm nickel from a calibration curve prepared with standard solutions.

### Reference

ASTM Methods for Chemical Analysis of Metals, American Society for Testing Materials, (1956).

## POTASSIUM

### Abstract

Potassium is determined by flame photometry.

### Reagents and Apparatus

Standard potassium solutions

Beckman Model DU flame spectrophotometer

Didymium Filter

Hydrochloric Acid

### Procedure

1. Dissolve the sample by dropwise addition of deionized water in an inert atmosphere. Acidify with hydrochloric acid.
2. Determine the approximate sample composition by reading trial samples. Prepare standard potassium solutions containing anions and cations to as closely match the sample composition as possible. Usually a set of standards must be made for each sample since compositions vary.
3. Determine luminosity (% transmittance scale) of the sample against deionized water blank using a Didymium filter.
4. Use the following settings:

<u>Wave Length</u>	<u>Slit Width</u>	<u>Sensitivity</u>
766 mu	0.05 - 0.15	Two to four turns from clockwise limit

5. Read ppm potassium from calibration curve prepared from standard solutions.

### References

1. Farquhar, Analysis of the Alkali Metals, American Potash & Chemical Corporation, Trona, California. (Meeting of the American Chemical Society, Chicago, Illinois, September, 1958).
2. F Burriel - Marti', J. Ramirez - Munoz, Flame Photometry A Manual of Methods and Applications, New York, Elsevier Publishing Company, (1957).

## SPECTROGRAPHIC DETERMINATIONS

### Abstract

Samples are dissolved by the dropwise addition of deionized water under an inert atmosphere, neutralized with sulfuric acid, evaporated, and loaded into craters drilled in graphite electrodes. The electrodes are arced to burn the samples and excite the impurity spectra. Different procedures are used, depending upon the elements sought.

### Reagents and Apparatus

18N Sulfuric Acid

2N Sulfuric Acid

Phenolphthalein Indicator

Jaco, 3.4 meter Ebert Spectrograph

### Procedure I Cobalt, Iron, Manganese, Molybdenum, Nickel, Titanium

1. Dissolve a 1 gm sample of sodium in a pyrex cup by the dropwise addition of deionized water under an inert atmosphere.
2. Neutralize using 18N sulfuric acid initially followed by 2N sulfuric acid to the phenolphthalein end point.
3. Evaporate to dryness then load 50 mg of sample into graphite electrodes.
4. Arc in the Ebert spectrograph for 2 minutes at 15 amps.
5. Develop plate and read on microphotometer in 2200-3500 Å range.

### Procedure II Silicon

1. Dissolve a 1 gm sample of sodium in a steel cup by the dropwise addition of deionized water under an inert atmosphere.
2. Neutralize using 18N sulfuric acid initially followed by 2N sulfuric acid to the phenolphthalein end point.
3. Evaporate to dryness then load 75 mg of sample into graphite electrodes.
4. Arc in the Ebert spectrograph for 2 minutes at 15 amps.
5. Develop plate and read on microphotometer in 2200-3500 Å range.



Procedure III Calcium

1. Dissolve a 1 gm sample of sodium in a steel cup by the dropwise addition of deionized water under an inert atmosphere.
2. Neutralize using 18N sulfuric acid initially followed by 2N sulfuric acid to the phenolphthalein end point.
3. Evaporate to dryness then load 10 mg of sample into graphite electrodes.
4. Arc for 2 minutes at 5 amps in the Ebert spectrograph with filters.
5. Develop plate and read on microphotometer in the 4200-6700 Å range.

ATTACHMENT B

ANALYTICAL PROCEDURES FOR ACTIVATED IMPURITIES

Chromium - 51

Manganese - 54 and 56

Iron - 59

Cobalt - 58 and 60

## CHROMIUM - 51

### Principle of Method

Chromium is oxidized to chromate, scavenged with  $\text{Fe}(\text{OH})_3$  to remove impurities then precipitated as  $\text{BaCrO}_4$ .

### Reference

UCRL-432

LA-1721

### Reagents and Apparatus

Chromium carrier (10 mg Cr(III)/ml)

Barium Nitrate (saturated - 10%)

$\text{KBrO}_3$  solid

1M Ammonium acetate 7.7 gm/100 ml

Iron carrier (10 mg Fe/ml)

KOH dilute

Phenolphthalein

Whatman Filter paper #540 w.4 cm.diameter

### Procedure

1. To 200 ml of aqueous sample add 2 ml chromium and iron carriers. Add KOH to phenolphthalein end point and heat to coagulate the precipitate. Allow to settle briefly and decant most of the supernatant liquid to waste. Centrifuge in a 50 ml centrifuge cone and discard supernate.
2. Dissolve in 4 - 6 drops of concentrated HCl. Add 15 ml  $\text{H}_2\text{O}$  and about 0.1 gm  $\text{KBrO}_3$  crystals. Place in beaker of boiling water for 5 - 10 minutes to oxidize chromium to chromate. Add KOH to phenolphthalein end point and centrifuge. Discard precipitate.
3. Add 3 - 4 ml saturated  $\text{Ba}(\text{NO}_3)_2$  and 3 - 4 ml 1M ammonium acetate. Centrifuge out the  $\text{BaCrO}_4$ .
4. Dissolve  $\text{BaCrO}_4$  in 6 drops HCl 10 ml water, dilute to 30 ml and reprecipitate with 5 ml ammonium acetate and 5 ml  $\text{Ba}(\text{NO}_3)_2$ .
5. Filter, mount and count gamma.

### Note

Chemical yields are good and correction for loss in purification is not normally required.

## MANGANESE - 54 and 56

### Principle of Method

Manganese is precipitated as  $MnO_2$  from nitric acid solution.

### Reference

UCRL-432.

### Reagents and Apparatus

Manganese carrier 3.0 g/100 ml  $MnSO_4 \cdot H_2O$  (10 mg Mn/ml)

NaOH 10%

Nitric acid 1:10

$KBrO_3$

$H_2O_2$  30%

### Procedure

1. To 200 ml of aqueous sample add 2 ml manganese carrier.
2. Add 5 ml of 10% NaOH to the cold solution then heat to boiling to coagulate the precipitate. Allow to settle briefly and decant most of the clear supernatant liquid to waste. Transfer slurry to a 50 ml centrifuge tube.
3. Centrifuge and discard supernatant liquid.
4. Dissolve in 10 ml of (1:10) nitric acid.
5. Add a small scoop (0.1 gm) of  $KBrO_3$  and place in boiling water for 5 - 10 minutes. Centrifuge and discard supernatant liquid.
6. Dissolve precipitate in 10 ml 1:10 nitric acid containing a drop of 30%  $H_2O_2$  (avoid large excess of  $H_2O_2$ ).
7. Add 2 small scoops (0.2 gm) of  $KBrO_3$  sufficient to destroy excess of  $H_2O_2$  and enough additional to precipitate  $MnO_2$  again. Place in boiling water 5 - 10 minutes.
8. Filter, mount, and count.

### Note

Chemical yields are good and correction for loss in purification is not normally required.

## IRON - 59

### Principle of Method

Iron is extracted in isopropyl ether from 9M HCl. The iron is then mounted as  $\text{Fe}(\text{OH})_3$ .

### References

UCRL-432

LA-1721

### Reagents and Apparatus

Iron carrier 4.8 gm/100 ml  $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$  (10 mg Fe/ml)

Cobalt carrier 10 mg Co/ml

Ammonium hydroxide

HCl - 9M (3:1)

Isopropyl ether

Whatman #540 filter paper 2.4 cm diameter

### Procedure

1. To 200 ml of aqueous sample add 200 of iron carrier, 5 drops Co carrier, an excess of ammonium hydroxide and heat to coagulate the precipitate.
2. Filter in a gravity funnel on #40 Whatman paper.
3. Dissolve the precipitate from the filter with 15 ml of 9M HCl collecting solution in a separatory funnel.
4. Extract iron with four 10 ml portions of isopropyl ether. Combine the ether layers in a 250 ml beaker, add water, boil to a low volume, add 1 ml 3%  $\text{H}_2\text{O}_2$  and 1 ml concentrated  $\text{H}_2\text{SO}_4$  and fume until organic matter is destroyed.
5. Take up in a small amount of HCl (4 drops concentrated), dilute with 10 ml water and warm. Remove from heat, add 5 drops cobalt carrier and add 5 ml concentrated  $\text{NH}_4\text{OH}$ . The large excess of  $\text{NH}_4\text{OH}$  keeps cobalt in solution. Allow precipitate to settle briefly.
6. Filter, mount, and count gamma.

### Note

Chemical yields are good and correction for loss in purification is not normally required.

## COBALT - 58 and 60

### Principle of Method

Cobalt is precipitated as Potassium Cobaltinitrite.

### Reference

UCRL-432

LA-1721

### Reagents and Apparatus

Potassium Nitrite (80 gm/100 ml, 8 ml glacial acetic acid)

Glacial acetic acid

Cobalt carrier (4.0 gm/100 ml  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ) (10 mg Co/ml)

KOH dilute

Phenolphthalein indicator

Whatman filter paper #540 2.4 cm diameter

### Procedure

1. To 200 ml of aqueous sample add 1.0 ml Co carrier a drop of phenolphthalein indicator. Warm and add KOH to end point to precipitate cobalt hydroxide. Allow to settle briefly and decant off most of clear supernatant liquid. Centrifuge remainder in a 50 ml centrifuge cone, discard supernatant liquid. Dissolve in HCl, dilute and reprecipitate.
2. Dissolve precipitate in 1 ml glacial acetic acid and 15 ml water. Add 15 ml potassium nitrate. Allow 3 minutes for complete precipitation. Centrifuge, discard supernate.
3. Slurry up in 30 ml water and filter, mount and count for beta or gamma.

### Note

Chemical yields are good and correction for loss in purification is not normally required.

### APPENDIX C - STEEL ACTIVATION

Prior to the use of the radioactive tracer technique in test operation, samples representative of the materials under investigation were irradiated and analyzed for long lived gamma emitters. In addition to checking out the radiochemical procedures for application to the mass transfer loops this work was used to determine if there were any tramp impurities in the steels that would interfere with this program.

The first portion of this Appendix "Activation and Radiochemical Analysis of Type 316 Stainless Steel and  $2\frac{1}{4}$ Cr-1Mo Alloy Steel Samples", describes in detail the procedures and results of the investigation. Based upon an assumed transfer rate of  $10 \text{ mg/dm}^2\text{-mo}$  used in design the results indicated that the techniques would provide satisfactory results.

The second portion of the Appendix, "Activation and Radiochemical Analysis of  $5\text{Cr}-\frac{1}{2}\text{Mo}-\frac{1}{2}\text{Ti}$  Alloy Steel Specimens", provides supplemental information for using the 5 Cr alloy steel as an activated sample.

RADIOCHEMICAL ACTIVATION AND ANALYSIS OF TYPE 316 STAINLESS STEEL  
AND 2 $\frac{1}{4}$ Cr-1Mo ALLOY STEEL SAMPLES

April 30, 1960

Prepared by T. J. Slosek

Summary

Capsule MI-1 which contained four metal strips, two each of 316 stainless steel and 2 $\frac{1}{4}$ Cr-1Mo alloy, was disassembled in the Radioactive Materials Laboratory. The metal strips were weighed and radiochemical analyses were performed. Both metals were found to contain four isotopes which should be suitable for application to tracer analysis techniques. These isotopes are chromium 51, manganese 54, iron 59, and cobalt 60.

Irradiation

Capsule MI-1 was irradiated for 772.8 MWD during GETR cycle 10. The irradiation period extended from February 2, 1960 to March 7, 1960. The capsule was located in GETR position XI with the bottom of the capsule 19 $\frac{1}{2}$ -inches above the bottom of the active portion of the reactor core. The average perturbed thermal flux in this location was estimated to be  $1.2 \times 10^{14}$  nv for 30 MW(t) reactor operation.

The irradiated capsule contained four metal samples. Two of these samples were type 316 stainless steel and the other two were a 2 $\frac{1}{4}$ Cr-1Mo steel alloy. All samples were 3/16-inch wide by 3-inches long by approximately 5-mils thick. After irradiation the capsule was transferred to the Radioactive Materials Laboratory where it was disassembled. The metal foils were removed, weighed, dissolved, and radiochemical analyses were performed.

Emission Spectrometer Analyses

Strips of both types of metal, which were taken from the same batch of material as those which were irradiated, were analyzed on an emission spectrometer. The makeup of the metals as determined from these analyses is tabulated in Table C-1.



TABLE C-1

EMISSION SPECTROMETER ANALYSIS RESULTS

	<u>316 SS</u> <u>(%)</u>	<u>2<math>\frac{1}{4}</math>Cr-1Mo</u> <u>(%)</u>
Iron	63.8	95.8
Chromium	18.5	2.3
Nickel	12.0	< 0.1
Molybdenum	2.5	0.82
Manganese	2.5	0.45
Silicon	1.5	0.45
Copper	0.22	0.12
Cobalt	0.03 - 0.04	nil

Radiochemical Analyses

The four metal strips were dissolved in aqua regia and aliquots of the resultant solutions were analyzed on a 256 channel gamma spectrometer. For all samples distinct activity peaks were observed for the following isotopes: chromium 51 (0.32 MEV), manganese 54 and/or cobalt 58 (0.84 MEV), and iron 59 and/or cobalt 60 (1.1 and 1.3 MEV). In order to obtain quantitative data the above elements were chemically separated using an ion exchange process. Gamma spectrometer analyses were repeated on the chemically separated elements. Pertinent data for each of the isotopes which were detected are given in Table C-2.

Sufficient quantities of chromium 51, manganese 54, iron 59, and cobalt 60 were present in both metals to permit the use of these isotopes in radioactive tracer studies in the sodium mass transfer loops. The amount of cobalt 58 was marginal. Its utilization is not important however since the cobalt 60 isotope will suffice. The results of the radiochemical analyses are given in Table C-3. All values listed in this table have been corrected for in-pile and out-of-pile decay.

TABLE C-2

NUCLIDE PROPERTIES

NUCLIDE	HALF-LIFE	$\lambda$ (SEC <sup>-1</sup> )	MECHANISM OF FORMATION	FORMATION REACTION
Chromium 51	27 days	$2.97 \times 10^{-7}$	(n, $\gamma$ )	Cr <sup>50</sup> $\xrightarrow{\sigma_c = 16 \text{ barns}}$ Cr <sup>51</sup> $\xrightarrow{\text{decay}}$ V <sup>51</sup> 4.4%
Manganese 54	300 days	$2.67 \times 10^{-8}$	(n, p)	Fe <sup>54</sup> $\xrightarrow{(n, p)}$ Mn <sup>54</sup> $\xrightarrow{\text{decay}}$ Fe <sup>54</sup> 5.9%
Iron 59	45 days	$1.78 \times 10^{-7}$	(n, $\gamma$ )	Fe <sup>58</sup> $\xrightarrow{\sigma_c = 0.9 \text{ barns}}$ Fe <sup>59</sup> $\xrightarrow{\text{decay}}$ Co <sup>59</sup> 0.33%
Cobalt 60	5.2 years	$4.19 \times 10^{-9}$	(n, $\gamma$ )	Co <sup>59</sup> $\xrightarrow{\sigma_c = 37 \text{ barns}}$ Co <sup>60</sup> $\xrightarrow{\text{decay}}$ Ni <sup>60</sup> 100%
Cobalt 58	71 days	$1.13 \times 10^{-7}$	(n, p)	Ni <sup>58</sup> $\xrightarrow{(n, p)}$ Co <sup>58</sup> $\xrightarrow{\text{decay}}$ Ni <sup>58</sup> 68%

TABLE C-3

RADIOCHEMICAL ANALYSIS RESULTS

(See Table C-6 for the 5Cr Results)

		<u>316 Stainless</u>		<u>2<math>\frac{1}{4}</math>Cr-1Mo</u>	
		<u>Sample (A)</u> 0.5226 g	<u>Sample (B)</u> 0.5308 g	<u>Sample (A)</u> 0.5103 g	<u>Sample (B)</u> 0.5194 g
Chromium 51	atoms/gram	$2.34 \times 10^{17}$	$2.35 \times 10^{17}$	$3.01 \times 10^{16}$	$2.95 \times 10^{16}$
	mc/gram	1882	1885	243	237
Manganese 54	atoms/gram	$3.78 \times 10^{14}$	$3.76 \times 10^{14}$	$5.21 \times 10^{14}$	$5.21 \times 10^{14}$
	mc/gram	0.272	0.271	0.376	0.376
Iron 59	atoms/gram	$4.59 \times 10^{15}$	$4.72 \times 10^{15}$	$7.45 \times 10^{15}$	$7.14 \times 10^{15}$
	mc/gram	22.2	22.8	35.9	34.4
Cobalt 60	atoms/gram	$7.89 \times 10^{16}$	$7.75 \times 10^{16}$	$5.89 \times 10^{15}$	$6.04 \times 10^{15}$
	mc/gram	9.06	8.89	0.666	0.683

### Discussion of Results

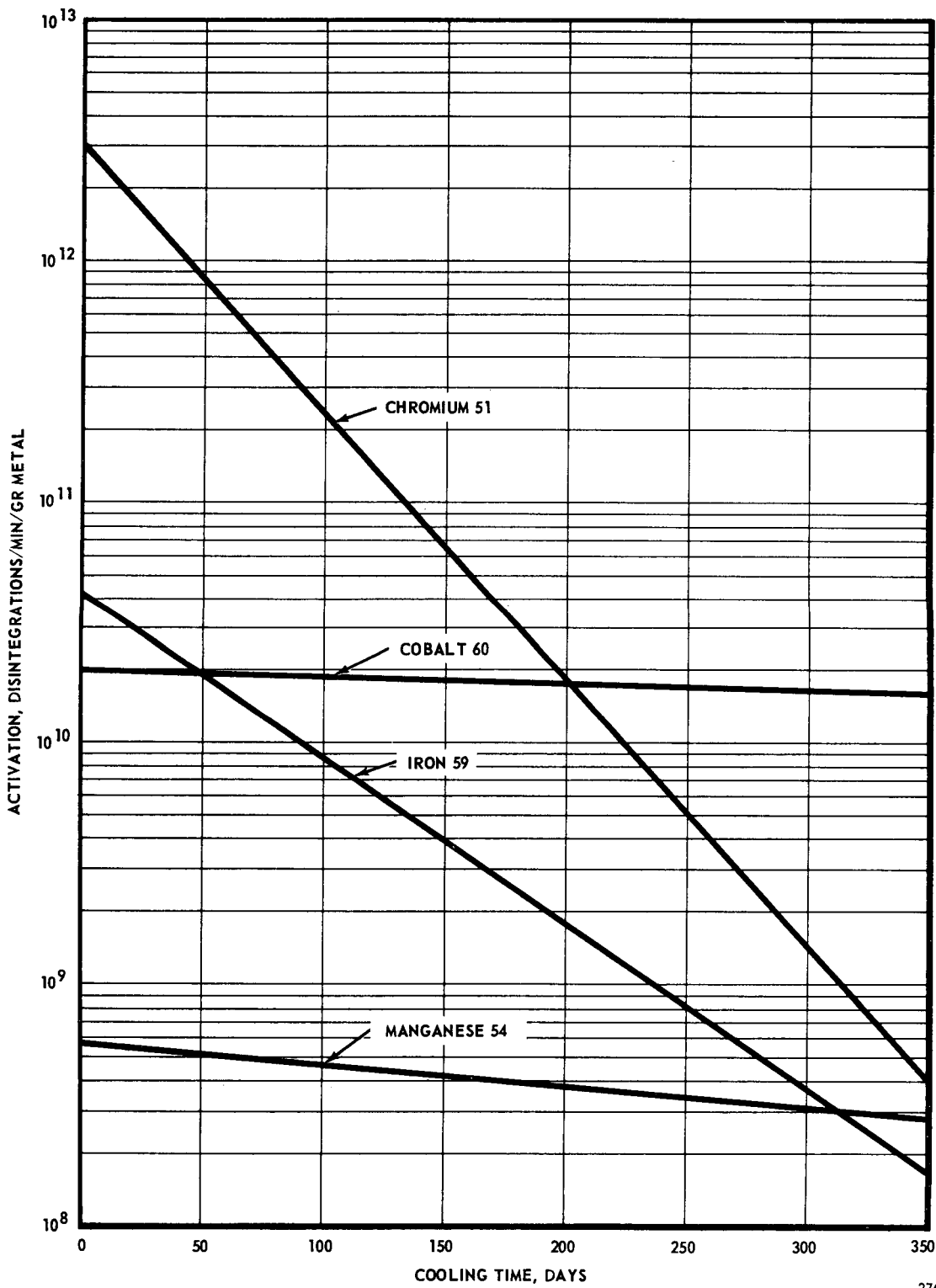
The results reported in Table C-3 indicate that analytical reproducibility between pairs of each type of metal is  $\pm 3\%$  or better. Millicuries/gram of each isotope were converted to disintegrations per minute per gram of material. Curves of disintegration rates as a function of cooling time were prepared. These curves are given in Figures C-1 and C-2. These curves serve to give a better indication of what may be anticipated during the course of the conduct of sodium mass transfer loop experiments.

In order to evaluate the mass transfer rates required to give accurate radiochemical analysis data, the curves of Figures C-1 and C-2 were used together with the following assumptions.

1. Test samples will contain approximately 0.5 grams of metal and will have dimensions of 3/16-inch x 3-inches x 0.006-inch.
2. Test samples will receive the same exposure as those which were included in capsule MT-1.

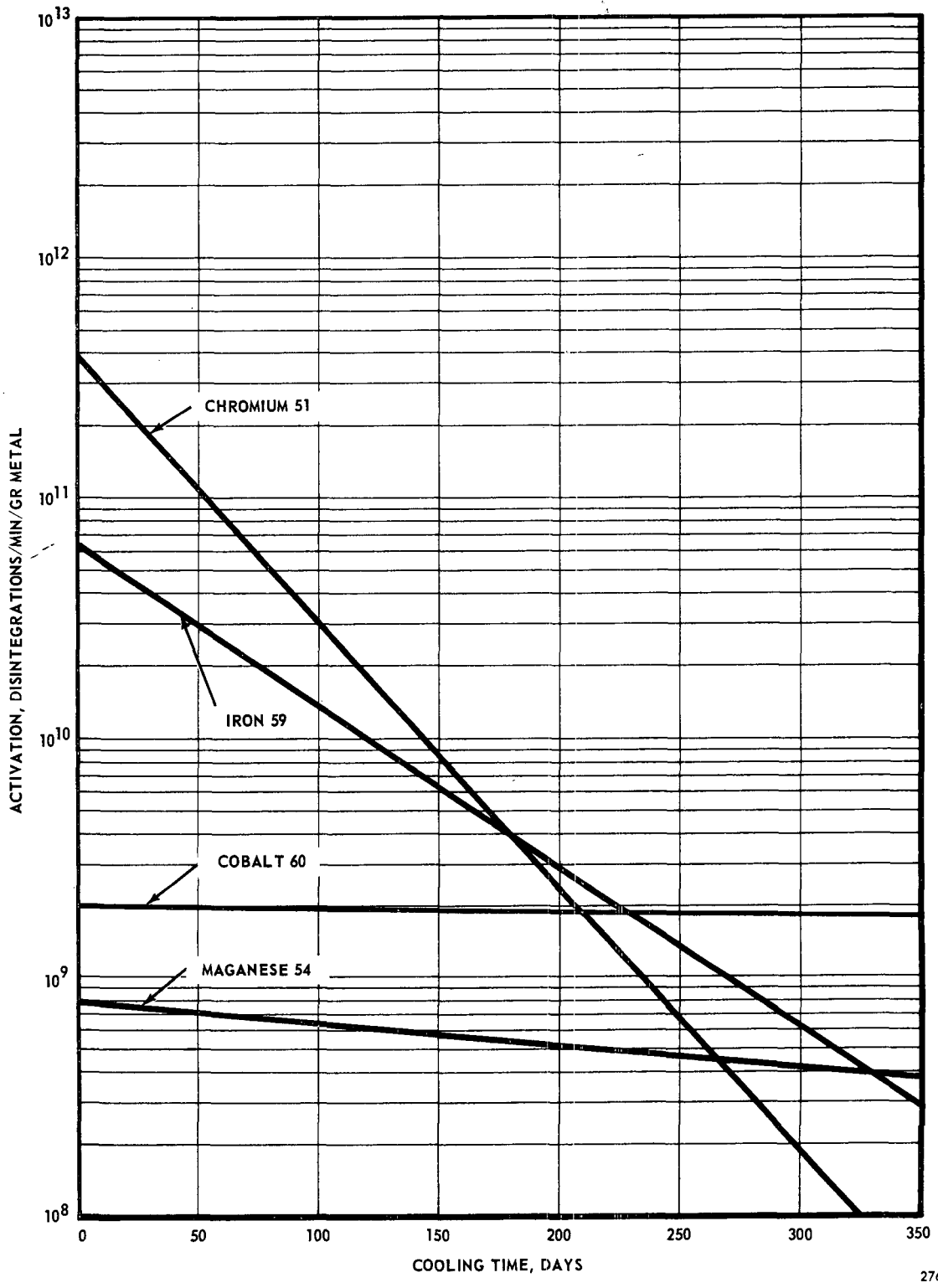
It is estimated that approximately  $10^4$  disintegrations/minute of a given isotope are required in order to obtain quantitative results which are accurate to  $\pm 5\%$ . For the case of chromium 51, this value is approximately  $5 \times 10^4$  since only about 20% of the disintegrations result in a 0.32 MEV gamma. Using the above values as guides, the fraction of initial radioactive sample material required to achieve  $\pm 5\%$  accuracy was calculated. For example, from Figure C-2 the dose rate from the cobalt 60 will be approximately  $2 \times 10^{10}$  d/m per gram of metal or approximately  $1 \times 10^{10}$  d/m per sample. As was stated above, approximately  $10^4$  d/m are required to achieve  $\pm 5\%$  reliability in the analytical results. This means that any one hot or cold leg "pickup" sample will have to contain  $10^4/10^{10}$  or  $10^{-6}$  of the original irradiated sample in order to permit the detection of cobalt 60 with a  $\pm 5\%$  reliability.

The same analysis was applied to the other isotopes. For purposes of calculation, it was assumed that the loop operation would cover approximately three months. The results are tabulated in Table C-4.



276-12

FIGURE C-1 ISOTOPIC ACTIVATION IN 316 STAINLESS STEEL  
 ( AFTER GETR IRRADIATION FOR 772.8 MWD IN  $1.2 \times 10^{14}$   $n_v$  THERMAL FLUX )



276-9

FIGURE C-2 ISOTOPIC ACTIVATION IN 2 1/4 Cr 1 Mo ALLOY  
 ( AFTER GETR IRRADIATION FOR 772.8 MWD IN 1.2 X 10<sup>14</sup> n<sub>v</sub> THERMAL FLUX )

TABLE C-4

RADIOACTIVE ISOTOPE ACCUMULATION REQUIREMENTS

<u>ISOTOPE</u>	<u>Fraction of Irradiated Sample which is Required to Permit Analysis to ±5%</u>	
	<u>316SS</u>	<u>2¼Cr-1Mo</u>
Chromium - 51	10 <sup>-6</sup>	10 <sup>-5</sup>
Manganese - 54	5 x 10 <sup>-5</sup>	5 x 10 <sup>-5</sup>
Iron - 59	5 x 10 <sup>-6</sup>	2 x 10 <sup>-6</sup>
Cobalt - 60	10 <sup>-6</sup>	10 <sup>-5</sup>

Supplementary Analyses

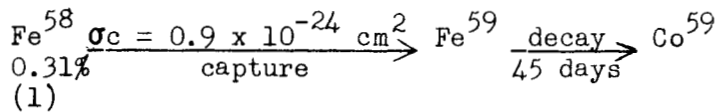
It is interesting to apply some checks to the results obtained. For instance, the metal foils were all located in approximately the same flux and therefore the activation of individual elements should be proportional to the amount of these elements which were present in the two types of steel tested. Table C-5 indicates that such a correlation was in fact obtained.

TABLE C-5

COMPARISON OF ACTIVATION BETWEEN THE TWO METALS

<u>Metal</u>	<u>Element</u>	<u>%</u>	<u>Millicuries/ Gram of Metal</u>	<u>Ratio mc/%</u>
316SS	Iron	63.8	22.5	0.353
2¼Cr-1Mo	Iron	95.8	35.1	0.366
316SS	Chromium	18.5	1883	102
2¼Cr-1Mo	Chromium	2.3	240	104

The correlation shown above indicates that (1) the fraction of each element present as determined by emission spectrometer analysis does not vary from one sample to the next and (2) the radiochemical analyses, at least for the elements listed in Table C-5 are accurate to ±3%. A rough check can also be applied to the reported thermal flux level ( $1.2 \times 10^{14}$  nv) in the reactor position occupied by capsule MT-1. Using iron 58 as a flux monitor and neglecting any corrections to the reported thermal neutron cross section due to resonance peaks, reactor moderator temperatures, etc., the following results are obtained:



$$\frac{N_2^t}{N_1^o} = 1 - e^{-\sigma_c \Phi t}$$

where:  $N_1^o$  = atoms of iron 58 present in the foil prior to irradiation

$N_2^t$  = atoms of iron 59 formed during irradiation

$\sigma_c$  = thermal neutron capture cross section of iron 58

$\Phi$  = thermal neutron flux

$t$  = irradiation time =  $2.22 \times 10^6$  seconds (equivalent full power operation)

If  $\sigma_c \Phi t \ll 1$ ;  $1 - e^{-\sigma_c \Phi t} \approx \sigma_c \Phi t$

$$\frac{N_2^t}{N_1^o} = \sigma_c \Phi t \quad \text{and}$$

$$\Phi = \frac{N_2^t}{N_1^o \sigma_c t}$$

Using sample 316SS (A) as an example, we get from Table C-4:

$$N_2^t = 2.40 \times 10^{15} \text{ atoms}$$

$$\begin{aligned} N_1^o &= \frac{(W) (A^o) (F_{58})}{MW} \\ &= \frac{(0.5226) (6.0 \times 10^{23}) (0.0031) (0.638)}{(58)} \\ &= 1.075 \times 10^{19} \text{ atoms} \end{aligned}$$

$$\Phi = \frac{N_2^t}{N_1^o \sigma_c t} = \frac{(2.40 \times 10^{15})}{(1.075 \times 10^{19}) (0.9 \times 10^{-24}) (2.22 \times 10^6)} = 1.12 \times 10^{14} \text{ nv}$$

The above value is in good agreement with the predicted flux of  $1.2 \times 10^{14}$  nv.



ACTIVATION AND RADIOCHEMICAL ANALYSIS OF 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti

ALLOY STEEL SAMPLES

October 14, 1960

Prepared By  
P. W. Mathay

Capsule MT-2 was irradiated for 772.8 MWD during Cycle 14 at the GETReactor. The exposure cycle covered the period from June 22, 1960 to July 22, 1960. The capsule was located in the space X7 of the southeast facility just outside the reactor core. The bottom of the capsule was 15 inches above the base of the active core. The average perturbed thermal flux in the X7 hole was estimated at  $1.4 \times 10^{14}$  nv for the 30 megawatt reactor operation.

The capsule contained four metal strips, two of 316 stainless steel and two of 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti alloy. The samples were 3.00-inches long by 0.1875-inches wide by about 0.006-inch thick. RML Operations received, disassembled and removed the samples. Radiochemistry weighed the samples and performed the chemistry analysis. The stainless strips were weighed and stored in a shielded container. One stainless sample, MT-2A, will be inserted in Sodium Loop No. 1 at San Jose. The other stainless steel sample, MT-2B, will be analyzed radiochemically at the time MT-2A is analyzed following removal from the test loop.

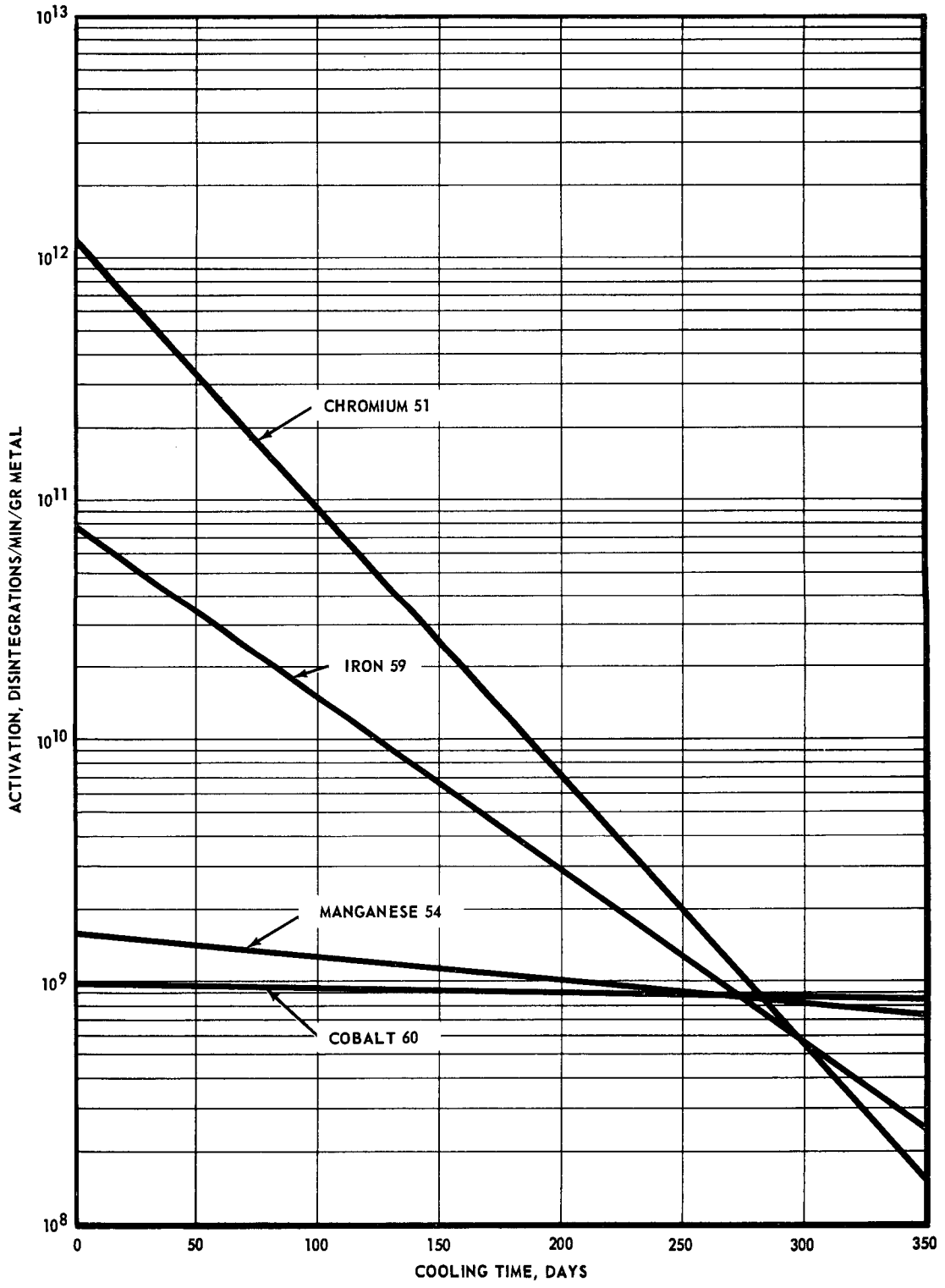
The two low alloy strips were weighed and analyzed by radiochemical methods. These samples contained sufficient amounts of four active isotopes to make them applicable for tracer techniques.

Chromium 51, Manganese 54, Iron 59, and Cobalt 60 isotopes are common to the 316 stainless steel, the  $2\frac{1}{4}$ Cr alloy and the 5Cr alloy. Table C-6 gives the radiochemistry results on the two 5Cr- $\frac{1}{2}$ Mo- $\frac{1}{2}$ Ti samples. Curves showing disintegration rate as a function of cooling time are given in Figure C-3.

TABLE C-6

(See Table C-3 for 316SS and 2 $\frac{1}{4}$ Cr Results)

		<u>5Cr-<math>\frac{1}{2}</math>Mo-Ti</u>	
		<u>Sample (A)</u> 0.4554 g	<u>Sample (B)</u> 0.4627 g
Chromium 51	atoms/gram mc/gram	$6.74 \times 10^{16}$ 541	$6.69 \times 10^{16}$ 536
Manganese 54	atoms/gram mc/gram	$9.69 \times 10^{14}$ 0.678	$1.18 \times 10^{15}$ 0.851
Iron 59	atoms/gram mc/gram	$5.57 \times 10^{15}$ 26.8	$5.48 \times 10^{15}$ 26.4
Cobalt 60	atoms/gram mc/gram	$4.98 \times 10^{15}$ 0.563	$2.89 \times 10^{16}$ 0.326



276-10

FIGURE C-3 ISOTOPIC ACTIVATION IN 5 Cr-1/2 Mo-1/2 Ti ALLOY  
 ( AFTER GETR IRRADIATION FOR 772.8 MWD IN 1.4 X 10<sup>14</sup> n<sub>v</sub> THERMAL FLUX )

## APPENDIX D

### STEEL SAMPLE REMOVAL UNDER OPERATING CONDITIONS

This document describes the study that was made to determine the feasibility of developing a sample handling mechanism which could be used to remove or insert a steel corrosion sample under operating conditions (1300 F - 10 psig sodium) of the mass transfer loops.

As a result of this report and the lack of evidence in the literature indicating that draining and cooling down a test loop for sample removal is detrimental to the corrosion data, it was recommended that at this time no further work should be devoted to developing such a device. This appendix, however, does indicate several possible methods of developing such a mechanism.

Included in this appendix are:

- D1 Purpose
- D2 Summary and Conclusions
- D3 Recommendations
- D4 Discussion
- D5 Cost

GENERAL ELECTRIC COMPANY  
ATOMIC POWER EQUIPMENT DEPARTMENT  
SAN JOSE, CALIFORNIA

FEASIBILITY STUDY  
OF  
OPERATIONAL SAMPLE HANDLING MECHANISMS  
FOR  
SODIUM MASS TRANSFER

December 24, 1959

Prepared By  
H. S. Jakuc

## STEEL SAMPLE REMOVAL UNDER OPERATING CONDITIONS

### D1 PURPOSE

This report is the result of a study to ascertain the feasibility of developing a sample handling mechanism which is to be used during operation of a high temperature sodium mass transfer loop. This mechanism has as its function the removal of a sample coupon from the sodium loop while the loop is operating thus eliminating the need of shutting down the loop each time a sample is removed.

### D2 SUMMARY AND CONCLUSIONS

It has been the intent of this report to investigate and describe a method or methods of removing sample coupons from an operating sodium system without shutting down the system in order to determine their feasibility. It is felt that the various schemes as presented in this report conceptually indicate that removing samples from an operating sodium system is feasible, but that further investigation is required.

The presentation of the various devices has been described in such a manner as to show concepts rather than detailed design. This does not mean, however, that details were neglected in considering the major advantages or disadvantages of the schemes. Major consideration was given to satisfying the requirements of the system as listed in the specification (see Attachment A) and as further described in the discussion. Specific mechanical methods or details were left to be completed when more detail is desired.

In the allotted time, a number of concepts were investigated, three of which are described in some detail in Sections I, II, and III of the Discussion Section of this report. It is felt that two of these are worthy of further consideration, and recommendations are listed below as to the direction to be taken.

A cost estimate for the components of the various schemes presented indicates that the manufacturing costs are approximately the same. However, the critical factor in the cost of any of these schemes would depend on the amount of engineering and drafting time required.

### D3 RECOMMENDATIONS

Two schemes, the valve-bellows arrangement and the EM pump-drybox arrangement, are worthy of further consideration.

In the valve-bellows arrangement, a positive means of guaranteeing a balanced system pressure and a positive means of indicating liquid level location is necessary. The use of existing liquid level technology could provide a satisfactory solution to these problems.

In the EM pump-drybox arrangement, tests should be performed to determine the effectiveness of the pump in balancing system pressure. This would determine the maximum pressure that a given size pump could safely balance within the current limitations of the pump cell and at the same time would determine whether the current flowing in the transformer is a good indication of the liquid sodium level.

For both of these arrangements, the ease with which manipulators inside the drybox can remove a Marman clamp and the cap from the system should be determined.

At this time, it is felt that the EM pump-drybox arrangement is the most promising system investigated to date, and component tests should be initiated to verify this conclusion.

### D4 DISCUSSION

The general requirements of the material handling mechanism are described in the specification in the attachment. Prior to elaborating on various schemes investigated, a discussion of the system conditions and sample removal sequence is presented.

The system conditions as stated in the specification may be varied if necessary to certain minimum values without affecting the basic requirement -- an operating system. During the sample removal, the temperature may vary a maximum of 100 F, the flow rate and the pressure may be lowered as long as flow continues, and leakage into the system of an inert gas in small quantities is permissible. Any portion of the handling system that represents

a permanently fixed extension of the sodium loop must be designed for a pressure of 100 psig and a temperature of 1300 F. Any material in the mechanism coming in contact with sodium shall be the same material as the loop material.

The material handling sequence requires the removal of three samples one at a time at different time intervals. Simply stated, the procedure for each sample would be: (1) removal from flow channel; (2) removal from sodium system; and (3) insertion into container for transportation to analytical laboratory.

Analyzing each step further, general functional principles of the mechanism become defined, a number of modes of attack become apparent, and the mutual dependence of various steps becomes evident. Step one can be accomplished in one of two ways: the sample can be moved from the flow channel to another part of the loop which can be separated from the main loop; or the sample can be separated from the remaining portion of the loop by shutting off the fluid flow to that particular channel but without stopping the flow through the remaining channels.

Step two, the removal of the sample from the sodium system, can only be accomplished by separating the portion of the system containing the sample from the remaining portion of the system. This can be accomplished by using valves, either gate or globe type depending on the procedure to be used to remove the sample; by using a freeze plug solidifying the sodium in this portion of the system; by using gas pressure to work against the pressure of the system; and by using the electromagnetic motor principle to obtain a force that operates against the pressure of the system.

Step three, insertion into the container for transportation to the analytical laboratory, can be accomplished manually in a standard glove box and no mechanism is necessary for its fulfillment.

Steps one and two are mutually dependent and will be considered as such in evaluating the above mentioned methods of attack.



From the alternatives mentioned in steps one and two, various combinations can be established and evaluated. These combinations are listed below:

1. Sample remaining in sample holder - Freeze plug
2. Sample remaining in sample holder - Globe valve
3. Sample removed from sample holder - Gate valve - Bellows
4. Sample removed from sample holder - Gas pressure
5. Sample removed from sample holder - Motor principle

Considering the operating requirements of the system and the high thermal conductivity of the fluid which results in high cooling requirements, Scheme No. 1 can be eliminated as unfeasible.

Scheme No. 2 requires a change in the sample holder and additional flow measuring devices. The present holder contains four flow channels, three are for low velocity flow samples and one is for high velocity flow samples. In this arrangement, a separate sample holder would be supplied for each sample so that shutting off the flow to any one sample would not stop the flow to the other samples. This results in: (1) a different loop configuration; (2) possible flow variations past the sample as compared to the present holder; (3) a change in flow conditions past the remaining samples when one sample is being removed; (4) an increase in the heat losses of the system; and (5) the use of at least two flow measuring devices to determine how much fluid is flowing in the other channels. These changes would question the comparability of the data obtained using this method with the data obtained using the present holder. A sketch of this arrangement is shown in Figure D-1.

The remaining methods require the sample to be moved from the sample holder to another portion of the loop. Each of these methods requires a means of controlled entry into the loop, controlled movement of a device to move the sample, and a reclosing of the loop.

In the first method (Scheme No. 3), a gate valve and a long stroke bellows assembly in combination with inert gas pressure is the means through which entry and exit is accomplished. A detailed description of the mechanism is provided in Section I and a sketch is shown in Figure D-2.

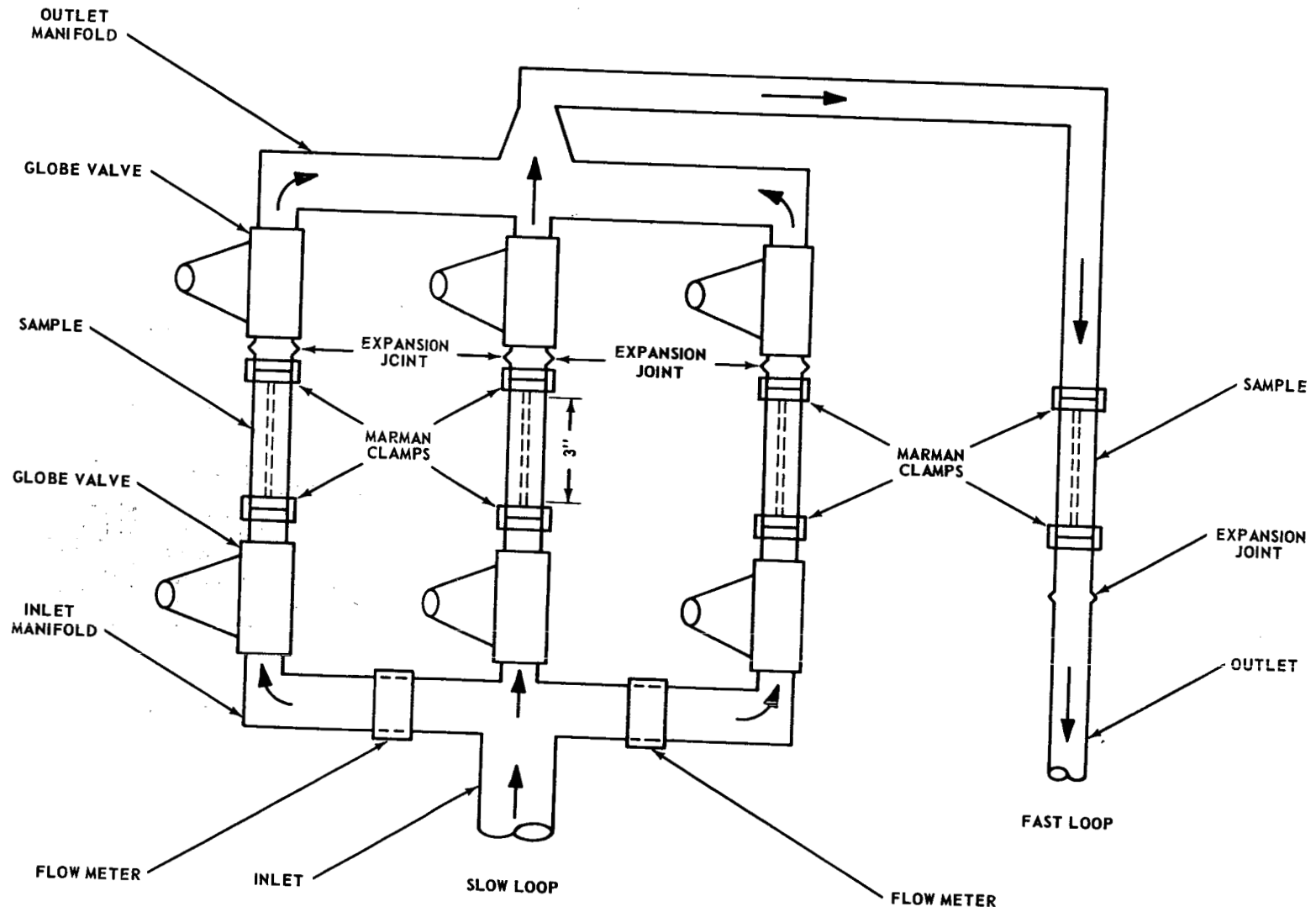
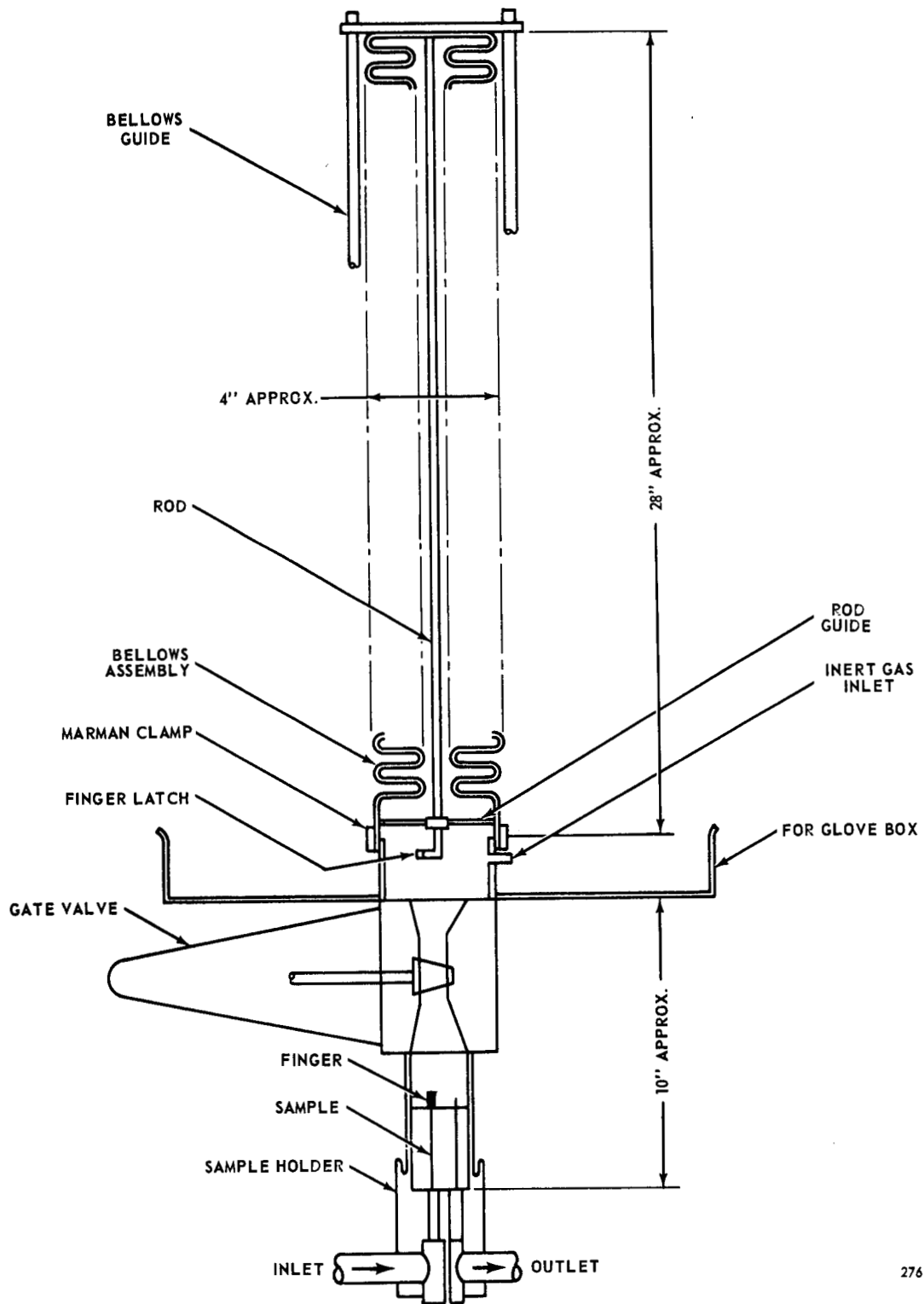


FIGURE D-1 FREEZE VALVE ARRANGEMENT



276-7

FIGURE D-2 VALVE - BELLOWS ARRANGEMENT

In the second method (Scheme No. 4), gas pressure is supplied to a circular drybox, containing mechanical tools, which is affixed to the system when sample removal is required. Pressure in the box is increased until it balances the pressure in the system. At that time, entry is made into the system through the standard flanged attachment. This method is described in Section II and is shown in Figure D-3.

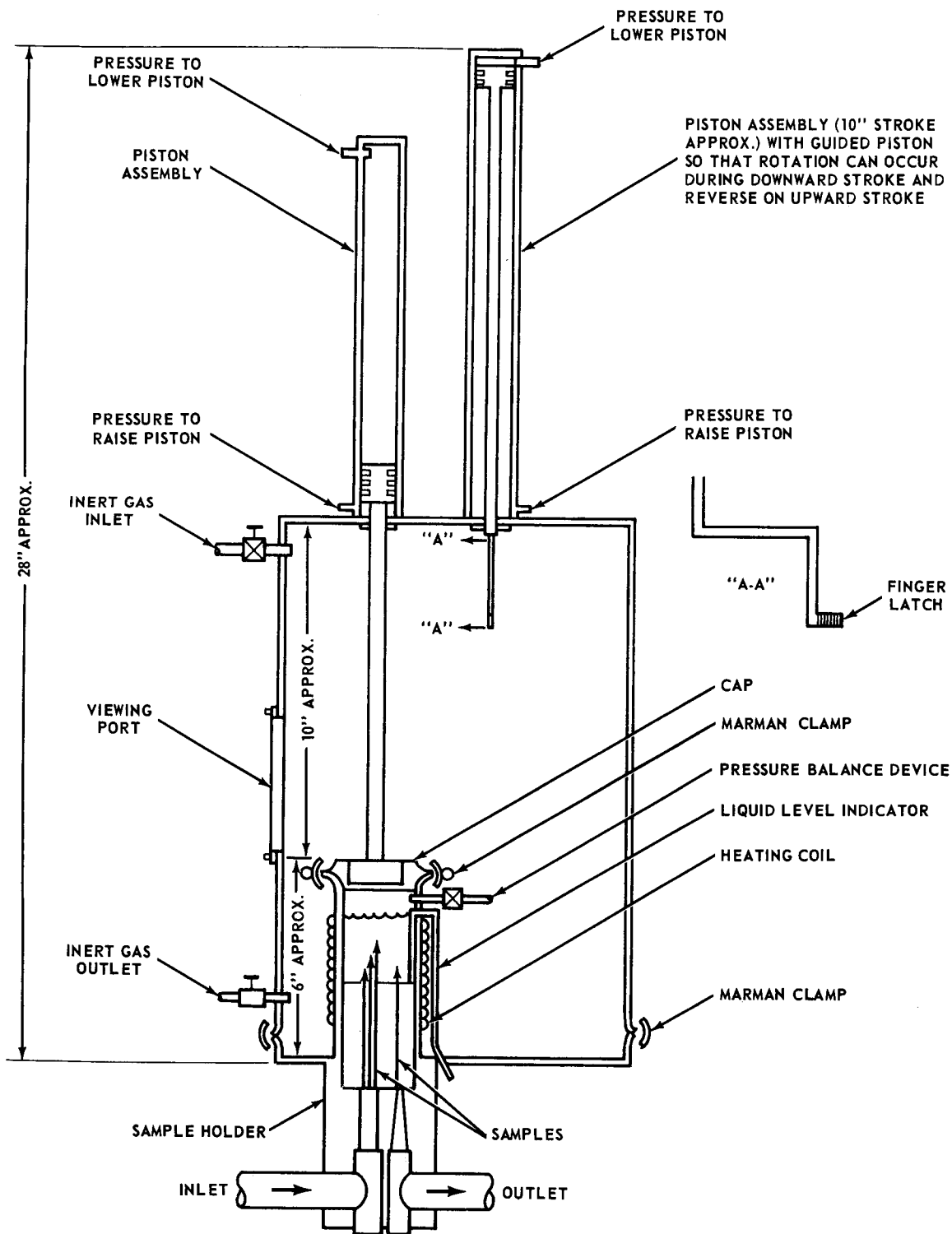
The third method (Scheme No. 5) used the electromagnetic principle that a conductor carrying current in a magnetic field produces a force. An EM pump is used to obtain the required force and in conjunction with a low pressure filled drybox containing mechanical tools permits entry, exit, and removal of the sample from the system. This method is described in Section III and is shown in Figure D-4.

## SECTION I

### VALVE - BELLOWS ARRANGEMENT

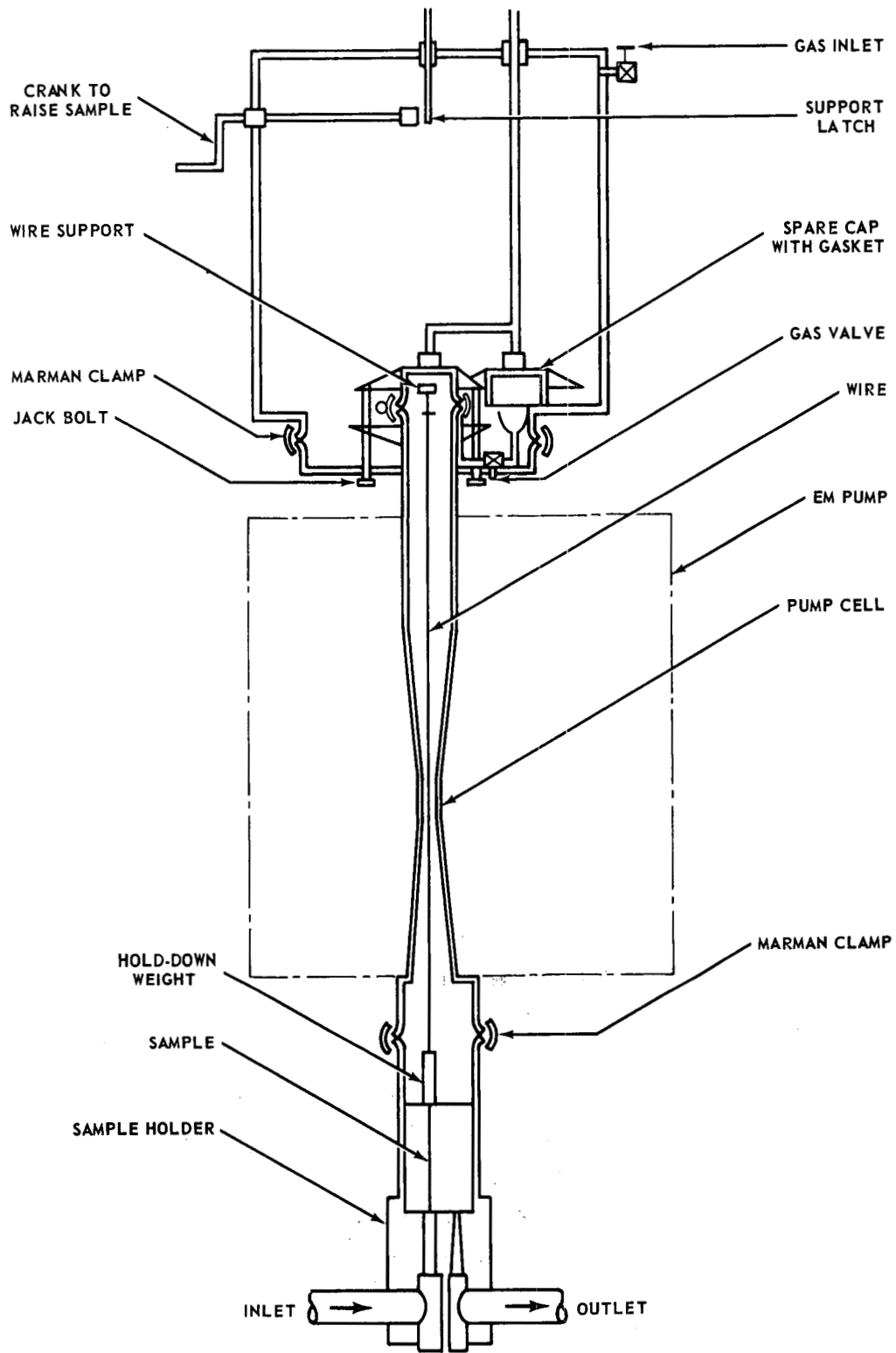
The device consists of a gate valve permanently fixed in the system and a bellows assembly containing a rod with a finger latch. The bellows assembly is fastened to the system by a Marman clamp. When the loop is being operated and no sampling is required, the bellows assembly can be replaced by a cap. The use of the cap permits the opening of the gate valve during normal operation and eliminates any metal-to-metal contact of the valve parts except for short periods of time during sample removal. The entire bellows assembly would be enclosed in a glove box to permit opening the system under an inert atmosphere. A means of guarantying a balanced system pressure and a means of indicating liquid level location must be used prior to opening the system. A preliminary layout of this arrangement is shown in Figure D-2.

Inquiries as to the availability of a suitable valve and bellows were made to a number of manufacturers. Listed below are the names of the ones who can definitely satisfy our requirements:



276-13

FIGURE D-3 PISTON ASSEMBLY - DRYBOX ARRANGEMENT



276-8

FIGURE D-4 EM PUMP - DRYBOX ARRANGEMENT

### Valve

1. Powell Valve Company  
Cincinnati, Ohio
2. P-K Industries  
North Arlington, New Jersey

### Bellows

1. Nucleodyne Division  
Cook Electric Company  
Franklin Park, Illinois
2. Solar Aircraft Company  
San Diego, California

### Operating Sequence

1. With the cap on the system, insert an inert gas at a pressure above that of the system pressure. Using a liquid level indicator, insure that the sodium level is below the valve and then check to insure that the system pressure is balanced.
2. Close the gate valve (liquid sodium below the valve, inert gas above).
3. Bleed off the gas between the valve and cap until it equals that of the glove box.
4. Remove cap and replace with bellows assembly.
5. Fill bellows with inert gas until pressure equals that of loop and open gate valve.
6. Compress bellows to indicated point for engagement of sample. (Finger latch on the rod engages with the finger on the sample.)
7. Expand bellows removing sample from sodium to inert gas and close gate valve.
8. Equalize pressure in bellows to that of glove box.
9. Open clamp and remove sample.
10. Replace bellows assembly on valve.
11. Secure clamp, fill bellows with inert gas, and open valve.
12. Place sample in gas filled container and remove from glove box.

### Advantages

1. Completely enclosed system.
2. Straight axial motion of bellows is only required.

3. Bellows can be replaced by cap during normal operation.
4. Valve surfaces not in contact except for short periods of time when removing sample.
5. Bellows assembly can be used on all the sample holders.
6. Stainless steel bellows can be used on both loops.

#### Disadvantages

1. Obtaining a reliable gate valve to operate properly at the conditions of the system.
2. Bellows manufacturers will not guarantee life of bellows.
3. Requires use of a positive liquid level indicator to indicate that liquid level is below the valve seat.

## SECTION II

### PISTON ASSEMBLY - DRYBOX ARRANGEMENT

This device consists of a drybox containing a number of manipulators operating in an inert atmosphere. These manipulators are used to remove the cap from the system and to remove the sample from the sample holder. The removal of the sample is accomplished by a finger latch attached to a rod. The rod's motion is axial and rotational, both accomplished by a guided piston which rotates 90° during the length of its stroke. A liquid level indicator and a means to insure pressure balance between the drybox and the liquid metal system determines when the system can be safely opened. A heating coil is provided to keep the temperature of the sodium constant. A preliminary layout of the devices is shown in Figure D-3.

#### Operating Sequence

1. Insert gas into drybox until pressure in box balances that of the system. Check liquid level indicator to insure that level of sodium is at the specified level.
2. Admit pressure to upper side of piston cylinder (cap removal) and open Marman clamp.
3. Raise cap.



4. Admit pressure to piston assembly to lower finger latch. Let piston move to indicated position to insure that the finger on the sample is engaged with latch.
5. Raise finger latch with sample.
6. Replace cap on system and tighten clamp.
7. Reduce pressure in drybox and remove sample.

#### Advantages

1. It can be used on all the sampling loops by interchanging the finger latch.
2. The liquid metal system is completely enclosed during normal loop operation.
3. No portion of the handling device comes in contact with sodium except the finger latch and this only occurs during sample removal. All operations for sample removal are accomplished by manipulators within the drybox.

#### Disadvantages

1. Maintaining a gas pressure to constantly and consistently balance the system pressure and yet not hinder flow or permit spillage.
2. Use of a heating coil to prevent cooling of liquid.
3. Large number of mechanical devices in a small volume (pneumatic, electrical, and mechanical).
4. Maintaining a leak-tight drybox (this applies to all dryboxes) but especially this scheme because of the large number of rotating and sliding seals required in the drybox.

### SECTION III

#### EM PUMP - DRYBOX ARRANGEMENT

This device consists of an electromagnetic pump, a drybox containing manipulators and viewing ports, and wires permanently affixed to the samples. The EM pump is used to balance the pressure of the system and in doing so keeps the sodium level below the throat of the pump cell. The drybox per-

mits removal of the cap, removal of the sample and replacement of the cap in an inert atmosphere. This is accomplished through the use of manipulators inside the drybox which are operated outside the drybox through seals. After the clamp is opened, the cap with gasket attached is raised and rotated so that a clear area above the opening is available. A spare cap with a gasket replaces the removed cap once the operation is completed. This is necessary because Marman clamp type gaskets are not reusable and must be replaced each time the cap is removed. The wires attached to the samples are suspended through a support plate located beneath the cap. The upper end of the wire is attached to a latch mechanism which engages to a support latch to raise the sample to an initial height. Once the latch is raised to a specific height, it is engaged by a crank. This crank raises the sample from the sodium system by winding the wire on a drum.

An ammeter associated with the pump transformer indicates the amount of current flowing in the pump secondary. This current indicates whether there is sodium in the throat of the cell or below the throat. A gas valve in the system insures that the system can be checked for balanced pressures prior to removing the cap. A preliminary layout of the device is shown in Figure D-4.

#### Operating Sequence

1. With drybox in place, start EM pump and check current. (The amount of current indicates if sodium is in the throat of the cell or below it.)
2. Insert inert gas into drybox, then open gas valve leading from the loop. If no sodium rises into the bowl, system is balanced.
3. Remove cap.
4. Using support latch, raise sample to be removed.
5. Engage latch with crank mechanism and raise sample completely out of system.
6. Close system with spare cap.
7. Close valve and EM pump.
8. Remove sample from drybox.

Advantages

1. System is completely enclosed during normal loop operation.
2. The pump cell remains in the system, but the transformer section can be utilized on the other sample removal section.
3. All operations are enclosed in a low pressure drybox and can be seen through a viewing port.
4. Any loop can be converted into an operational sampling loop by moving the cell section from one loop to another.
5. Liquid level can be ascertained by the current flowing in the transformer.
6. No portion of the device not permanently fixed to the system comes in contact with sodium at any time.

Disadvantages

1. During normal operation an external support must be used to protect the pump cell from distortion due to the pressure and temperature conditions.
2. Hold-down weights must be used to keep the sample in its holder during normal operation.
3. The unknowns of the physical and electrical conditions existing within the cell which determine the net force acting against the sodium. Prototype component testing will be required to clarify these unknowns.
4. Disturbing effect on force balance as sample is removed through cell.

D5 COST

The cost for the components of the schemes presented in Figures D-2, D-3, and D-4 are in the same price range, approximately \$4,000\*. However, this figure only considers one unit. If more than one unit is required, certain parts such as the bellows in Figure D-2 and the EM pump's transformer can be used on more than one unit thus decreasing the cost on the other units.

-----

\* Shop cost - not selling price.

Engineering cost on the other hand can be quite differentiated depending on the scheme considered. The complexity of the scheme shown in Figure D-3 would invariably involve more engineering development than the schemes shown in Figures D-2 and D-4. However, because the schemes are not detailed, any of the schemes may present a problem which requires a large investment in engineering or drafting time. Further investigation is required before realistic estimates can be determined.

ATTACHMENT A  
OF APPENDIX D

PROJECT  
SODIUM MASS TRANSFER

November 25, 1959

SPECIFICATION TITLE  
OPERATIONAL SAMPLE HANDLING REQUIREMENTS

REQUISITION NUMBER

474-30754

Prepared By  
M. R. Lane  
Advanced Engineering

A. SCOPE

The intent of this specification is to set forth the functional and general design requirements of the two sample handling mechanisms to be designed for use during operation of a high temperature sodium mass transfer loop.

B. REFERENCES

1. G.E. Drawing (Dated November 22, 1959) - Sample Holder
2. G.E. Drawing (Preliminary - No Number, Dated November 22, 1959) - Loop layout

C. GENERAL REQUIREMENTS

1. The sample handling mechanism shall provide a means of removing samples, one at a time, from flow channels in the sample holder shown in Reference No. 1 while maintaining the following system conditions:
  - a. System temperature - 1300 F maximum.
  - b. Sodium flow rate past sample - 3 fps. (reduced from normal conditions)
  - c. System pressure - 10 psig maximum.
  - d. Temperature of sodium in the vicinity of the sample holder being opened must be maintained at or very close to that of the sample holder.
  - e. No leakage of sodium to the atmosphere is allowed.
  - f. No leakage of air to sodium is allowed.
2. The samples to be handled are in accordance with the following:
  - a. Dimensions:  
1/32 x 3/16 x 3 inches long with a longitudinal crimp in the center as described in subsection III-B.
  - b. Accessibility:  
One end of the sample protrudes from the sample holder flow channel, approximately 1/8-inch to provide opportunity for gripping.
  - c. Material:
    - (1) Stainless Steel Type 316
    - (2) Alloy Steel Type 2 1/4Cr-1Mo

3. Sample Handling Sequence

Three samples are to be removed, one at a time at different intervals.

Each sample removal will include the following sequence of events:

- a. Removal of flow channel.
- b. Removal from sodium system.
- c. Insertion into a gas tight container for transportation to the analytical laboratory.
- d. Handling must be of such a nature that no sample material is lost.

4. Design Requirements

a. Materials:

Materials contacting sodium shall be the same as the loop material, i.e., one type 316 stainless steel and one 2 $\frac{1}{4}$ Cr-1Mo.

b. Design Strength:

Any portion of the handling system that represents a permanently connected extension of the sodium system must be designed for a pressure of 100 psig and a temperature of 1300 F.

c. Space Restrictions:

A 10-inch diameter space is available at each sample holder and the space above each sample holder is clear.

d. Safety Considerations:

The system design must be such that it can be operated safely without undue risk of fire or other hazards.

## APPENDIX E

### PRESSURE DROP CALCULATIONS

This appendix contains the pressure drop calculations and analysis data for the six sodium mass transfer test loops covered in the design report. The calculations herein are those which have been revised from the original calculations and are based on the number one loop operation since its installation in the test building. The calculations presented, differ from the original calculations due to modifications in the sample insert pattern (additional sample capacity).



## APPENDIX E

### PRESSURE DROP CALCULATIONS

June 8, 1961

Prepared by A. G. Silvester

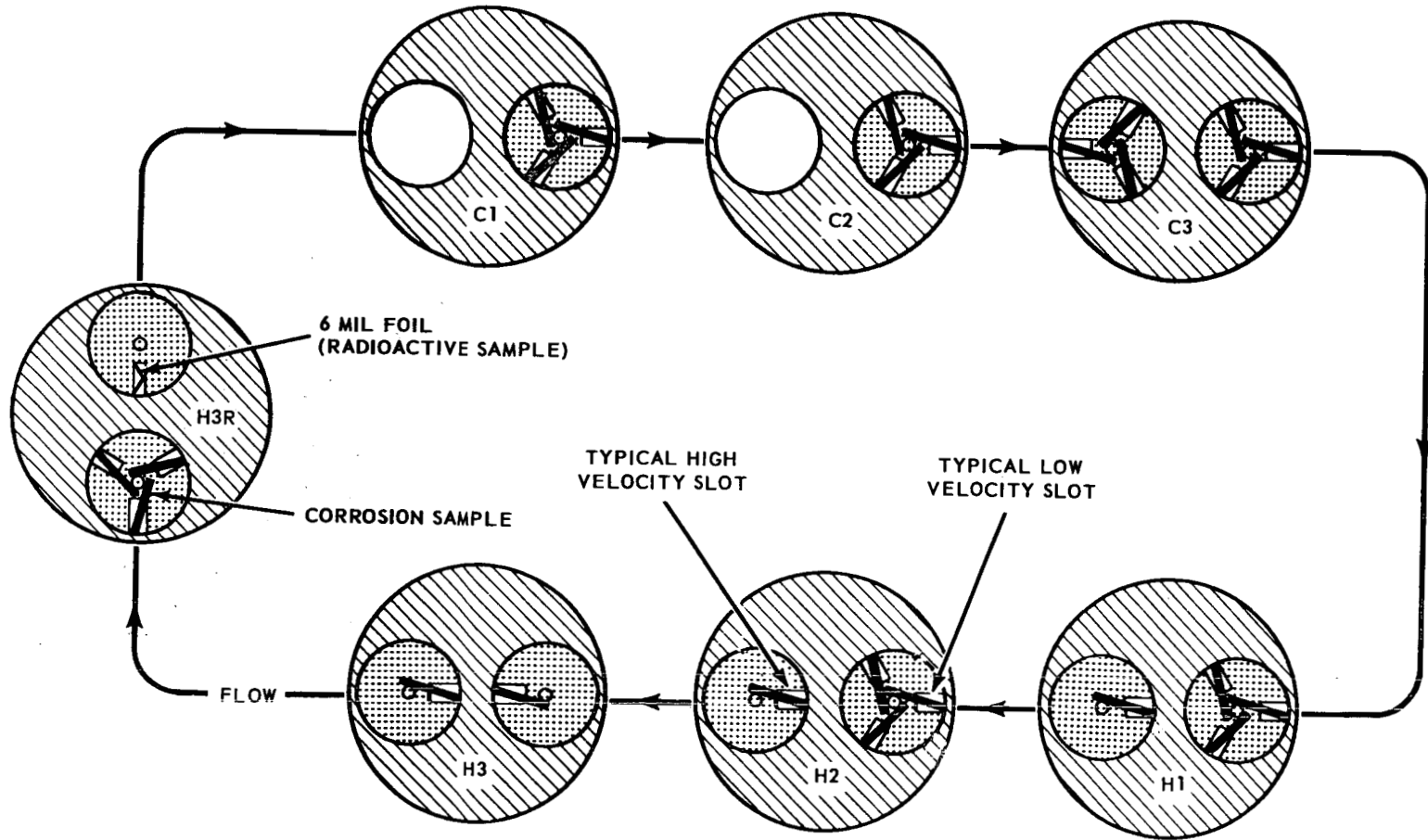
#### INTRODUCTION

The six geometrically identical sodium mass transfer loops, which were installed at General Electric Company, Atomic Power Equipment Department during the summer of 1960 have been analyzed for pressure drop, or required pump head, using standard pressure drop equations and constants for flow of liquids.

At the time of analysis, only loop number one had been in operation for a sufficient length of time to render test data to compare with the pressure drop calculations. It is believed however, that all six loops will have a nearly identical pressure drop. This belief is based on the fact that all loops were fabricated from identical drawings and all machined parts were manufactured within shop tolerances.

Each sodium mass transfer loop consists of seven specially designed sample holders, an electromagnetic pump, a dump tank, a surge tank, heaters, coolers, and interconnecting piping. The major sources of pressure loss are the sample holders and the piping. At the design flow rate of 1 gpm, the sum of the heater, cooler and piping losses are calculated to be about  $\frac{1}{4}$  psi compared to a loop  $\Delta P$  of over 100 psi. These losses, therefore, were considered negligible and have been omitted from this analysis. Thus, all loop pressure drop calculations were based on the flow resistance of the seven sample holders.

The pressure drop in each holder depends on the number of parallel flow passages in each insert and the number of samples installed. Figure E-1 shows the standard insert and sample loading pattern for each loop. All coolant slots measure 0.115-inch x 0.180-inch except the H3R high velocity foil slot which is 0.062-inch x 0.187-inch. The length of each slot is



SAMPLES: (25) REGULAR + (1) 6 MIL FOIL

FIGURE E-1 SAMPLE LOADING PATTERN

2-15/16-inches. The sample inserted into all slots except H3R high velocity foil slot is 0.062-inches thick, 0.170-inches wide and 3-inches long. The H3R sample (irradiated) size is 0.006-inch thick, 0.216-inch wide and 3-inches long and is crimped into "V" shape. Following is a discussion of the primary flow resistance considerations.

1. Entrance and Exit Losses

Twenty-five abrupt changes in velocity occur in a circuit of the test loop. The majority of these sudden changes also involve a change in direction either at the same point or close enough to the point of change to make the analysis difficult. This is the area in which the greatest lack of confidence exists in the results of these calculations. The expansion losses are expressed in the form of "velocity change squared divided by 2 g", while entrance and turning losses are calculated by applying an empirical factor to the velocity head formula. The product of these factors can result in an answer that may vary as much as plus or minus 50%. Since these combined losses represent one-half of the hot leg sample holder losses and 32% of the cold leg losses, these pressure drop evaluations can be quite in error.

The slope of the resulting pressure drop vs. flow curves also may be slightly in error since the factors change with the effective direction angle. For example, the cross-over losses at the top of the sample holder include two 90-degree turns, an expansion and a contraction loss, and friction loss between the two sample slot cylinders (inserts). At very low velocities, the change in flow direction can be made gradually with a low percentage of losses going into eddy currents, entrainment of surrounding fluid, etc. As the velocity increases centrifugal forces become greater, and an increasing percentage of losses occur due to impingement and lateral dispersion of the main stream after striking the underside of the sample holder cover. For this reason, one would expect that there should be a change in the K or multiplying factor as velocity changes in this region.

2. Turbulent - Laminar Flow

The flow in the sample holder slots at 700 F is turbulent (that is, where the Reynolds number is greater than 4000) under all conditions above a flow of 0.3 gpm. The high velocity slots do not reach the transition zone until the flow is reduced to 0.144 gpm. With no samples in these slots, the critical transition zone is obtained at about 0.25 gpm. The critical Reynolds number for flow transition is thought to be conservative since: 1) transition conditions are frequently reported as low as 2000, and 2) since the physical properties of sodium are a function of temperature, slightly lower flow rates are required for transition conditions at operating temperature.

3. Pressure Drop Due to Friction

Moody friction factors were used in the standard hydraulic friction equations, but there is an opportunity to vary this factor by as much as two to one for the same Reynolds number depending on the roughness factor chosen. Crane Technical Paper No. 409 - May, 1942 however, includes an additional allowance for an increase of apparent roughness factor when hydraulic diameters are very small, i.e., in the region below 0.30-inch diameter. For a typical case such as the high velocity slots, where the hydraulic diameter is 0.04-inch, Crane suggests that the roughness factor should be greater by 37%, which would result in a constant value of 0.053 for friction factor independent of Reynolds number up to  $10^7$ . This would result in pressure drops being about twice as high as calculated with the standard equation. It is probably true that the actual roughness factor should be greater than that for a smooth brass tube, when hydraulic diameters are very small, and where a scale deposit may have built up on some of the surfaces. Therefore, the calculated friction pressure drops in this report should be considered as the lowest probable value, and may be 15 to 30% higher in actual practice.

4. Deposition Plugging of Slots

The flow area of the sample slots with samples installed becomes zero when 23 mils of deposit have accumulated uniformly on the surface of the flow area. The actual plugging point may be reached before 23 mils

have been deposited, if the deposit thickness is not uniform. Velocity variation across the passage may possibly affect the uniformity of the deposit. As the remaining flow passage becomes smaller the chances of sudden plugging by particulate matter also becomes greater.

5. Temperature Effects

The effects of temperature on the physical dimensions of the sample holders, and in the density and viscosity of the sodium was considered. The sample holder slots and samples were machined to size, at room temperature in the shop, to a tolerance of  $\pm 0.003$ -inch. When the loop is heated, the flow passages within a sample holder will increase in area and the pressure drop would be less than calculated when room temperature measurements are used. To estimate the amount of pressure drop change to be expected when the sample holders are heated, the  $\Delta P$  in sample holder H3 was calculated. It has a slot length change of 0.002-inch and a width change of 0.0013-inch for a temperature rise of 1130 F (room temperature to 1200 F). The sample length and width will change 0.00192 and 0.0007-inch, respectively. The resultant flow area increase is 0.00022-inch<sup>2</sup> which reduces the pressure drop by only 3%. From this, one may conclude that heated sample holders will operate with 2 to 3% less pressure drop than calculated. This change is negligible since it is also within the error that might be caused by tolerance limits on the holder and sample dimensions.

The ratio between sodium velocity at a higher temperature and at 700 F at the same mass flow rate through the same passage, is given by the density ratio as follows:

Temperature, F	700	800	900	1000	1100	1200	1300
Viscosity, lb/ft-hr	0.71	0.65	0.59	0.55	0.52	0.48	0.45
Sodium Density, $\rho$ , lb/ft <sup>3</sup>	53.8	53.0	52.2	51.3	50.4	49.5	48.6
Velocity Ratio $V_T/V_{700}$	1.000	1.015	1.030	1.050	1.068	1.088	1.106

### PRESSURE DROP ANALYSIS

Standard hydraulic pressure drop equations and coefficients were used to calculate the pressure drop, or required pump head, for the test loops. As previously described, the significant head loss in each of the six loops occurs in the corrosion sample region within the seven sample holders. For this analysis therefore, only the drop within the sample holders will be considered.

Geometrical and test conditions for this evaluation were obtained from other sections of this report - particularly in section III (Design of Loop Components) - and the drawings presented in Appendix F. The physical properties of sodium and the hydraulic coefficients and formulae were obtained from one of the following references:

1. Liquid Metals Handbook - Sodium NaK Supplement, TID 5277, AEC - Dept. of Navy - US Government Printing Office, 1955.
2. Heat Transmission - McAdams.
3. Introduction to Heat Transfer - Brown and Marco.
4. Crane Technical Paper No. 409 - May, 1942.

The flow meter calibration and pump head data is obtained under isothermal conditions, but since the loops are operated with a temperature differential, the pressure drops in the sample holders of the test loop were calculated for each of the following conditions:

1. Flow measured at a temperature of 700 F and sample holders at the operating temperature indicated below.

<u>SAMPLE HOLDER</u>	<u>TEMPERATURE</u>
C3 (and flowmeter)	700 F
C2, H1	1,000 F
C1, H2	1,100 F
H3, H3R	1,200 F

This was done both with, and without samples installed.

2. Loop Isothermal at 700 F - with and without samples.
3. Assuming varying amounts of depletion of the hot leg samples and slots and deposition on the cold leg samples and slots. Sample holder and flow meter temperatures were the same as condition 1 above.

Within each sample holder the pressure drop was evaluated at the following locations:

- |                                     |                                 |
|-------------------------------------|---------------------------------|
| 1. Sample holder entrance           | 5. and 9. Second slot entrance  |
| 2. First slot friction              | 6. and 10. Second slot friction |
| 3. First slot exit                  | 7. and 11. Second slot exit     |
| 4. Cross-over loss between passages | 8. Sample holder exit           |

Figure E-2 shows the location of the individual  $\Delta P$  evaluations for the hot leg sample holders. Numbers in the previous list correspond with those on the figure and with the following equations and results. Similarly Figure E-3 describes the location of the pressure drop analyses for the cold leg sample holders. The equations and coefficients used to evaluate the pressure drop at these locations are listed below.

1. Sample Holder (insert) Entrance.

$$\Delta P = K \frac{V^2}{2g} \left( \frac{\rho}{144} \right)$$

2. Slot Friction Loss

$$\Delta P = f (L/D_H) \frac{V^2}{2g} \left( \frac{\rho}{144} \right)$$

3. Slot Exit and Turn Losses.

$$\Delta P = \left[ \frac{(\Delta V)^2 + V^2}{2g} \right] \frac{\rho}{144}$$

4. Cross-Over Loss

$$\Delta P = f \frac{L}{D_H} \left( \frac{V_c^2}{2g} \right) \frac{\rho}{144}$$

5. Second Slot Entrance

Same\* as location 1.

6. Second Slot Friction Loss

Same\* as location 2.

Where:

$\Delta P$  = pressure drop, psi

$\rho$  = Na density, lb/ft<sup>3</sup>

$V$  = velocity in slot, fps

$g$  = acceleration of gravity  
= 32.2 ft/sec<sup>2</sup>

$K$  = entrance coefficient  
0.50 for single sample  
0.47 for triple sample

$f$  = friction factor (Moody)

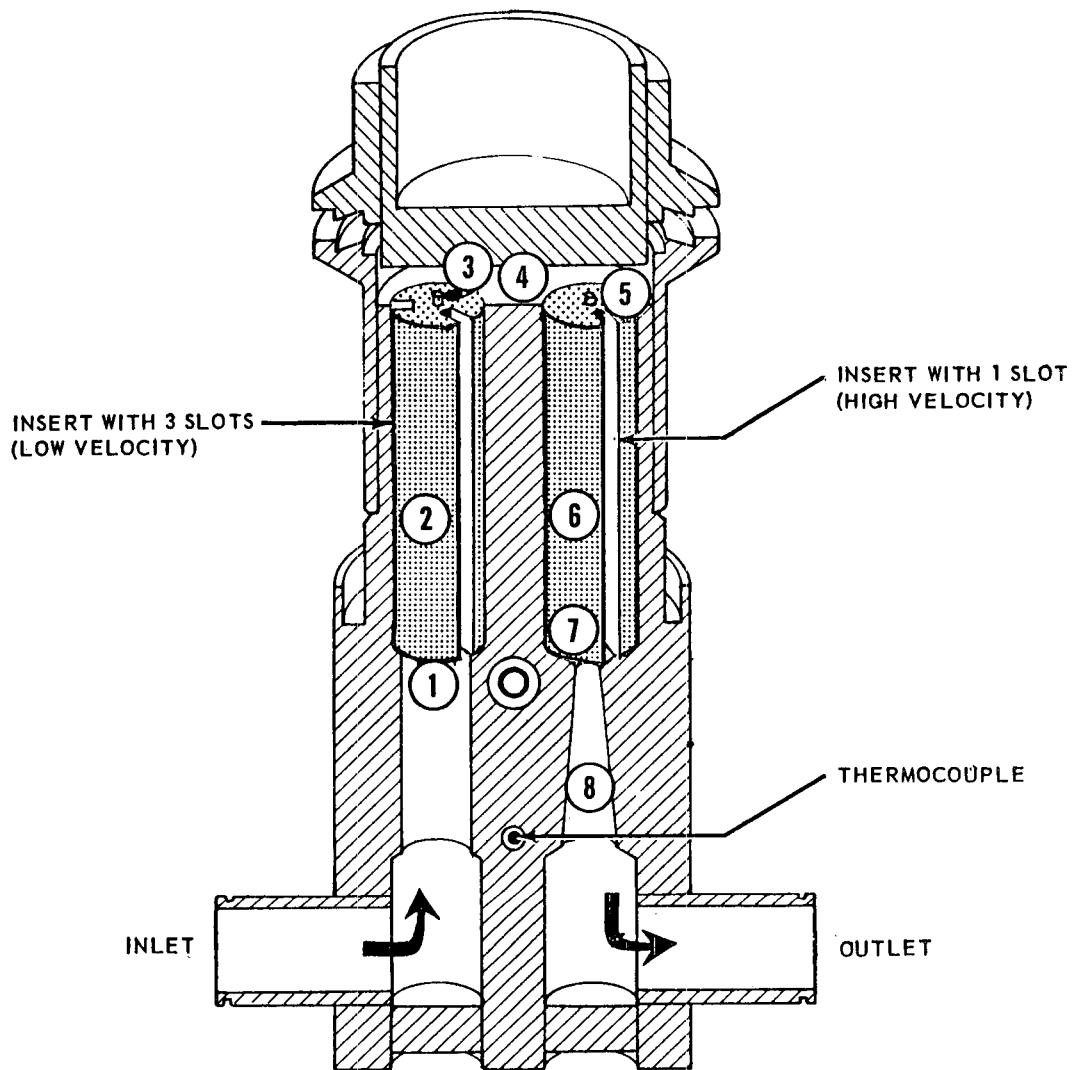
$L$  = length of slot

$D_H$  = hydraulic diameter  
=  $\frac{4 (\text{Flow Area})}{(\text{Wetted Perimeter})}$

$\Delta V$  = change in velocity  
=  $V - V_c$

$V_c$  = Cross-over velocity

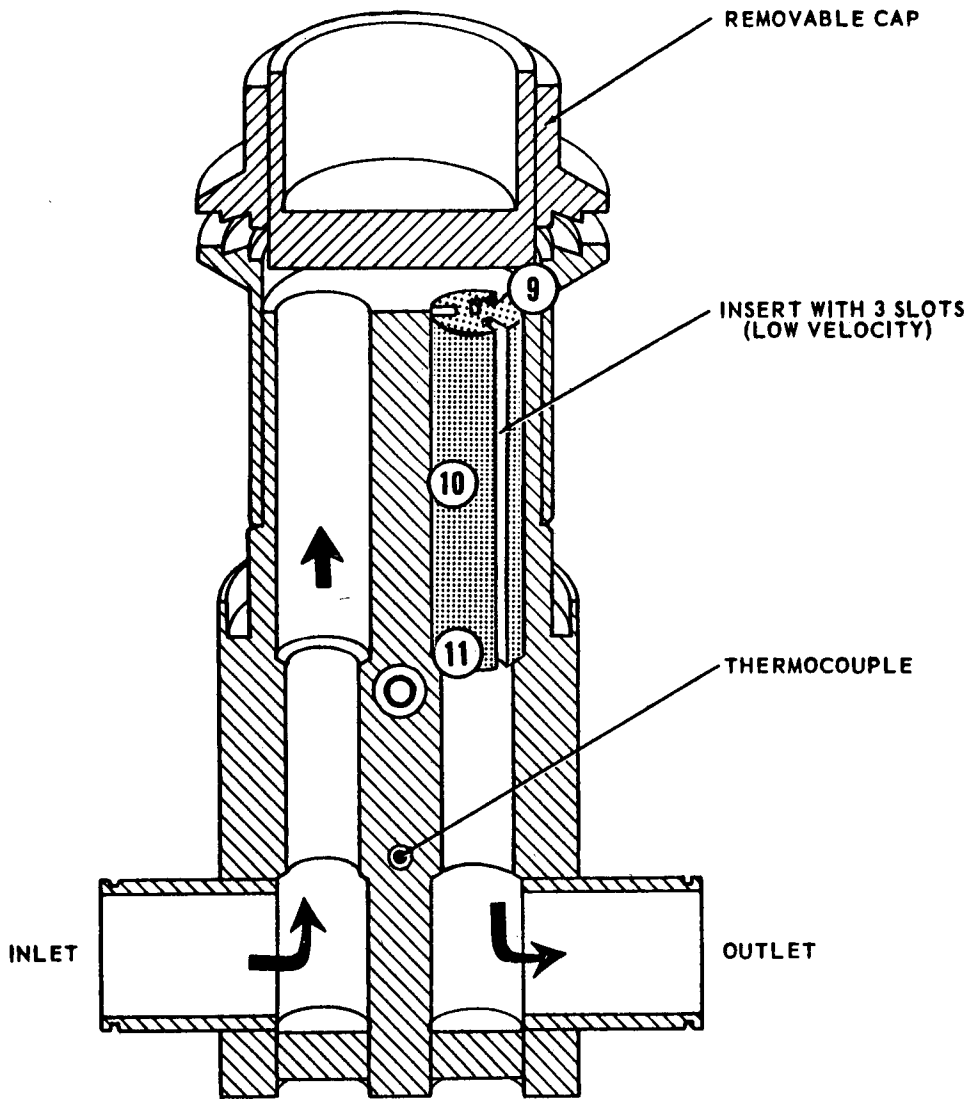
$V_x$  = vel. in transition, fps



276-6

FIGURE E-2 HOT LEG SAMPLE HOLDER - PRESSURE DROP LOCATIONS





276-36

FIGURE E-3 COLD LEG SAMPLE HOLDER - PRESSURE DROP LOCATIONS

7. Second Slot Exit Loss

$$\Delta P = \frac{V^2}{2g} \left( \frac{\rho}{144} \right)$$

$\Delta V_x$  = change in velocity;  
 $= V - V_x$

$V_d$  = average velocity in  
diffuser, fps

8. Hot leg, Second Slot Diffuser Losses.

$$\Delta P = \left[ \frac{(\Delta V_x)^2 + V_x^2 + f (L/D_H) V_d^2}{2g} \right] \frac{\rho}{144}$$

9. Cold Leg Slot Entrance.

Same\* as location 1.

10. Cold Leg Slot Friction Loss.

Same\* as location 2.

11. Cold Leg Slot Exit Loss.

Same\* as location 7.

-----  
\* "Same" indicates identical equation but velocities and coefficients are evaluated for local conditions.

Flow areas and velocities used in the evaluations are itemized below:

<u>Location</u>	<u>Flow Area</u> in <sup>2</sup>	<u>Velocity</u> (@ T & 1 gpm) fps
One slot - with sample	0.0207	31.4
without sample	0.0420	15.5
Foil slot - with sample	0.0210	31.0
without sample	0.0235	27.6
Three slots - with sample	0.062	10.5
without sample	0.125	5.2
Cross-over (average)	0.141	4.6
Transition preceding Diffuser	0.0342	19.0
Diffuser (average)	0.0482	13.5

## RESULTS

The results of evaluating the loop pressure drop at five flow rates under the first two (temperature) conditions and at one flow rate for the third condition (sample loss or deposit) described above are presented in both tabular and graphical form. The tables provide representative numerical results and the figures provide graphical representation of all the evaluations made. The following itemized list of conditions and corresponding tables and figure numbers is provided for more rapid identification.

<u>Analysis Condition</u>	<u>Condition Number</u>	<u>Table Number</u>	<u>Figure Number</u>
Individual Sample holder $\Delta P$ (0.5 gpm)	1	E1	E-4 to E-7 and E-9
Loop $\Delta P$ at operating conditions (5 flow rates)	1	E2	E-4
Same without samples	1	E3	E-5
Isothermal Loop $\Delta P$ @ 700 F (5 flow rates)	2	E4	E-6
Same without samples	2	E5	E-7
Hot leg depletion; cold leg deposits (0.5 gpm)	3	E6	E-9

Table E1 provides a breakdown of the pressure drops within each of the seven sample holders at 0.5 gpm flow and at operating temperature.

TABLE E1

SAMPLE HOLDER PRESSURE DROP - SAMPLES INSTALLED - 0.5 gpm FLOW

<u>H1 Sample Holder - 1000 F</u>		<u>H2 Sample Holder - 1100 F</u>	
Entrance(1)	0.085 psi	Entrance(1)	0.091 psi
First slot friction(2)	0.500	First slot friction(2)	0.510
Second slot entrance(5)	0.320	Second slot entrance(5)	0.319
Second slot friction(6)	3.460	Second slot friction(6)	3.480
Second slot exit(7)	0.980	Second slot exit(7)	0.980
Exit(8)	<u>2.620</u>	Exit(8)	<u>2.620</u>
H1 TOTAL	7.965 psi	H2 TOTAL	8.000 psi

NOTE: First slot exit, and cross-over were negligible for both holders.

<u>H3 Sample Holder - 1200 F</u>		<u>H3R Sample Holder - 1200 F</u>	
Entrance(1)	0.810 psi	Entrance(1)	0.085 psi
First slot friction(2)	3.500	First slot friction(2)	0.500
First slot exit(3)	0.520	First slot exit(3)	0.320
Cross-over(4)	1.200	Cross-over(4)-(Negligible)	-
Second slot entrance(5)	0.600	Second slot entrance(5)	0.940
Second slot friction(6)	3.500	Second slot friction(6)	3.420
Second slot exit(7)	0.240	Second slot exit(7)	0.220
Exit(8)	<u>2.360</u>	Exit(8)	<u>2.300</u>
H3 TOTAL	12.730 psi	H3R TOTAL	7.785 psi

<u>C1 Sample Holder - 1100 F</u>		<u>C2 Sample Holder - 1000 F</u>	
Entrance(9)	0.090 psi	Entrance(9)	0.090 psi
Second slot friction(10)	0.510	Second slot friction(10)	0.500
Exit(11)	<u>0.140</u>	Exit(11)	<u>0.140</u>
C1 TOTAL	0.740 psi	C2 TOTAL	0.730 psi

<u>C3 Sample Holder - 700 F</u>	
Entrance(1)	0.080 psi
First slot friction(2)	0.478
Cross-over(3,4,5)	0.334
Second slot friction(10)	0.478
Exit(11)	<u>0.110</u>
C3 TOTAL	1.480 psi

NOTE: The potential pressure drops not itemized in C1, C2, and C3 were negligible for this flow.

TOTAL LOOP ΔP - 39.43 psi

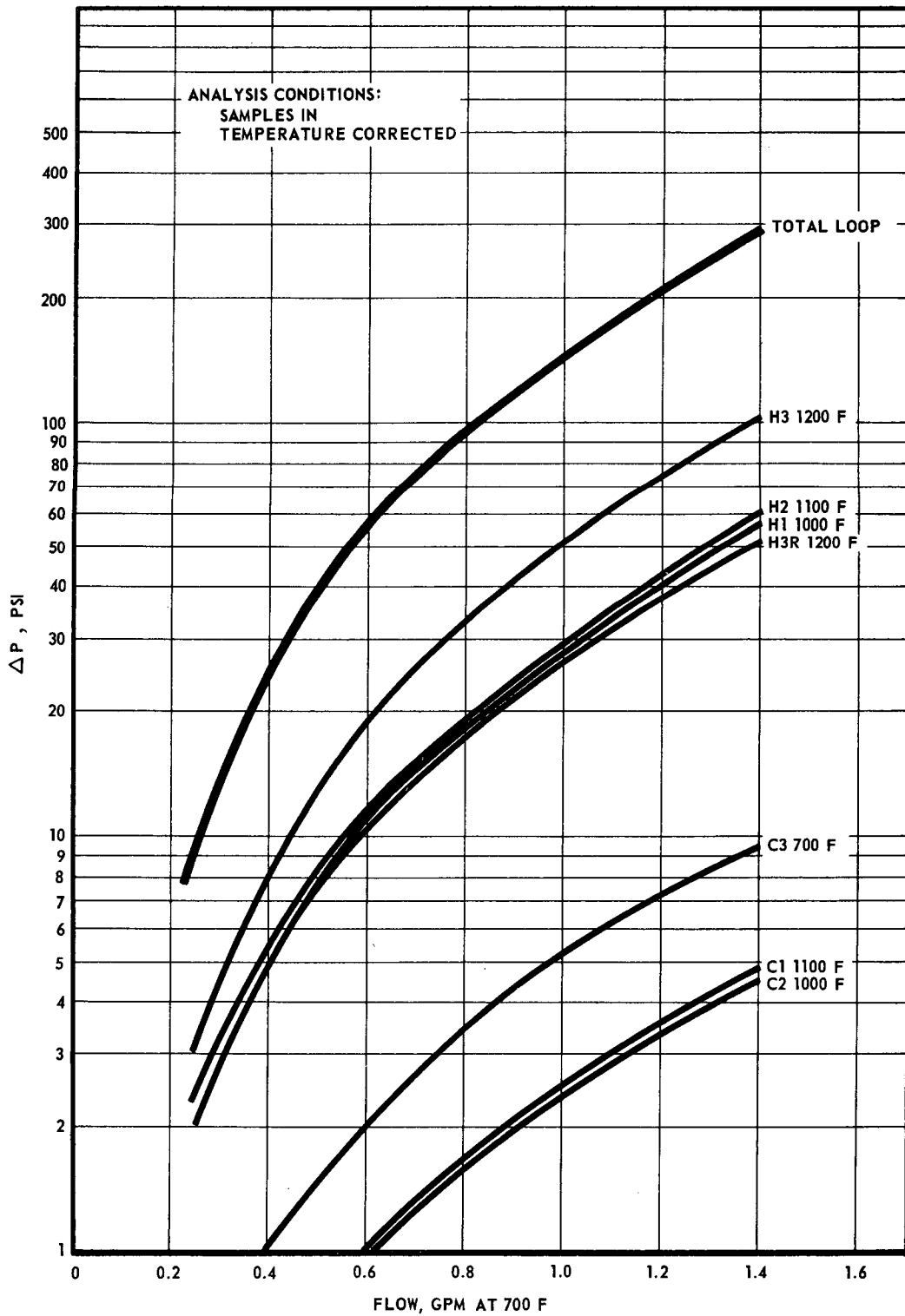


FIGURE E-4 CALCULATED LOOP PRESSURE DROP

276-4

TABLE E2

PRESSURE DROP WHEN FLOW IS MEASURED AT 700 F  
AND SAMPLE HOLDERS ARE AT OPERATING TEMPERATURES

<u>FLOW, gpm</u>	<u>0.3</u>	<u>0.5</u>	<u>0.7</u>	<u>1.0</u>	<u>1.3</u>
<u>Sample Holder</u>					
H1 - 1000 F	2.96	7.96	14.72	28.30	50.40
H2 - 1100 F	3.02	8.00	15.00	28.80	51.20
H3 - 1200 F	4.40	12.73	25.00	51.00	87.00
H3R - 1200 F	2.85	7.79	13.60	26.62	44.00
C1 - 1100 F	----	0.74	1.29	2.49	4.03
C2 - 1000 F	----	0.73	1.27	2.45	3.96
C3 - 700 F	<u>0.49</u>	<u>1.48</u>	<u>2.65</u>	<u>5.28</u>	<u>8.40</u>
TOTAL LOOP $\Delta P$ , psi	13.72	39.43	73.53	144.94	248.99

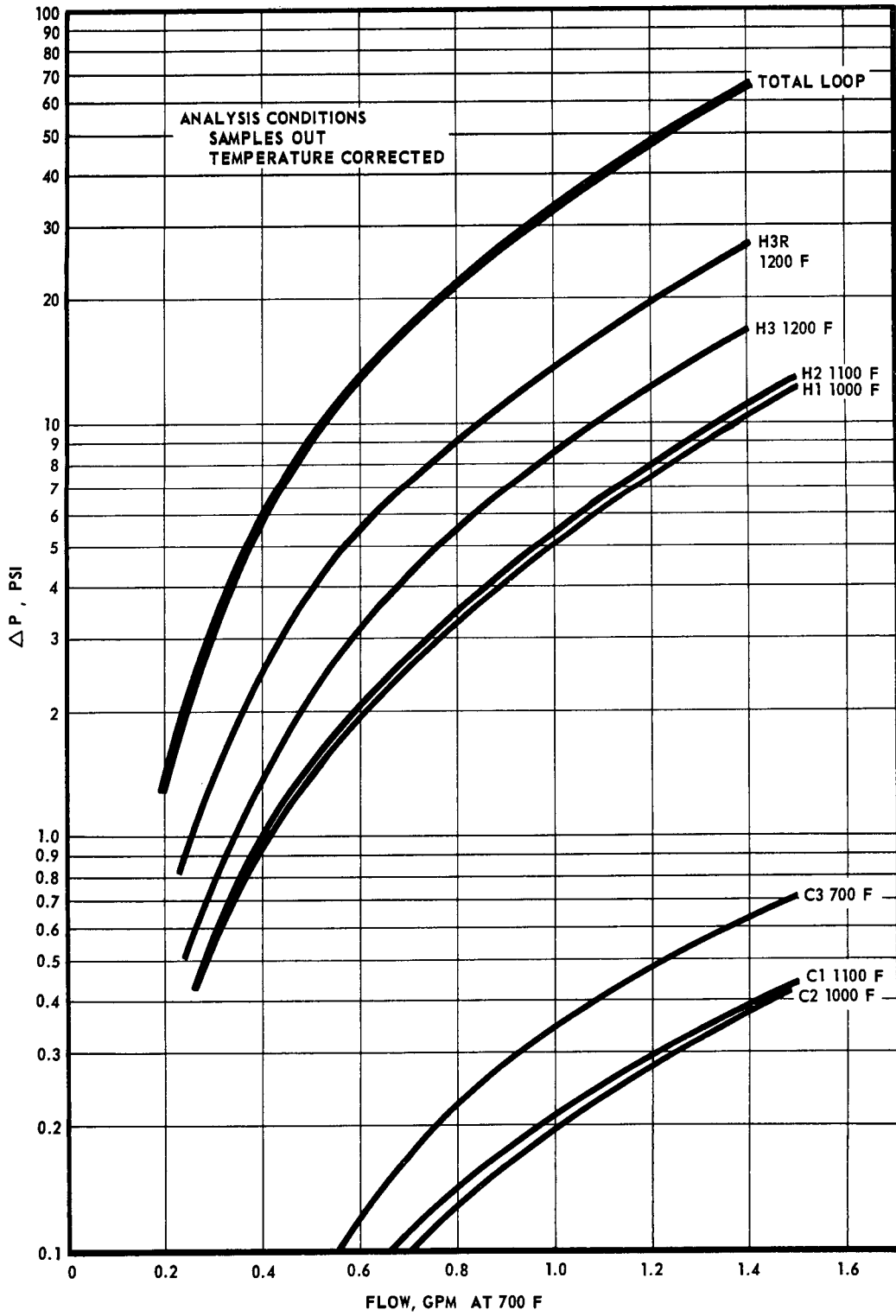
The calculations of pressure drops through the loop with the samples removed from the sample holders are tabulated and presented in Table E3. The calculations were made under the same flow and temperature conditions as those for "samples installed". Refer to Figure E-5 for plotted curves on "samples removed" conditions.

TABLE E3

SAME AS TABLE E2 EXCEPT SAMPLES ARE REMOVED

<u>FLOW, gpm</u>	<u>0.3</u>	<u>0.5</u>	<u>0.7</u>	<u>1.0</u>	<u>1.3</u>
<u>Sample Holder</u>					
H1 - 1000 F	0.54	1.44	2.70	5.15	9.10
H2 - 1100 F	0.56	1.47	2.75	5.24	9.30
H3 - 1200 F	0.78	2.16	4.25	8.66	14.30
H3R - 1200 F	1.39	4.04	7.10	13.93	23.00
C1 - 1100 F	0.02	0.06	0.11	0.21	0.34
C2 - 1000 F	0.02	0.05	0.10	0.20	0.33
C3 - 700 F	<u>0.03</u>	<u>0.10</u>	<u>0.17</u>	<u>0.34</u>	<u>0.55</u>
TOTAL LOOP $\Delta P$ , psi	3.34	9.32	17.18	33.73	56.92

With the entire loop isothermal at 700 F, the pressure drops were calculated by applying a velocity (density) correction to the pressure drops given in Tables E2 and E3. These calculations are tabulated in Table E4 and plotted in Figure E-6 for "samples installed" conditions and Table E5 and Figure E-7 for "samples removed" conditions.



276-2

FIGURE E-5 CALCULATED PRESSURE DROP WITHOUT SAMPLES

TABLE E4

LOOP ISOTHERMAL AT 700 F -- SAMPLES INSTALLED

<u>FLOW, gpm</u>	<u>0.3</u>	<u>0.5</u>	<u>0.7</u>	<u>1.0</u>	<u>1.3</u>
<u>Sample Holder</u>					
H1	2.82	7.57	14.02	26.95	47.90
H2	2.82	7.57	14.02	26.95	47.90
H3	4.04	11.70	22.95	46.90	80.00
H3R	2.62	7.25	12.50	24.45	40.40
C1	----	0.69	1.21	2.33	3.77
C2	----	0.69	1.21	2.33	3.77
C3	<u>0.49</u>	<u>1.48</u>	<u>2.65</u>	<u>5.28</u>	<u>8.40</u>
TOTAL LOOP ΔP, psi	12.79	36.95	68.56	135.19	232.14

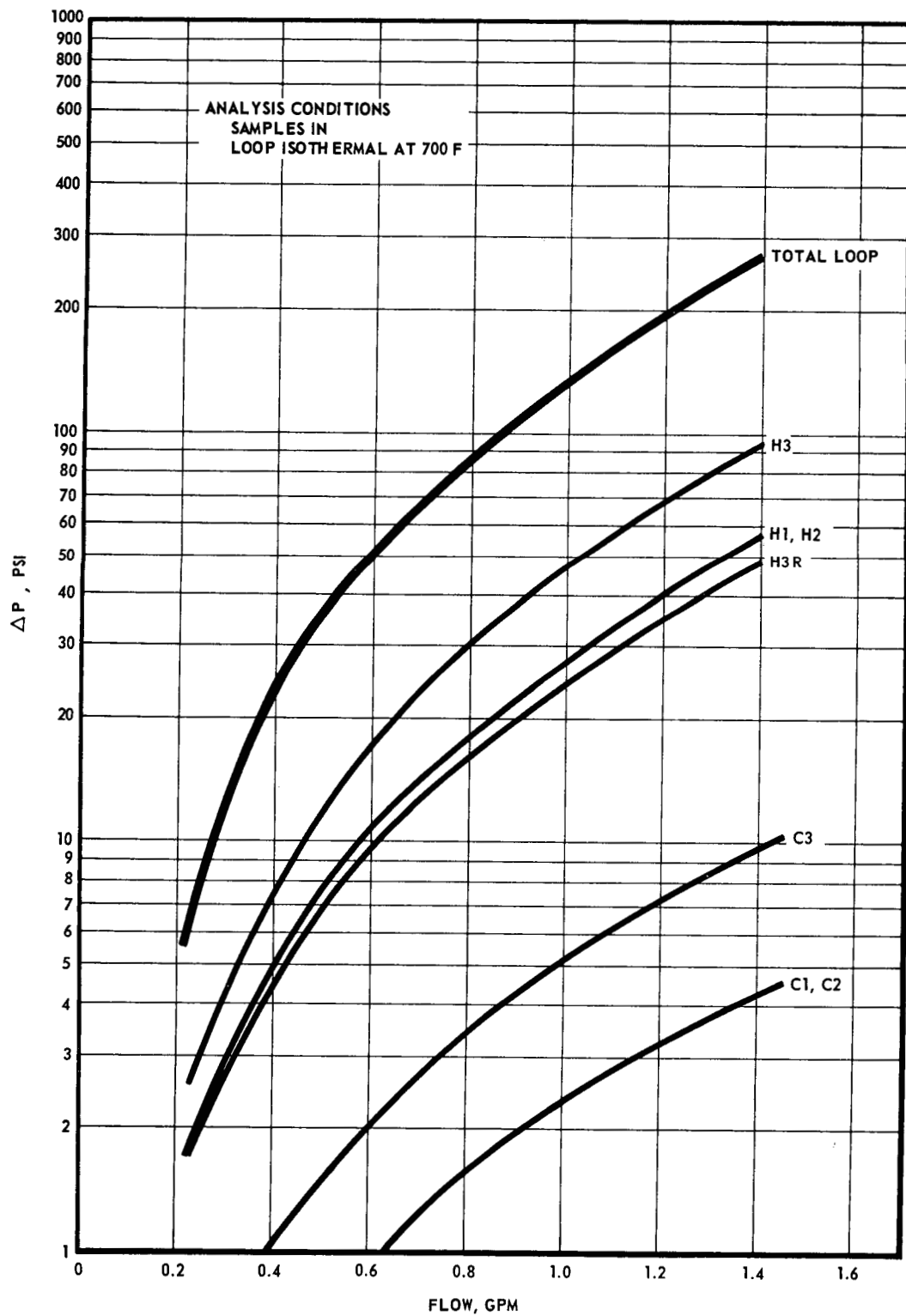
TABLE E5

LOOP ISOTHERMAL AT 700 F -- SAMPLES REMOVED

<u>FLOW, gpm</u>	<u>0.3</u>	<u>0.5</u>	<u>0.7</u>	<u>1.0</u>	<u>1.3</u>
<u>Sample Holder</u>					
H1	0.52	1.37	2.57	4.90	8.70
H2	0.52	1.37	2.57	4.90	8.70
H3	0.72	1.99	3.90	7.96	13.15
H3R	1.28	3.72	6.53	12.80	21.15
C1	0.02	0.06	0.10	0.20	0.31
C2	0.02	0.05	0.10	0.20	0.31
C3	<u>0.03</u>	<u>0.10</u>	<u>0.17</u>	<u>0.34</u>	<u>0.55</u>
TOTAL LOOP ΔP, psi	3.11	8.66	15.94	31.30	52.87

To calculate the effect of a depletion (corrosion) of the walls of the hot leg sample holders lots and samples, and/or the deposition of material on the walls of the cold leg sample holder slots and samples, the pressure drops were recalculated with modified dimensions. It was assumed that the depletion or deposition was of uniform thickness around the perimeter of the flow passage. The results are shown in Table E6 and plotted on Figure E-8. Figure E-9 shows the progressive plugging of a cold leg sample.





276-1

FIGURE E-6 CALCULATED ISOTHERMAL LOOP PRESSURE DROP

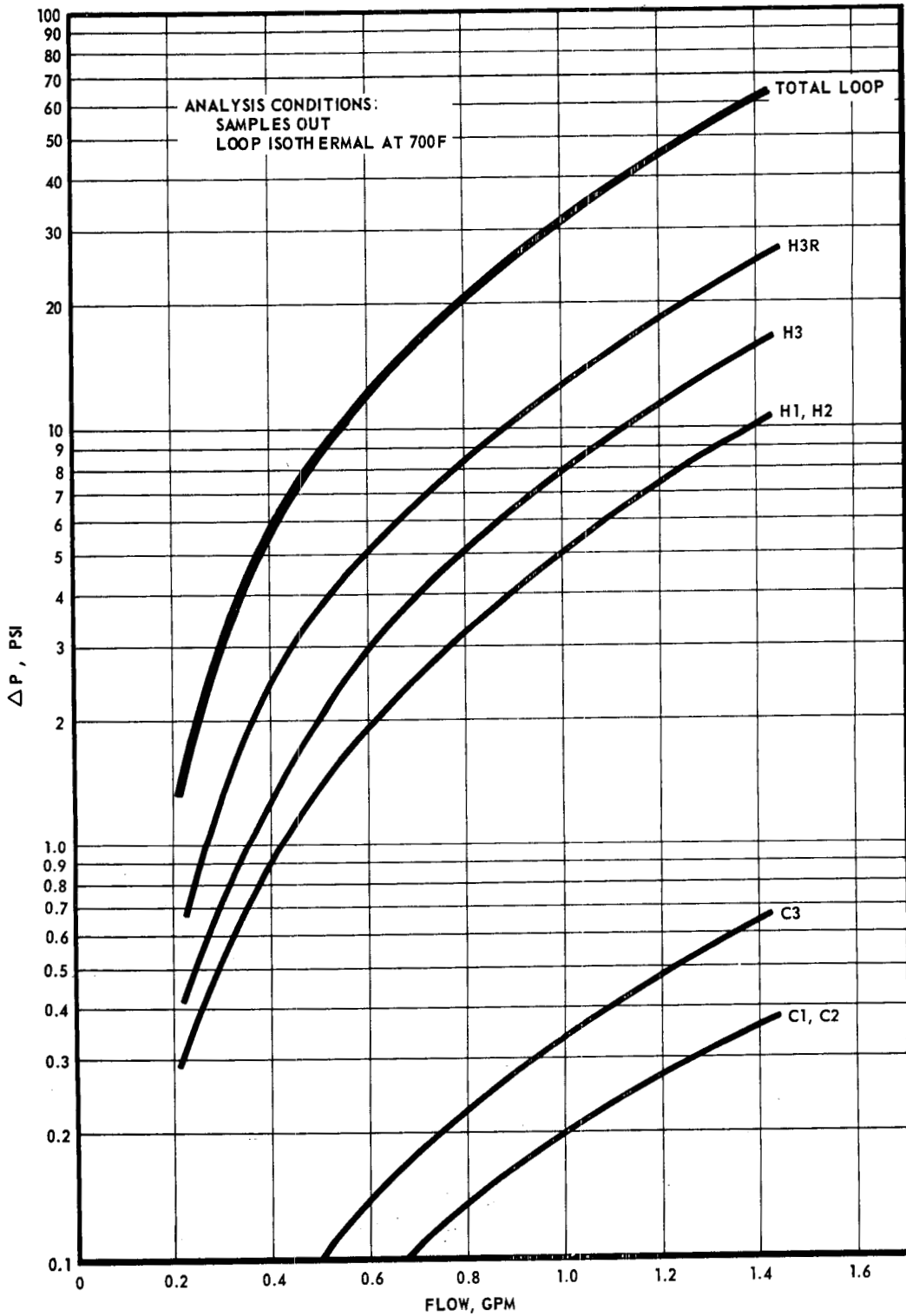


FIGURE E-7 CALCULATED ISOTHERMAL PRESSURE DROP WITHOUT SAMPLES

276-3

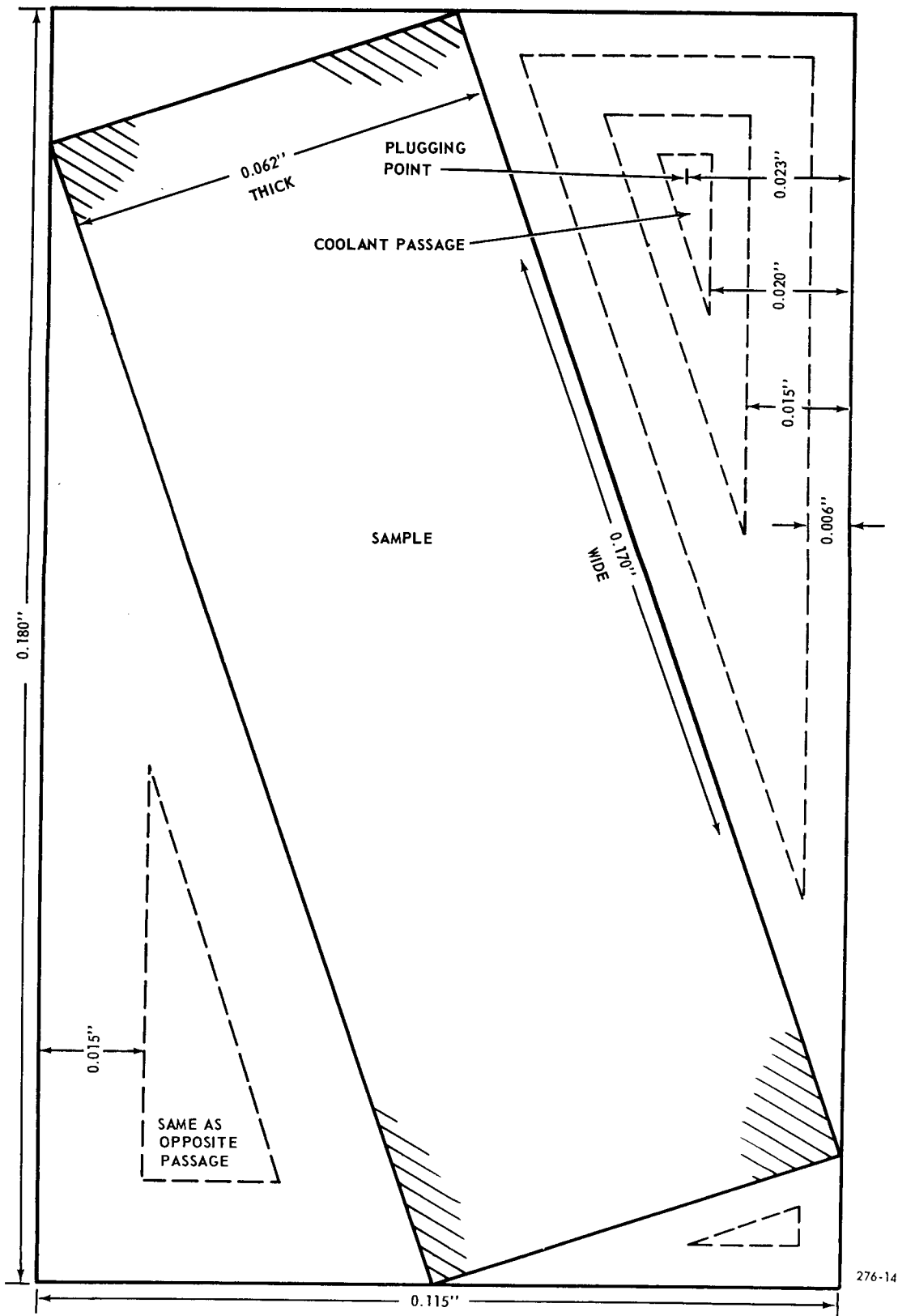


FIGURE E-8 PLUGGING OF A COOLANT PASSAGE

TABLE E6

PRESSURE DROP (psi) @ 0.5 gpm AS A FUNCTION OF MILS DEPLETION OF HOT LEG  
AND DEPOSITION ON COLD LEG SURFACES

<u>MILS CHANGE</u>	<u>0</u>	<u>1</u>	<u>3</u>	<u>6</u>	<u>10</u>	<u>15</u>	<u>20</u>	<u>31</u>
<u>Sample Holder</u>								
H1 - 1000 F	7.965	5.890	4.092	2.657	---	---	0.992	0.92
H2 - 1100 F	8.000	6.175	4.269	2.817	---	---	1.068	1.00
H3 - 1200 F	12.730	10.770	7.280	4.762	---	---	1.429	1.19
H3R - 1200 F	7.785	6.290	3.620	2.841	---	---	1.44	1.13
C1 - 1100 F	0.740	1.055	2.002	3.232	12.66	111.20	---	---
C2 - 1000 F	0.730	1.008	1.915	3.087	11.31	99.36	---	---
C3 - 700 F	1.480	2.035	3.801	6.113	19.71	173.30	---	---

Note that in the case of the cold leg samples C1, C2 and C3 the flow passages become plugged when 23 mils deposit has been made, and the pressure drop will have increased to infinity. For the hot leg samples, all of the samples will have been lost when 31 mils has been removed, however, the pressure drop will not change significantly after approximately 20 mils have been removed (and there is still a thin sample left) because the difference in hydraulic diameter is not controlling when the flow passage has already become so large.

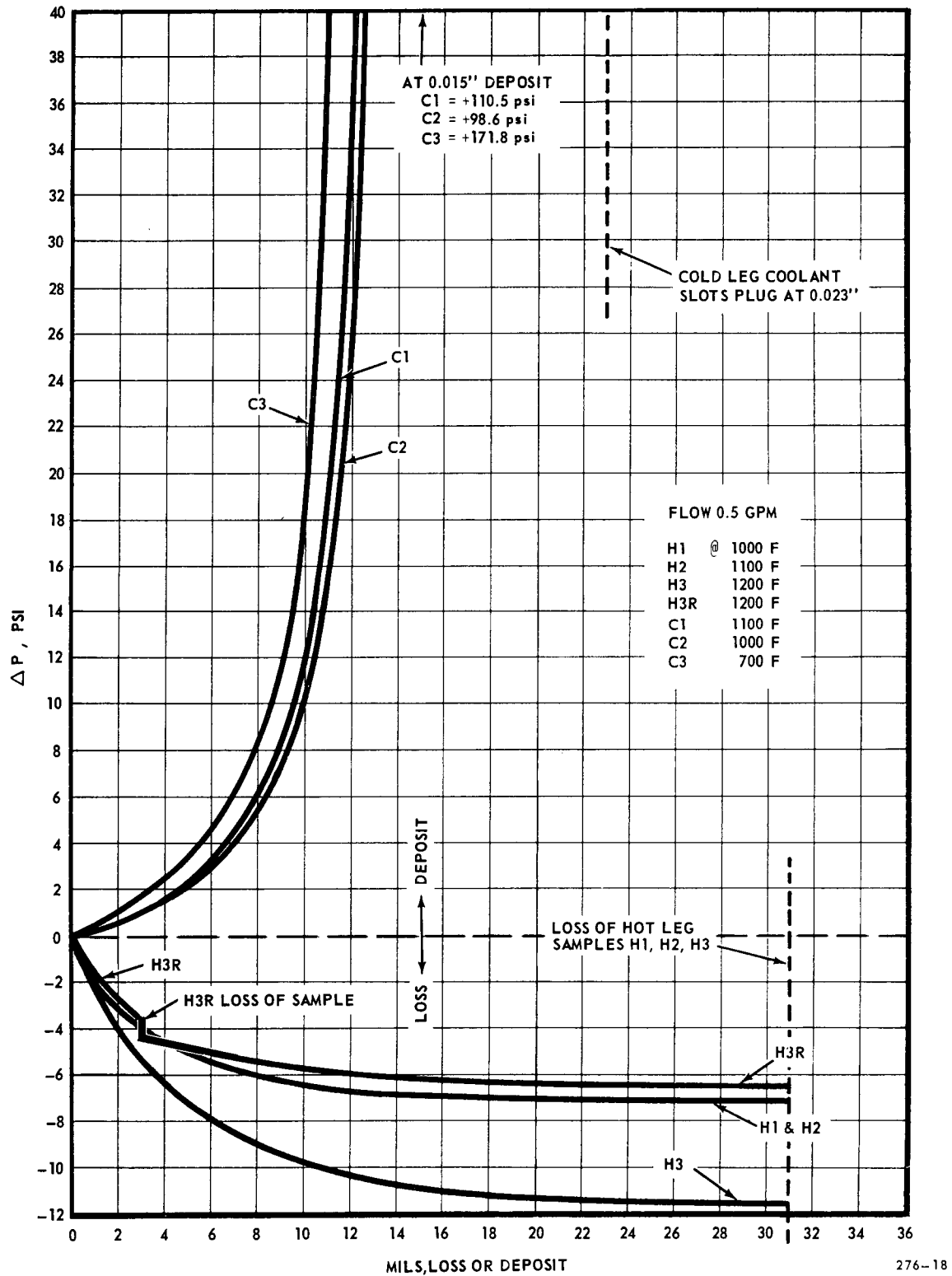


FIGURE E-9 CHANGE IN SAMPLE PRESSURE DROP vs SAMPLE LOSS FROM HOT LEG AND DEPOSIT ON COLD LEG

APPENDIX F

REFERENCE DRAWINGS

This appendix contains a list (see parts lists) of all General Electric Company drawings that were required for the fabrication and assembly of the six Sodium Mass Transfer Test Loops. Reproductions of a few of the more pertinent or representative drawings are presented herein. All inquiries concerning the drawings should be directed to the General Electric Company.

The drawings and parts lists tabulated below are presented in this appendix:

<u>DRAWING NUMBER</u>	<u>TITLE</u>
932C218 and PL	Test Facility Layout
*197E782 and PL	Test Loop Assembly
197E796	Elementary Diagram
197E797	Elementary Diagram
585D389	Heater (300 F $\Delta T$ )
693C849	Heater (100 F $\Delta T$ )
612D573	Sample Holder (H1 and H2)
612D706	Sample Holder (Details)
932C133	Sample Holder (H3)
932C134	Sample Holder (H3R)
932C135	Sample Holder (C1, C2, C3)
5534-1 (Allied Engineering and Production Corp.)	Vacuum Dry Box

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\* This drawing is representative of all six test loops insofar as design and configuration is concerned. The basic differences in the six loops are the materials of construction.

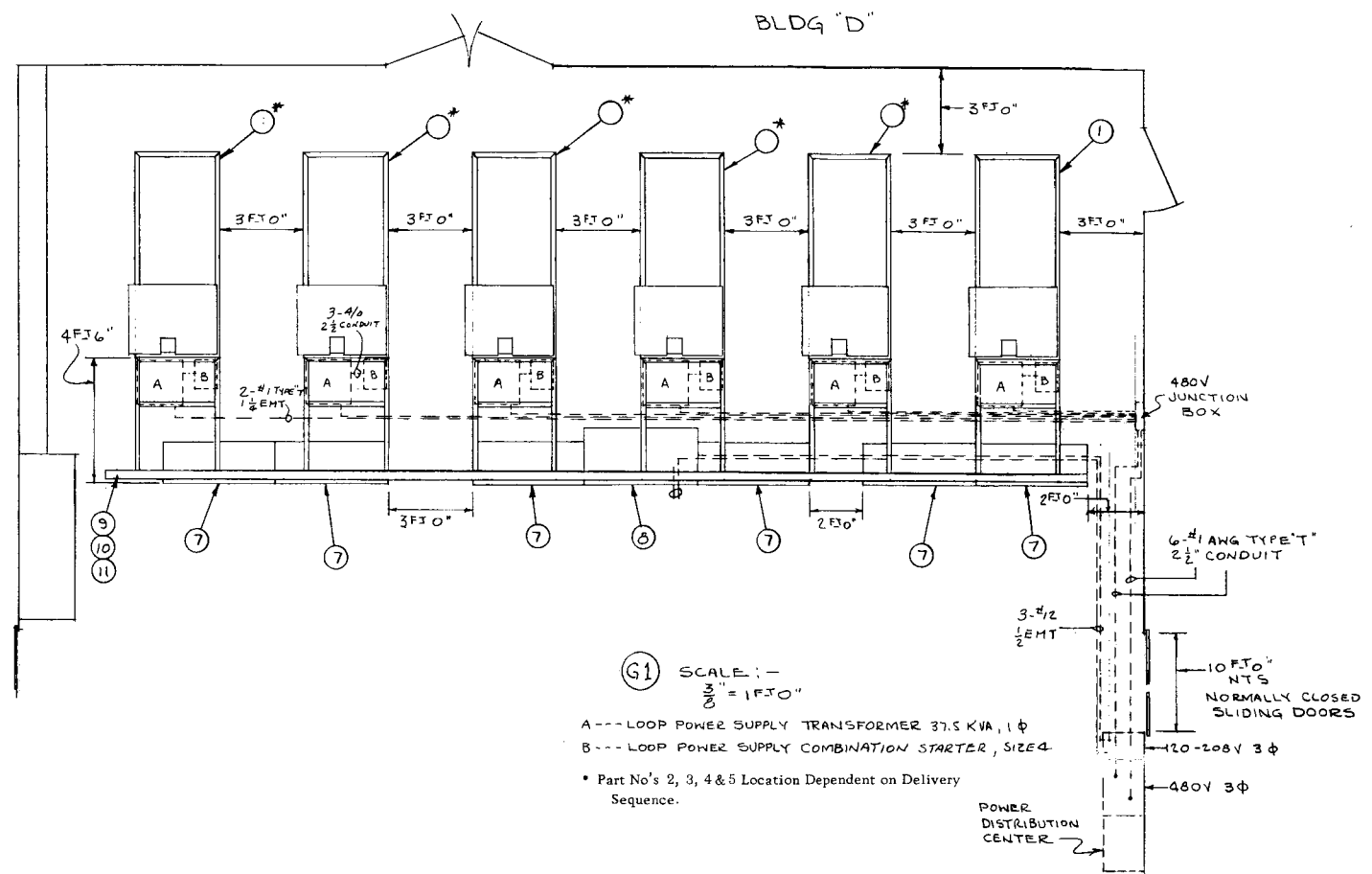
932C218  
CONT. OF SHEET  
SHEET NO.

UNLESS OTHERWISE SPECIFIED USE THE FOLLOWING:			
APPLIED PRACTICES	SURFACES	TOLERANCES ON DIMENSIONS	
✓	✓	FRACTIONS	DECIMALS
		±	±
		±	±
		±	±
		±	±

TITLE  
**Na MASS TRANSFER TEST FACILITY LAYOUT**  
 FIRST MADE FOR

PL ISSUED

188  
 A  
 B  
 C  
 D  
 E



(G1) SCALE: -  
 $\frac{3}{8}'' = 1 FT 0''$

A --- LOOP POWER SUPPLY TRANSFORMER 37.5 KVA, 1  $\phi$   
 B --- LOOP POWER SUPPLY COMBINATION STARTER, SIZE 4

• Part No's 2, 3, 4 & 5 Location Dependent on Delivery Sequence.

POWER DISTRIBUTION CENTER

REVISIONS	PRINTS TO

PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
1	1	Frame	197E742G1
2	X	Elementary Diagram	197E797
3	X	Flow Diagram	693C840
4 through 9		NOT USED	
10	1	E. M. Pump	585D519G4
11	1	Heater	585D389G1
12	2	Sample Holder	612D573G1
13	1	Sample Holder	932C133G1
14	1	Sample Holder	932C134G1
15	2	Fintube	693C843P6
16	3	Sample Holder	932C135G2
17	1	Cooler	693C843G2
18	2	Flow Meter	655C498G4
19	1	Surge Tank	932C111G2
20	1	Dump Tank	612D458G2
21	2	Fintube	885B548P2
22	1	Plugging Plate	885B549P2
23	1	Cold Trap	693C846G2



PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
24	2	Reducer	885B736P2
25	1	Reducer	885B763P2
26	2	Heater	693C849G1
27		NOT USED	
28	4	Liquid Metal Bellows Valve, 1½" Globe, 150# 1000 F Sodium per vendor drawing D-42390, 2¼% Cr, 1% Mo alloy steel body, Wm. Powell Co., Cincinnati, Ohio - or Equal	
29	AR	Filler Metal, 1/16 dia. Type 316SS Wire	
30	AR	Filler Metal, 1/16 dia. McKay 2Cr Mo or Equal	
31	AR	Filler Metal, 1/16 dia. Type 309 McKay 25-12 or Equal	
32	12	90° Elbow, ½" Sch. 40 ASTM A 234 Gr WP22	
33	4	Tee, ½" Sch. 40 ASTM A 234 Gr WP22	
34	2	Concentric Reducer, 1" x ½" Sch. 40 ASTM A 234 Gr WP22	
35	8	90° elbow, 1" Sch. 40 ASTM A 234 Gr WP22	
36	1	Tee, 1" Sch. 40 ASTM A 234 Gr WP22	
37	2	Socket Weld Reducer, ½" x ¼" Sch. 40 ASTM A 234 Gr WP22	

PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
38	1	Pipe, $\frac{1}{2}$ " Sch. 40 x 15', ASTM A 335 Gr P22	
39	1	Nipple, 1" Sch. 40 x 4", ASTM A 335 Gr P22	
40	1	Pipe, $\frac{1}{2}$ " Sch. 40 x 5', ASTM A 312 Gr TP316	
41	5	90° Elbow, $\frac{1}{2}$ " Sch. 40, ASTM A 403 Gr WP316	
42	1	90° Elbow, 1" Sch. 40, ASTM A 403 Gr WP316	
43	1	Concentric Reducer, 1" x $\frac{1}{2}$ " Sch. 40, ASTM A 403 Gr WP316	
44		NOT USED	
45	1	Gas Valve, $\frac{1}{4}$ Female, NPT, Hoke Inc. or Equal	Cat. #380M
46	2	Swagelok Male Connection, Crawford Fitting Co. or Equal - Cleveland, Ohio	Cat. #810-1-4
47	2	Swagelok Tee, Crawford Fitting Co. or Equal - Cleveland, Ohio	Cat. #810-3
48	1	Inert Gas Line, $\frac{1}{2}$ O.D. x 0.035 (20 GA) Wall x 30', Hard Drawn Copper Tube	
49		NOT USED	

PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
50	1	Blower, Aladdin E-X Size 9 ARGT 9 Disc-Down, 600 QFM against 3" S.P. Constant Speed 240 V, 60 cy. Motor Aladdin Heating Corp., San Leandro, Calif. - or Equal	
51	1	Auto-Damper Regulator, Brown Industrial Motor 115 V, 60 Cy. Minneapolis-Honeywell Co. - or Equal	Cat. #801D6K-98-60
52	1	Cooling Air Ducts	612D608G1
53	6	Dry Box Seal	885B559G1
54	1	Support	612D614G1
55	1	Support	612D614G2
56	6	Spring Supports	612D614G3
57	4	Cooler Cover	612D614P10
58	2	Cooler Cover	612D614P11
59		NOT USED	
60	1	Insulator	612D614P5
61	2	Bushing	612D614P6
62	1	Hanger	612D614P16
63	3	Spring Hanger, Fig247 Size 2, Grinnell Co. Cat. #54 or Equal	
64	2	Hanger Rod, Fig140 3/8 x 8" lg., Grinnell Co. Cat. #54 or Equal	

PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
65	1	Hanger Rod, Fig140 3/8 x 24" lg., Grinnell Co. Cat. #54 or Equal	
66	1	Eye Rod, Fig248 3/8 x 8" lg., Grinnell Co. Cat. #54 or Equal	
67	1	Hanger Rod, Fig140 3/8 x 14" lg., Grinnell Co. Cat. #54 or Equal	
68	60	Shielding Bricks, 2 x 4 x 8, cast lead	
69	AR	Brazing Alloy, 1/8 dia. Easy-Flo #3 or Equal	
70	AR	Flux, Hand Flux or Equal	
71	1	Bridge, See Detail	
72	1	Heater Protection Strip, 0.015 (28 GA) x 3" wide x 50', TP. 304SS Sh. 2B Fin.	
73 through 75		NOT USED	
76	2	Keystone Wireway, General Electric Supply Corp. or Equal	Cat. #KFW445
77	1	"U"Connector, General Electric Supply Corp. or Equal	Cat. #KFUC
78	1	Closing Plate, General Electric Supply Corp. or Equal	Cat. #KFCP
79		NOT USED	

PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
80	AR	Wire, 600 V Flamenol #18 Stranded AWG	SI-58175
81	AR	Wire, 600 V Flamenol #10 AWG (soft)	SI-58175
82	AR	Wire, 600 V Flamenol #12 AWG	SI-58175
83	AR	Wire, 600 V Flamenol #14 AWG	SI-58175
84 through 87		NOT USED	
88	33	Thermocouples	885B776G1
89	1	Heating Element	932C200P1
90	1	Heating Element	932C200P2
91	1	Heating Element	932C200P3
92	1	Heating Element	932C200P4
93	1	Heating Element	932C200P5
94	1	Heating Element	932C200P6
95	2	Heating Element	932C200P7
96	1	Heating Element	932C200P8
97	1	Heating Element	932C200P9
98	1	Heating Element	932C200P10
99	1	Heating Element	932C200P11
100	1	Heating Element	932C200P12

PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
101	7	Heating Element	932C200P13
102 through 104		NOT USED	
105	1	Heating Element	932C200P17
106 through 109		NOT USED	
110	1	Transformer, Type S1 - 1 cy. 240/480 120/240 V, 37½ KVA Hevi-duty Electric Co., Milwaukee, Wisconsin or Equal	Cat. # D-44880A
111	1	Combination Starter, NEMA Size 4 General Electric Co. or Equal	Cat. # CR7008F100A2AA
112 through 115		NOT USED	
116	12	Uniclamp, Uniclamp Inc., Glendale, Calif. or Equal	302-6
117	8	Uniclamp, Uniclamp Inc., Glendale, Calif. or Equal	302-8
118	14	Uniclamp, Uniclamp Inc., Glendale, Calif. or Equal	302-11
119	12	Uniclamp, Uniclamp Inc., Glendale, Calif. or Equal	302-17
120	3	Uniclamp, Uniclamp Inc., Glendale, Calif. or Equal	302-22

PARTS LIST

SODIUM TEST LOOP

Drawing Number 197E782

Item No.	Quantity Per Assembly	Nomenclature	Part Number Or Drawing Number
121	2	Screw Hex. Head, $\frac{1}{4}$ -20 x $\frac{3}{4}$ lg., Stl. Cad. Pl.	
122	2	Washer, $\frac{9}{32}$ I.D. x $\frac{3}{4}$ O.D. x $\frac{1}{16}$ Thk., Stl. Cad. Pl.	
123	3	Screw Hex. Head, $\frac{3}{8}$ -16 x $\frac{3}{4}$ lg., Stl. Cad. Pl.	
124	3	L. Washer for $\frac{3}{8}$ Screw, Stl. Cad. Pl.	
125	18	Screw Hex. Head, $\frac{1}{2}$ -13 x $1\frac{1}{4}$ lg., Stl. Cad. Pl.	
126	18	Nut Hex. $\frac{1}{2}$ -13 Std., Stl. Cad. Pl.	
127	18	L. Washer for $\frac{1}{2}$ Screw, Stl. Cad. Pl.	
128	5	Nut Hex. $\frac{3}{8}$ -16 Std., Stl. Cad. Pl.	