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MSG TEST REPORT - REMOVAL OF RESIDUAL SODIUM

I INTRODUCTION

As part of the company funded Liquid Metal Fast Breeder Reactor (LMFBR) program, Atomics International designed and fabricated a prototype sodium heated steam generator. This unit, termed the Modular Steam Generator (MSG), was tested by the Atomic Energy Commission (AEC) under Contract No. AT(04-3)-813 in the Liquid Metal Engineering Center's (LMEC) Sodium Component Test Installation (SCTI) facility. At the completion of the scheduled test program, the residual sodium was removed from the MSG by an alcohol cleaning process. This report presents the results of the cleaning operations, along with a description of the cleaning loop and other related activities.

II CLEANING PROCESS

The alcohol cleaning process used to remove residual sodium from the MSG was patterned after a draft of a standard titled "Use of Alcohol to Remove Sodium from Sodium Coated Items" (Attachment to "MSG Cleaning Instructions" Reference 1). This draft procedure gives a description of the process, equipment requirements, methods and procedures, material properties, safety requirements and quality assurance requirements. Various changes were made because of the characteristics of the item to be cleaned and because of the unavailability of certain cleaning materials. The draft standard has several optional steps in which the solvent is diluted with water and a water rinse is used. These steps were not used for MSG cleaning because:

1. The MSG is fabricated of 2 1/4 Cr - 1 Mo which is susceptible to oxidation.
2. Subsequent to cleaning, the MSG will be modified for testing in the Large Leak Test Rig (LLTR) requiring welding and post weld heat treatment. It was felt that the formation of hydroxides and the possibility of caustic stress corrosion was not sufficiently understood to warrant the risk of using water.

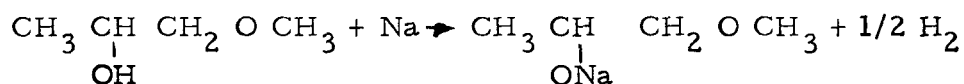
The basic process for removal of sodium from sodium coated items using alcohol consists of the following major steps:

1. 2-butoxyethanol flush to remove sodium

2. Ethanol flush to remove 2-butoxyethanol and reaction products
3. Dry residual ethanol after draining

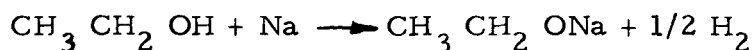
Attempts to procure the 2-butoxyethanol (Dowanol EB) in the quantities required were unsuccessful due to the current oil shortage. A substitute solvent was used after evaluation of several alternates. The substitute selected was propylene glycol methyl ether (Dowanol PM). Tests performed (Section IV) to determine the characteristics of this solvent indicated that the Dowanol PM was as good, if not better than the Dowanol EB. In addition, the material used for the ethanol rinse was found to be slightly different than the originally specified material. The originally specified ethanol was to contain 100 volumes of ethyl alcohol and 5 volumes of methyl alcohol. The actual procured commercially available ethanol used for cleaning was further modified with the addition of 10 volumes of isopropyl alcohol and one volume of methyl isobutyl ketone. Laboratory scale test indicated there was no discernible difference between the two types of ethanol.

The Dowanol PM was used to remove the bulk of the sodium from the MSG. The reaction of this glycol ether with the sodium is as follows:



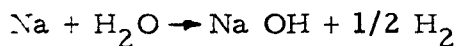
Sodium reacts with the glycol ether to form an organic sodium salt and free hydrogen. The Dowanol PM was selected because of its relative low reaction rate with sodium. This was important since there are areas of bulk sodium where excessive temperatures could occur. In addition, impurities such as oxides or carbonates were found to be more soluble in the Dowanol PM than in ethanol.

The ethanol was to be initially used as a flush to remove the residual Dowanol PM with the associated reaction products. Ethanol was selected for this step because of its relatively low cost and ease of drying (low boiling point). Ethanol also reacts with sodium at a somewhat faster rate than Dowanol. This reaction is as follows:



In this reaction sodium ethoxide and free hydrogen are formed. The ethanol was used, for an extended period of time, to remove residual sodium from crevice areas in the MSG, in addition to a final flush.

Since both the Dowanol and ethanol are hygroscopic, they contained a small amount of water. This water impurity in the solvents also reacts with the sodium to form sodium hydroxide and free hydrogen as follows:



The NaOH in turn reacts with the solvents and regenerates the water. The reaction rate of the solvents with sodium has been found to be a function of the water impurity concentration.

The reaction rate can be determined by measuring the rate of change of the sodium reaction product concentration by atomic absorption spectrophotometry or by measuring the hydrogen evolution rate. Both methods were used during MSG cleaning to track the reaction rates of the solvents with the sodium.

The final step in the cleaning was drying the residual ethanol after draining. This was done by heating to 150-180°F and purging with argon gas. The effluent was monitored until the ethanol concentration in the argon sparge gas was less than 300 ppm.

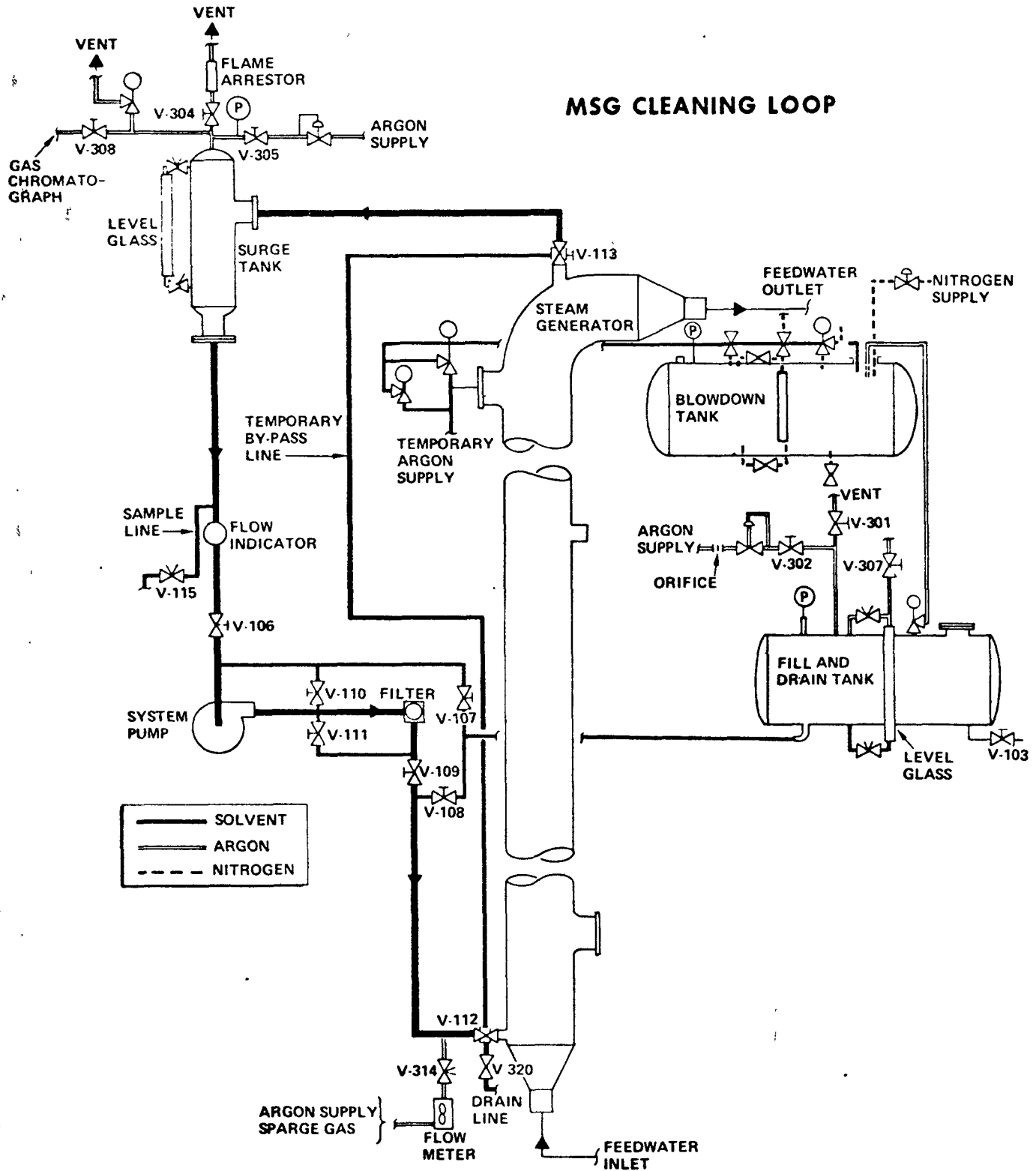
III LOOP DESCRIPTION

Cleaning of the MSG was performed in place at the SCTI. The sodium inlet and outlet lines were cut and capped and a cleaning loop connected to the MSG at the lower drain line and upper flowing vent line. The P & I drawing of the cleaning loop is shown in Figure III-1. The loop consisted of a fill and drain tank, blowdown tank, surge tank, circulating pump, filter, interconnecting piping and valves. In addition, various SCTI facility services were used such as the argon supply system, nitrogen supply system, feedwater system, and DAS (Data Acquisition System).

The fill and drain tank was a stainless steel tank 4 1/2 ft. in diameter and 12 ft. long. It was of sufficient size to hold the entire inventory of solvent. It had an argon pressurization system for filling the MSG and cleaning loop, and a pressure relief system to protect against over pressurization. A sight glass was also included to determine the liquid level in the tank. The blowdown tank was of similar size and material as the fill and drain tank. The discharge from the fill and drain tank and MSG pressure relief systems were routed to this tank. This tank was included in

Figure III-1

MSG CLEANING LOOP



the loop so that in the event of a pressure relief system actuation, the discharged solvent would be contained in a closed system. A nitrogen cover gas was maintained in the blowdown tank during all phases of cleaning. Both the fill and drain tank and blowdown tank were located at grade level (about 40 ft. above the drain line elevation).

The surge tank was a stainless steel tank 3 feet in diameter and 7 feet seven inches high. It was used to accommodate the expansion of the solvent during temperature changes and sparging operation, and also to disengage the argon sparge and hydrogen gas from the solvent during circulation. The surge tank contained a sight glass for solvent inventory control during filling and makeup. The tank was located at an elevation slightly above the MSG hockey stick centerline.

The circulating pump had a 5 HP motor with an estimated capacity of 75-100 gpm during normal circulation. A 10 micron filter was located at the pump discharge and was used to remove any particulate matter the cleaning fluids might pick up from the loop. The pump and filter were located at grade level.

The majority of the interconnecting piping was one inch SCH 10 304 stainless steel. All connections were of the socket weld type with the exception of several pump and tank flanged joints. Welding of connecting piping and fittings were performed in accordance with ANSI B31.1.0 1967. One-inch hand controlled ball valves were located at various positions as shown in Figure III-1.

An argon sparge gas supply was connected to the cleaning loop near the sodium drain line (solvent inlet line) of the MSG. This sparge gas was used to agitate the solvent during circulation to enhance cleaning. Another argon supply was located at the surge tank vent line. This inert gas supply was used to dilute the released hydrogen during the initial fill to a safe level before discharging to the atmosphere. A flame arrester was also located at the discharge as an added safety precaution, in addition to a pressure relief valve included in the event the vent line was inadvertently closed.

The SCTI feedwater system was operated during the MSG cleaning. Feedwater was circulated through the tube side of the MSG during all cleaning operations. The temperature of the alcohol or MSG could be controlled between ambient and 400°F with the feedwater system. The

SCTI DAS (Data Acquisition System) was also used to monitor and record all MSG internal thermocouple readings as well as feedwater temperatures.

A sample line was provided in the cleaning loop to obtain solvent samples for chemistry analysis. A gas chromatograph was also provided at the surge tank vent line to monitor the effluent hydrogen. This instrument was used primarily during initial fill to assure the hydrogen concentrating in the vent line was discharged at safe levels.

The primary flow path during solvent circulation was from the pump discharge, through the filter and into the MSG at the drain line. The solvent then flowed upward through the shell side of the MSG and out the flowing vent line to the surge tank. The solvent level in the surge tank was maintained below the line leading from the MSG to provide for disengagement of the sparge gas and solvent. From the surge tank, the solvent passed through a flow indicator to the pump suction. A temporary by-pass line was also provided for cleaning and checkout of the cleaning loop components prior to filling the MSG with solvent. After loop cleaning and checkout, the temporary by-pass line was removed for MSG cleaning. The valves shown in Figure III-1 were used for process control during steps such as purging the loop, filling, draining and filter replacement. Prior to adding solvent to the loop, all welds, valves and other components were helium leak checked.

IV EXPECTED RESULTS

Prior to initiation of actual MSG cleaning, laboratory scale tests were conducted to confirm the adequacy of the cleaning process and to determine various process parameters. From these tests, calculations were performed in order to uncover any potential problems that might occur during the cleaning steps. The following summarizes the results of these efforts.

A. Residual Sodium

A review of the MSG sodium side geometry was made to estimate the total amount of residual sodium that should be expected after draining.

Three sources of sodium were considered as follows:

1. Film remaining on exposed vertical surfaces.
2. Heel remaining between the lower tube sheet and drain line.
3. Sodium remaining in crevices that would not drain because of

surface tension effects.

For the sodium remaining on vertical surfaces, it was determined that the total sodium-side surface area was 2420 ft². Results of tests conducted in other AEC programs were used to estimate the expected sodium thickness that would adhere to a vertical surface after draining. The results of these experiments (Reference 2) are summarized below.

Sodium Drain Temp. (°F)	Surface Thickness	
	mg/cm ²	in.
400	2	.00088
600	1.3	.00058

It can be noted that the residual sodium film thickness can be reduced by increasing the sodium drain temperature. The normal MSG sodium drain temperature was 400°F, however, based on the above test results, it was decided to drain at 600°F (maximum temperature within the capabilities of SCTI). At this drain temperature and the exposed surface area, the total sodium mass remaining on vertical surfaces was estimated to be 6.4 lb.

A sodium heel remains at the lower end of the MSG because the drain line is above the elevation of the lower tube sheet. A schematic of the lower end geometry is shown in Figure IV-1. It can be noted that there are thermal baffles resting on top of the tube sheet, resulting in a small annular gap between the tubes and thermal baffles. Based on the total free volume below the lower opening of the drain line nozzle, the sodium remaining in the heel was estimated to be 12.9 lb.

For sodium remaining in crevices that would not be expected to drain because of surface tension effects, two major sources were identified. The first is in the region between the tubes and the tube spacers, and the second between the tie rods and spacers. A schematic of the geometries for these two regions is shown in Figure IV-2. Surface tension calculations indicated that all the sodium between the tubes and tube spacers would remain after draining. For the region between the tie rods and spacers, it was calculated that the sodium would drain to a level 1.3 inch above the tube spacers. Based on the above volumes, the sodium remaining in crevices was estimated to be 1.5 lb.

Based on the above analysis, the total sodium remaining in the MSG following the 600°F drain was estimated to be 20.8 lb. It should be noted

FIG IV-1
SCHEMATIC-LOWER END GEOMETRY

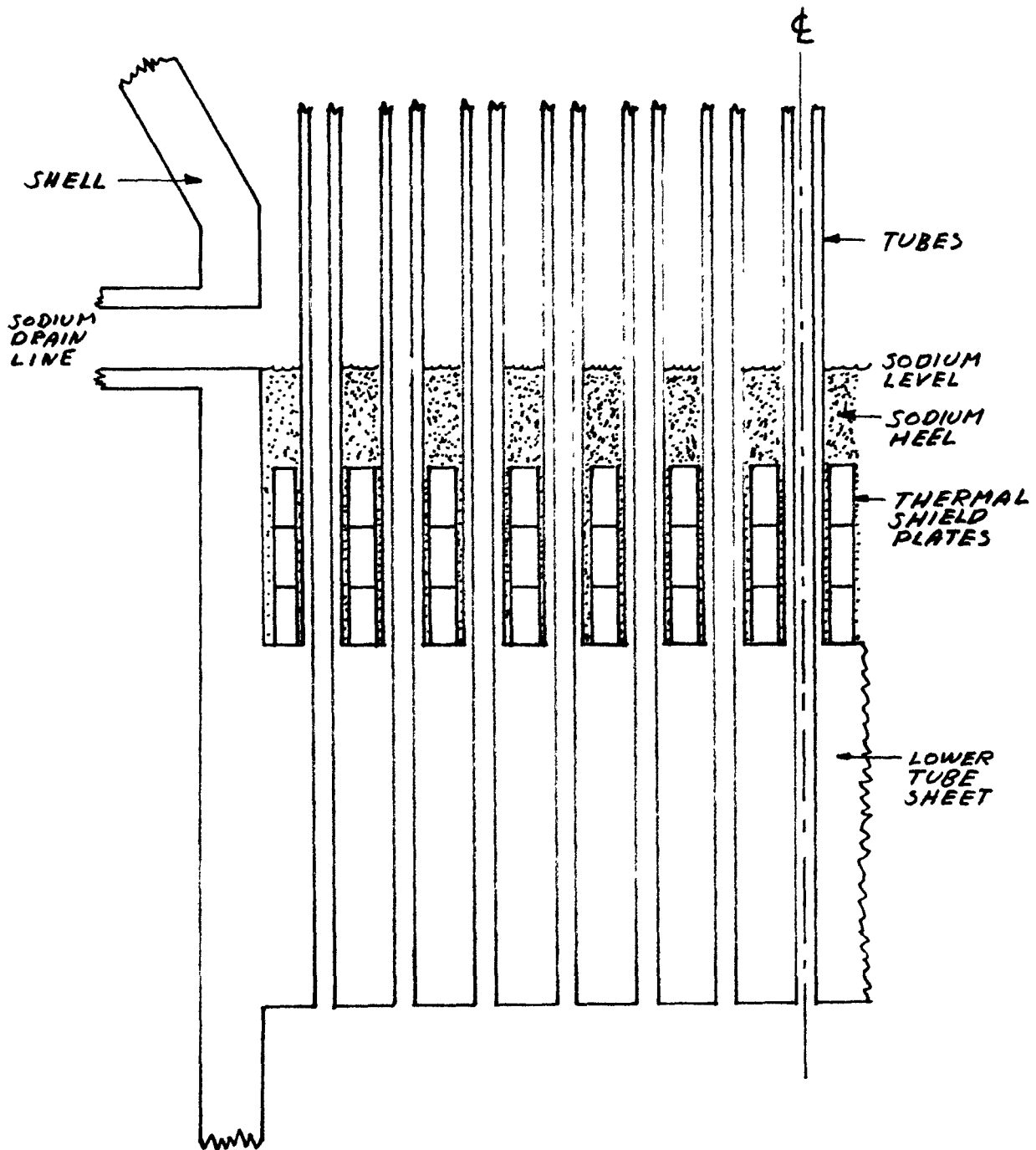
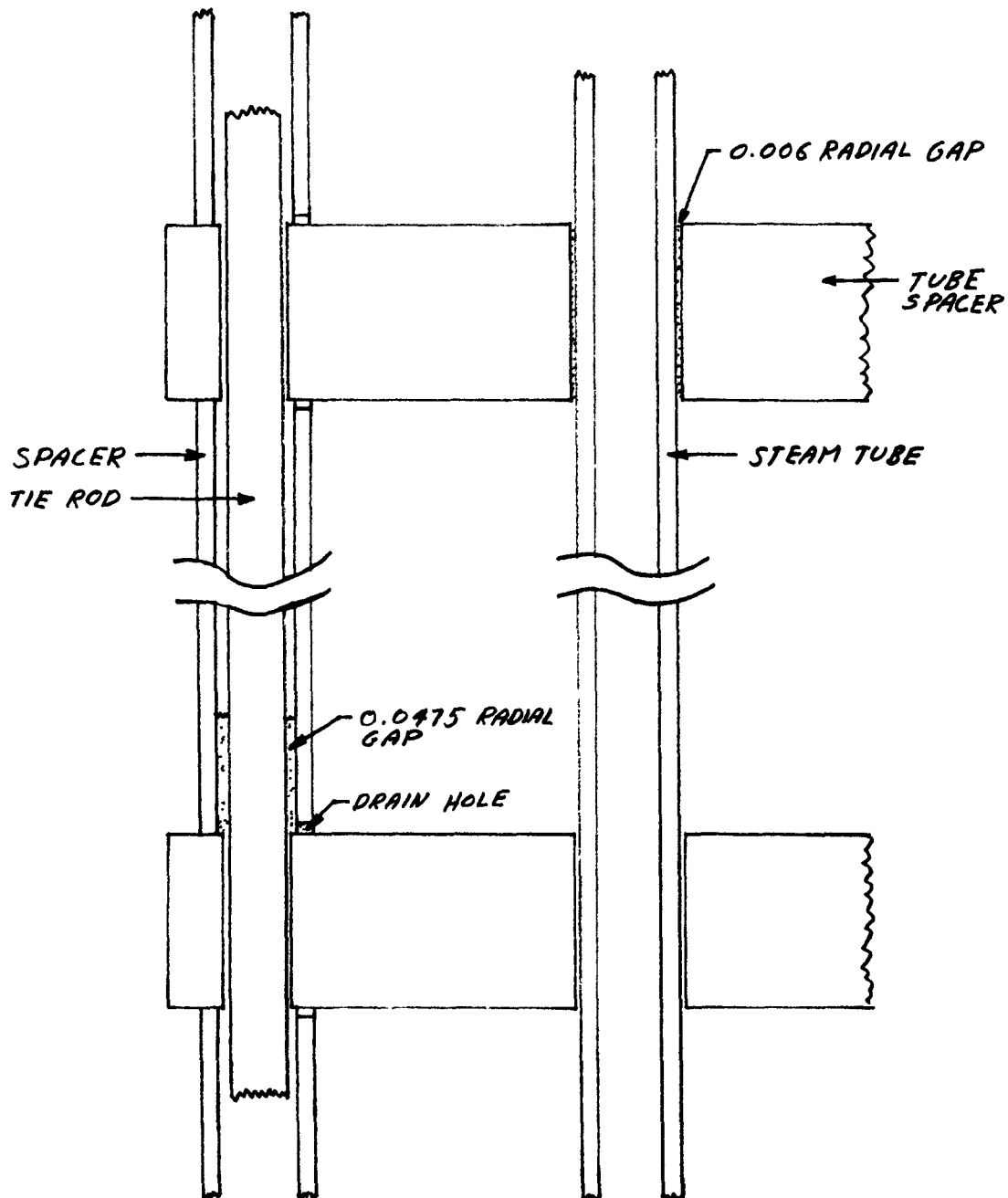


FIG. IV-2
SCHEMATIC - HOLDUP OF SODIUM IN CREVICES



that there can be a considerable tolerance in estimating the mass of sodium remaining on vertical surfaces and in crevices.

B. Reaction Tests

In order to evaluate possible problems of localized heating, several laboratory scale reaction tests were performed between the solvent and sodium. The initial test performed was to determine the approximate sodium heat of reaction. In this test a 2 1/2 gram cube of sodium sample was placed in a 200 ml volume of the solvent. The heat of reaction was determined by observing the temperature rise of the solvent. The results of these tests are as follows:

<u>Solvent</u>	<u>Heat of Reaction</u>
Dowanol EB	2800 Btu/Lb of Na
Dowanol PM	2100 Btu/Lb of Na

Since the temperature of the solvent varied during the tests (70-140°F), the above values are only approximate over the temperature range of interest for MSG cleaning. During these tests, the time to completely react the sodium was also measured. Time to react the sodium samples was about the same for both solvents, resulting in a recession rate of the sodium of about 0.01 in/min.

Using the above results, calculations were performed to determine the temperature rise that could be expected in the lower heel region. For conservatism it was assumed that no heat would be removed by the feedwater circulating inside the tubes. Based upon a solvent flow rate of 20 gpm (a lower limit value expected during fill), the steady-state flowing solvent temperature rise was calculated to be about 2°F. With feedwater circulation, it was estimated that there would be no noticeable temperature rise of the solvent from reaction of the sodium in the heel.

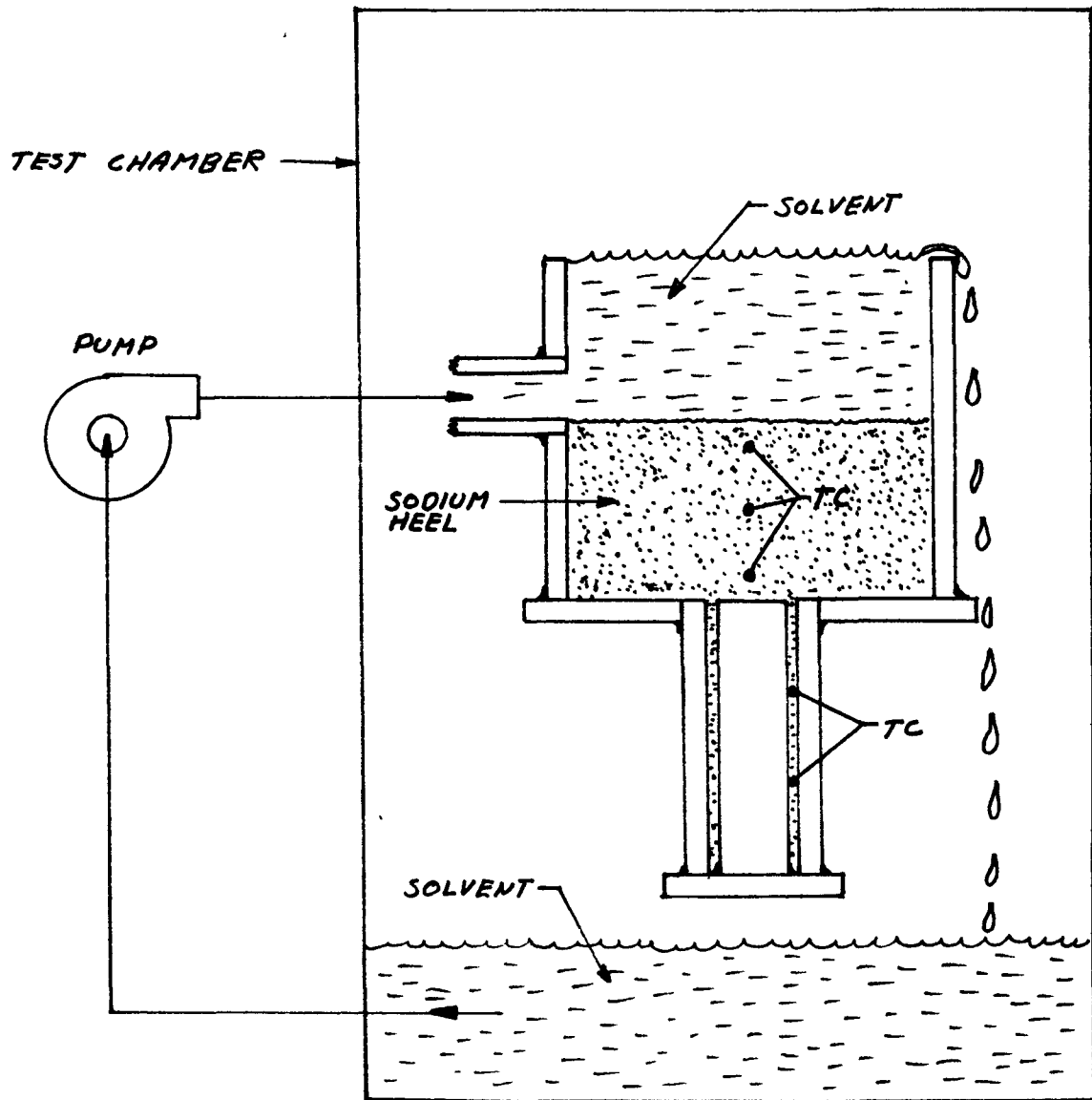
To further evaluate the reaction in the heel region, a laboratory scale test was performed mocking up the geometry in this region. In addition to determining actual temperatures that would be encountered, it was considered desirable to experimentally determine the time to react the sodium heel and the sodium in gaps between the steam tubes and thermal baffles. Also, it was important to have assurance that the

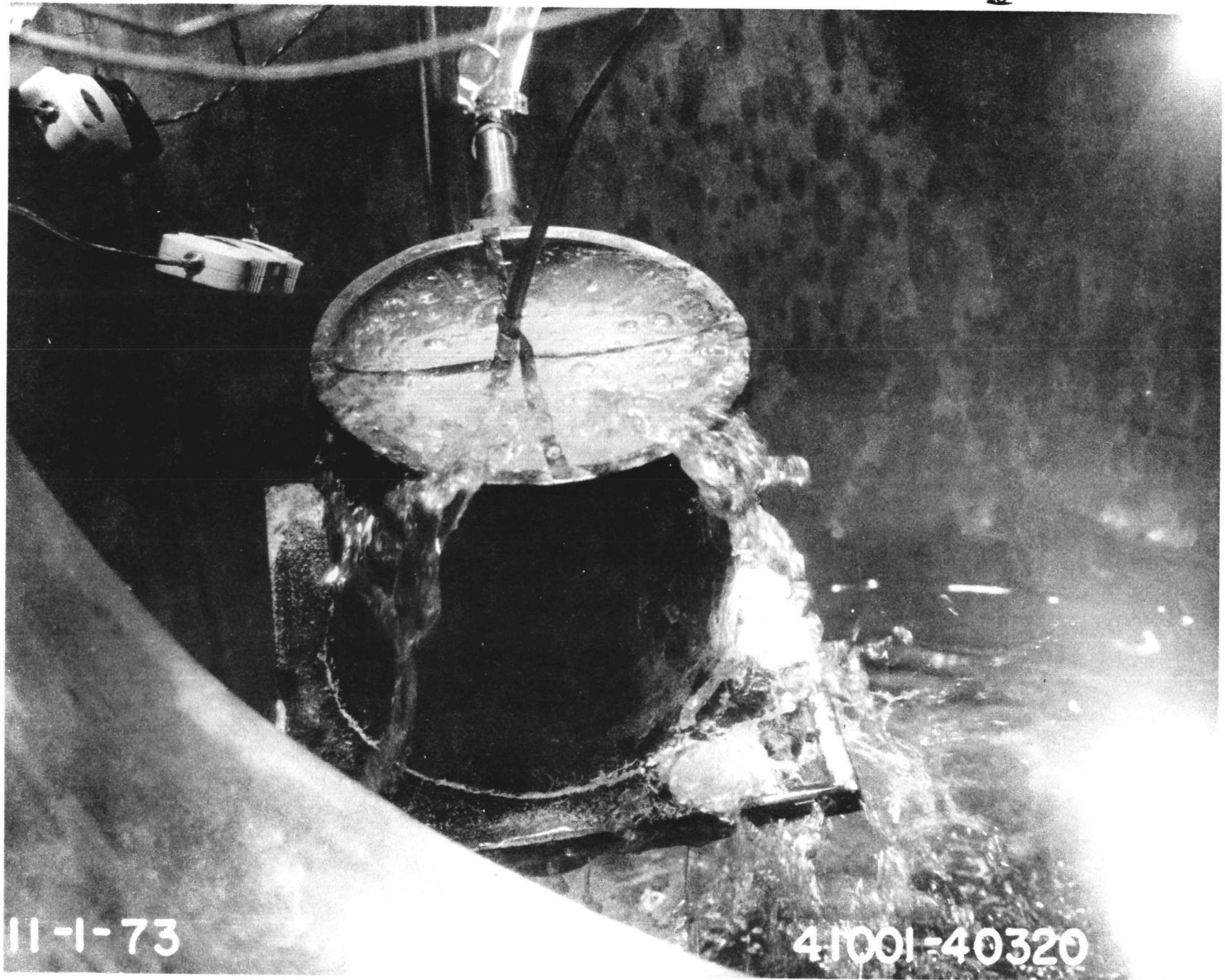
hydrogen evolution was not excessively violent and that all the sodium would be reacted. The sketch of experimental apparatus used for this test is shown in Figure IV-3. Thermocouples were located as shown. The apparatus was "cast" with sodium up to the lower edge of a 1/2-inch solvent inlet pipe. The sodium-filled assembly was then placed in a chamber and Dowanol PM was pumped over the sodium cake. A solvent flow rate of 1/2 gpm was used, which simulates the flow rate per unit surface area that would be used for the MSG cleaning. The solvent then overflowed the cast assembly to a chamber and pumped back through a 1/2-inch pipe.

Figure IV-4 shows a photograph of the actual test in progress. The agitation noted is from the solvent entering the four-inch pipe and not from the reaction with the sodium. During the test, while solvent was flowing, no increase in the temperature was observed from any of the thermocouples. Periodically the flow was stopped for several minutes to observe the hydrogen evolution. No temperature increase was observed during this period, and the hydrogen evolution was very gentle. At one point in the test, the solvent flow had to be stopped for 45 minutes to replace a pump impeller. During this period, the solvent in the upper region increased in temperature from 62°F to 192°F and that of the unreacted sodium in the 1/16 inch radial gap, to 135°F. (It should be noted that for MSG cleaning internal water circulation is maintained to continuously cool the alcohol. In the above experiment no such cooling was used.) The total elapsed time of the test for all hydrogen evolution to stop was 29 hrs. After all reaction had stopped, the apparatus was allowed to soak in Dowanol PM for two days, at which time a visual inspection was made. Feeler gauges were used to insert into the 1/16 inch annulus, which indicated there was no unreacted sodium or signs of residue.

Although potential temperature problems were identified in the above test in the event solvent flow had to be stopped or drained, the heat transfer to the feedwater was felt to be sufficient to alleviate any concern. However, as an added safety precaution, changes in the actual test procedure were made to minimize the chances of solvent draining during reaction of the sodium heel.

FIG. IV-3
SODIUM HEEL CLEANING TEST APPARATUS





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Figure IV-4 Sodium Heel Reaction Test in Progress

Additional heat transfer calculations were performed to determine the heat generated from exposed vertical surfaces during initial solvent filling. Potential problems were uncovered since it was found that essentially all the sodium reaction on vertical surfaces occurred in the upper one-foot of solvent. That is, the reaction rate was sufficiently rapid compared to the fill rate so that all the sodium was reacted before the sodium level moved about one-foot. Calculations showed that the feedwater could remove all the heat generated with only a fraction of a degree Fahrenheit temperature drop across the tube. Since the reaction was on the surface of the tube, it was anticipated that most of the heat generated would be transferred through the tube wall to the feedwater. It was not known, however, how much heat would be transferred to the solvent either by conduction, convection, or by the hydrogen gas that is evolved. Since there would be poor mixing of solvent, there was the potential of overheating the upper layer. To prevent a potential overheating problem, procedural changes were made to terminate filling if any of the internal thermocouples showed temperatures greater than 150°F. Such a procedure would allow sufficient time for the feedwater to remove the heat from the solvent until a safe level was obtained. Overheating in the heel region was anticipated to be a problem should such a procedure be required. With the height of solvent above the heel and natural convection mixing, only a small temperature rise would occur. Also, feedwater flow in the tubes would maintain relative low solvent temperatures.

C. Hydrogen Gas Evolution

During initial solvent filling, reaction rates are the largest due to the mass of sodium on vertical surfaces. During this reaction, hydrogen is evolved presenting a potential fire and explosion hazard, since the hydrogen is vented to the atmosphere. Based upon the laboratory scale reaction tests and the loop geometry, calculations were performed to estimate the expected hydrogen evolution rate.

Calculations of the hydrogen evolution rate were based on the know surface area, sodium film thickness, reaction rate, and chemical process. The resultant hydrogen evolution rate was determined to be as follows:

$$E_{H_2} = 0.11 Q + 0.4 \quad - 1 -$$

where

$$E_{H_2} = \text{Hydrogen Evolution rate (ft}^3/\text{min at STP)}$$

$$Q = \text{Solvent fill rate (gal/min.)}$$

The first term on the right hand side is the hydrogen evolved from vertical surfaces, which is a function of the solvent fill rate, and the second term from the sodium in the heel. Hydrogen evolution from crevices was not considered since it would be much lower than that released from vertical surfaces and the heel. To provide an upper limit of the hydrogen evolution for a "worst case" type analysis, it was assumed:

- 1) Fill rate is two times expected
- 2) Reaction rate is two times expected
- 3) Film thickness is two times expected

Equation (1) then becomes:

$$E_{H_2} = 0.44 Q + 0.8 \quad - 2 -$$

To determine the value of Q, the solvent fill rate, pressure drop calculations were performed for the filling process. The resultant fill rate was determined to be as follows:

$$Q = 2.85 \sqrt{425 - 2.91 L}$$

where

$$Q = \text{Solvent fill rate (gal/min)}$$

$$L = \text{Solvent height in MSG above inlet line (ft)}$$

The maximum fill rate occurs during the initial filling period ($L = 0$), resulting in a fill rate of 58.8 gpm. Combining this value in equation (1) for nominal conditions yields a hydrogen evolution rate of 6.9 scfm, and in equation (2) for "worst case" conditions, 26.7 scfm.

Prior to filling and during filling, an argon purge at the surge tank vent is maintained at a flow rate of 12.5 scfm. The resultant hydrogen concentration in the effluent gas was then estimated to be as follows:

<u>Conditions</u>	<u>Effluent Gas Hydrogen Concentration (Vol %)</u>
Nominal	35
"Worst Case"	68

It was expected that the actual hydrogen concentration in the effluent would be less than that indicated above, since the hydrogen would be further diluted with the argon cover gas maintained in the MSG and surge tank prior to filling. In addition, the effluent is exhausted to the atmosphere, resulting in further dilution. The calculated hydrogen evolution rate, however, is very small (.00065 - .0025 lb/sec corresponding to the nominal and "worst case" release rates). Hydrogen quantities considerably in excess of these values are routinely exhausted safely in rocket engine test programs. Consequently, no potential hazard problem was expected for the anticipated hydrogen evolution rates.

V. CLEANING PROCEDURE

Actual cleaning of the MSG was performed in accordance with written step-by-step procedures. Two separate procedures were used, the first for cleaning the loop prior to filling the MSG with solvent (Ref. 3), and the second for removing the residual sodium from the MSG (Ref. 4). The following presents a summary of the actual operations performed during the cleaning program.

A. Cleaning Loop Cleaning

The step-by-step procedure used for loop cleaning is given in Ref. 3. The cleaning procedure was written to meet the requirements at RRD Standard F5-1T (Ref. 5), except the fluid used for cleaning was Dowanol PM rather than water. Prior to initiating the procedure, the by-pass line (Figure III-1) was installed in the loop.

The initial steps in the procedure were to transfer the Dowanol PM from the vendor supplied 55 gal. drums to the fill and drain tank. Prior to this transfer, the fill and drain tank was cleaned with water and dried. The tank was inspected by Quality Assurance personnel and a surface wipe test conducted as specified in F5-1T (Ref. 5). After assuring cleanliness of the tank, the solvent transfer process was performed with a closed system to avoid moisture pickup by the solvent. Tubing was connected from a 55 gal. drum drain line to the fill and drain tank. The drum was pressurized to 1 ± 0.5 psig of argon to transfer the solvent. The above procedure was repeated until 20 drums of Dowanol PM were transferred to the fill and the drain tank (the estimated capacity of the cleaning loop and MSG was 17 - 18 drums). During the transfer operation, normal safety precautions were taken such as wearing of PVC gloves by all personnel, electrical grounding of drums, and no smoking in the vicinity of the cleaning loop. After all twenty 55 gal. drums of solvent were transferred to the fill and drain tank, a solvent sample was obtained and analyzed for water and chloride content.

The next step in the procedure was to fill the cleaning loop with solvent. Prior to this step, all lines and the surge tank were purged with argon. Filling was accomplished by pressurizing the fill and drain tank with 40 ± 5 psig of argon. With defined valve sequencing, the loop was filled with the Dowanol PM to a level in the surge tank 6 inches below the line leading from the top of the MSG. The pump was energized and solvent circulated through the loop for four hours. Following this period the pump was de-energized and the appropriate valves closed to allow disassembly of the filter. A bleached 100 yarn per inch "nainsook" (muslin) filter cloth

(per F5-1T) was installed over the filter element. The filter was reassembled and solvent circulation initiated. Circulation was continued for one hour and then terminated for inspection of the filter cloth. The above filter process was repeated until two successive filter cloth tests met the appropriate cleanliness requirements of F5-1T.

Following acceptance of the filter cloth cleanliness test, a Dowanol PM sample was obtained from the cleaning loop and analyzed for water and chloride content. After acceptably low levels were assured, the solvent in the cleaning loop was then transferred back to the fill and drain tank by pressurizing the surge tank with argon at a pressure of 20 ± 2 psig. The residual solvent remaining in the loop was drained from a drain line located at the low point in the loop. A final solvent sample was obtained from the fill and drain tank and again analyzed for water and chloride content. The cleaning system by-pass line was removed and two valves installed, completing the cleaning procedure for the cleaning loop.

B. MSG Cleaning

The step-by-step procedure used for MSG residual sodium removal is given in Ref. 4. The procedure has a list of prerequisites required to be completed before initiating the actual cleaning steps. These prerequisites include a loop pressure check, feedwater flow established at 600 lb/hr, gas chromatograph installed, and cleaning loop and MSG purged with argon to less than 0.5% oxygen.

The initial steps in the procedure were to fill the cleaning loop (exclusive of the MSG) with Dowanol PM, the same as that performed for filling the loop for cleaning above. The Data Acquisition System (DAS) was programmed to record all internal and selected shell thermocouple temperature readings every 5 sec. Selected internal TC readings were programmed for printout on a line printer for visual observation and for alarm at a temperature of 150°F. Feedwater flow was increased to 23,750 lb/hr at a temperature less than 100°F (actual temperature obtained was 66°F). The

gas chromatograph was placed in service. Filling of the MSG was accomplished by opening the vent line valve (V-113, Figure III-1) and the drain line valve (V-112). Temperatures were monitored during fill to assure that none of the internal TC's recorded temperatures higher than 150°F. Special steps were provided for terminating the fill if any internal temperature exceeded 150°F and allowing the MSG to cool until it dropped below 125°F before resuming the filling. In addition, a special emergency drain procedure was provided in the event any internal temperature exceeded 150°F for a 30 minute period. Filling of the MSG was completed when the solvent level in the drain tank increased by 4 inches.

When filling was completed the pump was energized to commence solvent circulation. After a 30-minute circulation period, the filter was brought on-line and argon sparge gas flow was initiated at a flow rate of 10 scfm (later reduced to 5 scfm because of the high argon consumption rate). The feedwater temperature was then increased to 120°F (later increased to 150°F and then to 175°F to increase the reaction rate). After temperature stabilization, Dowanol PM samples were taken on a periodic basis and analyzed for sodium concentration by atomic absorption spectrophotometry. The original cleaning procedure provided for termination of the Dowanol PM cleaning when six consecutive readings taken 1 hour apart were within plus or minus 15 ppm of sodium. This procedure was modified when it was found, that because of the high sodium ion background from reaction of the heel and exposed surface sodium, the sodium ion content could only be measured with an accuracy of about ± 100 ppm (5%). To provide an indication of the reaction rate, effluent gas bomb samples were obtained on a periodic basis and analyzed for hydrogen content. This method was used to track the sodium reaction rate during Dowanol PM circulation.

After 11 days of circulation, it was decided to drain the Dowanol PM and continue cleaning with ethanol (an additional supply of Dowanol PM could not be procured within a reasonable time period). Since the ethanol has a somewhat higher reaction rate with the sodium than the Dowanol PM, it was felt that there was a significant advantage in using a fresh solvent (with an initial low sodium ion content) for tracking reaction rate by sodium concentration analysis.

The Dowanol PM draining was performed by initially reducing feedwater temperature to less than 100°F, and terminating sparge gas flow. The Dowanol PM was drained from the cleaning loop and MSG by pressurizing the surge tank with argon and blowing down to the fill and drain tank. The Dowanol PM in the fill and drain tank was transferred to the 55 gallon drums by pressurizing the fill and drain tank with argon to 10 \pm 2 psig. A flexible drain line was connected from the fill and drain tank to a 55 gallon drum to make the solvent transfer. This procedure was repeated until all the Dowanol PM was transferred to 55 gallon drums. The same safety precautions were observed as for the transfer to the fill and drain tank.

Ethanol was then transferred from the vendor supplied 55 gallon drums to the fill and drain tank, and then transferred to the cleaning loop and MSG. These steps were performed in the same manner as with Dowanol PM loading. Circulation and sparging (at 5 scfm) were initiated, and the feedwater temperature was increased to 120°F (later increased to 140°F). Periodic solvent samples were obtained and analyzed for sodium ion content, as well as gas bomb samples, which were analyzed for hydrogen content. The procedure called for termination of the ethanol flush when six consecutive samples taken 4 hours apart were within 3 ppm of sodium. During the ethanol flush the sodium ion content showed a continuing increase. After more than two weeks of circulation, however, the ion level was sufficiently high so that interpretation of the results was confused by measurement inaccuracy. Consequently, a decision was made to repeat the ethanol flush using newly procured ethanol.

During the above mentioned ethanol flush, considerable evaporation of the solvent occurred. Because of late delivery of a second ethanol inventory several contingency actions were performed to reduce the evaporation rate. The initial action was to collect the ethanol condensate from the surge tank vent line and recycle it back into the loop. Before recycling, the condensate was analyzed for water content to assure that there was not excessive moisture pickup. Additional actions taken to reduce evaporation also included reducing the sparge interval from 24 to 12 hours per day, and reduce sparge rate from 5 to 2 scfm.

A second ethanol flush was initiated in an identical manner as previously mentioned. Solvent temperature was maintained constant at 140°F during the entire second ethanol flush. Sparging was performed on a 12-hr on - 12-hr off period at a flow rate of 2 scfm. Solvent samples were taken every four hours and analyzed for sodium ion content, and gas bomb samples taken near the end of each sparge gas period and analyzed for hydrogen content. The second ethanol flush was terminated 12 hours after six consecutive sodium ion samples taken four hours apart were within plus or minus 3 ppm of sodium. At this time, the gas bomb samples also showed a negligible sodium reaction rate as deduced from the hydrogen analysis.

The ethanol was drained from the cleaning loop and MSG to the fill and drain tank and then transferred to the individual 55 gallon drums. The MSG was then dried. This drying was accomplished by installing an alcohol detector at the surge tank vent and then increasing the MSG feedwater temperature to 180°F (later reduced to 150°F to preclude the possibility of stress corrosion cracking should any caustic form). Argon sparge gas flow was initiated at a flow rate of 5 scfm. Drying was to continue until the effluent alcohol content was less than 300 ppm. Continued high effluent alcohol readings occurred over a several day period. It was postulated that the alcohol evaporated in the MSG was condensed in the cold surge tank, giving a false reading. The surge tank was removed from the loop and the alcohol detector relocated.

Subsequent effluent alcohol readings showed values to be less than 300 ppm. The MSG was then isolated by closing the inlet and outlet valves for 12 hours while maintaining temperature. The valves were then opened and effluent alcohol readings taken to assure the values were still less than 300 ppm. Drying was continued for an additional two hours at which time a final effluent sample was procured and drying terminated. Feedwater temperature was then increased to 190°F and the water was drained from the MSG.

The final step in the MSG cleaning procedure was to perform a helium leak test to assure integrity of the unit. This was performed by initiating an argon carrier gas flow on the shell side, using the cleaning loop sparge gas system. The tube side was pressurized with helium to 100 psig. The vent line on the shell side was then monitored for helium for a 30 minute period. No hydrogen was detected, completing the MSG cleaning operations.

VI RESULTS

A. Solvent Chemical Analysis

Prior to and during cleaning, solvent samples were obtained and analyzed for water and chloride ion content. Water content was monitored to obtain base readings of the delivered solvent, and to assure there was not excessive moisture pickup during the various cleaning steps. Water content was measured by the Karl Fischer method. Chloride ion content was determined only for potential future information, as the MSG is fabricated from ferritic steel, and is not subject to chloride stress corrosion. Chloride ion content was measured by x-ray fluorescence. Chemical analysis for sodium ion content is presented in subsequent sections.

Table VI-1 presents the results of the chemical analyses for water and chloride ion. The first three samples (700, 701, and 702) were obtained from three 55 gallon drums of Dowanol PM as delivered from the vendor. Sample 745 was obtained from the drain tank following the initial loading of 20 drums of Dowanol. It can be noted that there was some water pickup. It is not known whether this increase occurred during transfer, or from residual water in the fill and drain tank, or possibly from water in the sample line. The next sample (746) was obtained following completion of the cleaning loop cleaning. It was obtained to assure that there was no major water pickup by the solvent during cleaning and that it was acceptable to return to the fill and drain tank. Sample 747 was obtained from the fill and drain tank prior to the initial MSG filling. Samples 771-774 were taken from vendor supplied 55 gallon drums that were to be used for makeup. It can be noted that the average water content from these drums was slightly higher than that from the initial batch (samples 700-702).

TABLE VI-1
SOLVENT WATER AND CHLORIDE ION CONTENT

Sample Lab No.	Solvent	Date Taken	Time Taken (Hr:Min)	Location	H ₂ O (wt %)	Cl ⁻ (ppm)
700	Dowanol PM	11-15-73	-	Vendor supplied 55 gal drums	0.154	< 30*
701	Dowanol PM	11-15-73	-	Vendor supplied 55 gal drums	0.159	< 30
702	Dowanol PM	11-15-73	-	Vendor supplied 55 gal drums	0.160	< 30
745	Dowanol PM	11-30-73	22:05	Fill & Drain Tank-Initial Solvent Fill	0.236	< 30
746	Dowanol PM	12-1-73	22:22	Cleaning loop - After loop cleaning completion	0.192	< 30
747	Dowanol PM	12-3-73	09:00	Fill & Drain Tank - Prior to MSG filling	0.254	< 30
766	Dowanol PM	12-7-73	10:20	Sample line - After 3 1/2 days of cleaning	0.153	< 30
771	Dowanol PM	12-13-73	-	Vendor supplied 55 gal drum - makeup	0.196	< 30
772	Dowanol PM	12-13-73	-	Vendor supplied 55 gal drum - makeup	0.207	< 30
773	Dowanol PM	12-13-73	-	Vendor supplied 55 gal drum - makeup	0.204	< 30
774	Dowanol PM	12-13-73	-	Vendor supplied 55 gal drum - makeup	0.210	< 30
782	Dowanol PM	12-14-73	10:12	Sample line - Prior to loop drain	0.021	< 30
788	Ethanol	12-20-73	15:48	Vent line - Condensate for recycle	0.058	-
789	Ethanol	1-3-74	19:00	Fill & Drain Tank - After fresh solvent fill	0.087	< 30

* Limit of detection - not detectable

Sample 766 was obtained from the sample line after about 3 1/2 days of cleaning, and sample 782 after 11 days of cleaning. Comparing these two readings with sample 747 (value prior to cleaning), indicates there was a noticeable decrease in the water content with cleaning time. It is not known whether this reduction is due to evaporation with time or resulted from the chemistry of the sodium reaction.

The final two samples were for ethanol - the first (788) was obtained from the condensate collected from the surge tank vent line. This sample was taken to determine if the condensate could be recycled back into the cleaning loop. The water content was sufficiently low to permit recycling. The final sample was obtained from the fill and drain tank after charging with a fresh supply of ethanol. It can be noted that the water content of the ethanol was significantly less than that of Dowanol PM. In all the samples (both Dowanol PM and ethanol), there was no detectable chloride ion content (less than 30 ppm based on the lower detection limit of the analytical technique).

B. Cleaning Loop Cleaning

As mentioned in section V, the cleaning loop was cleaned and checked out prior to filling the MSG with solvent. The cleaning was performed in accordance with RRD standard F5-1T (Ref. 5). Prior to loading the initial charge of solvent into the fill and drain tank, the inside surface was visually inspected and a wipe test performed. The visual inspection revealed several areas of discoloration. The areas, however, were only a few square inches, which is much less than the 1% as allowed by F5-1T. The wipe test showed normal surface contaminants, such as nickel, chromium, and iron oxide, in addition to trace amounts of metals such as aluminum, copper, zinc, etc. These contaminants, however, were expected in a stainless steel system indicating an acceptable cleanliness of the tank.

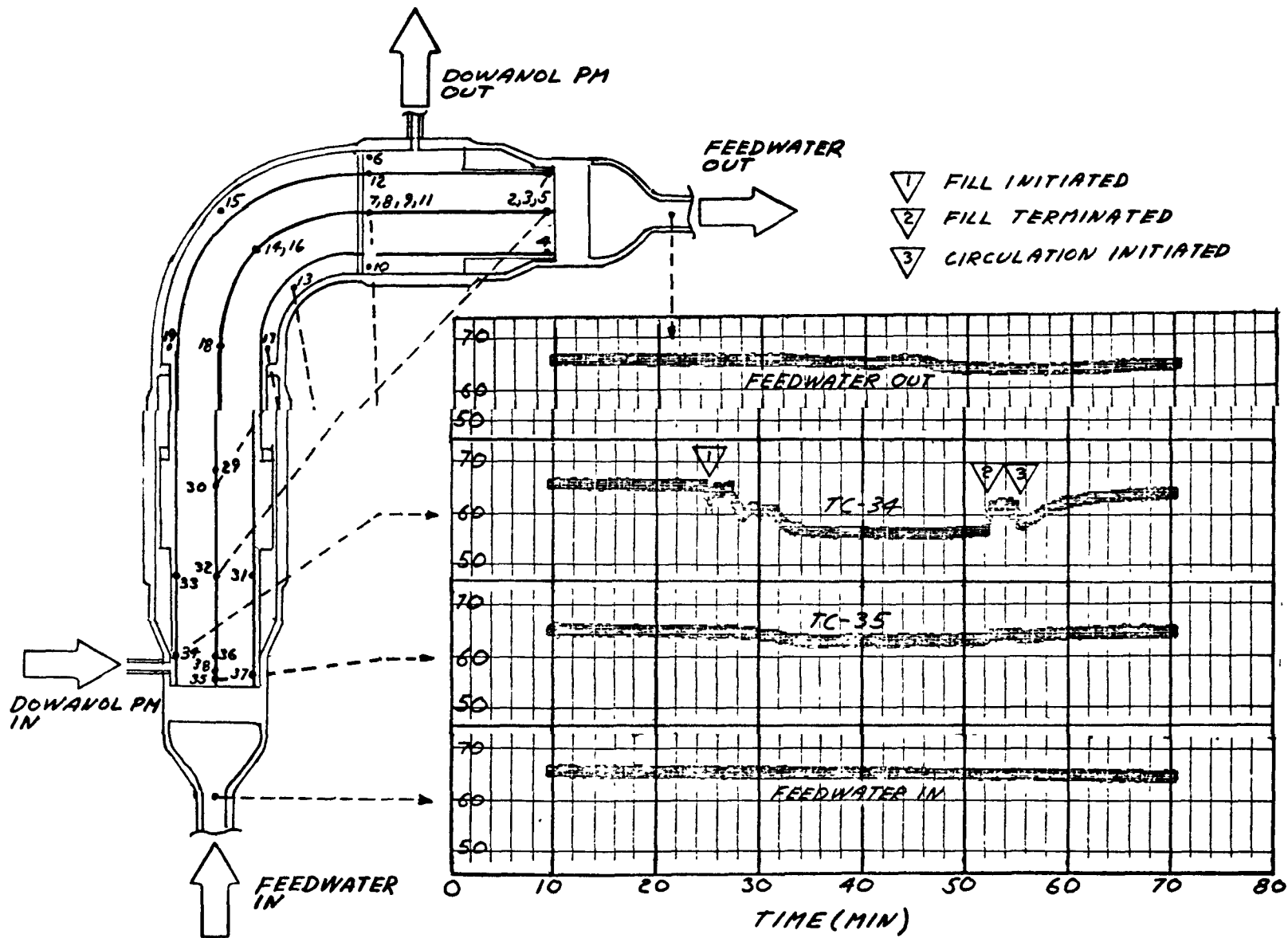
After filling the cleaning loop (with MSG by-pass line in place) with Dowanol PM, cleanliness was checked by circulating the solvent through a muslin filter cloth. The filter cloth was inspected and the process repeated. Inspection of the cloth from both flushes revealed that the general appearance, collected particle size, and other contaminant collection met the flush acceptance criteria of F5-1T.

C. MSG Initial Fill

During the initial filling of the MSG with Dowanol PM, internal temperatures and the effluent hydrogen concentration were monitored. Figure VI-1 presents selected internal temperatures at various elevations during filling. The fill time history can be noted from the temperatures recorded from TC-34, which was located adjacent to the MSG drain line (Dowanol PM fill line). Prior to filling, the unit was at a uniform temperature of about 66°F, the temperature of the circulating feedwater. Only slight differences can be noted among the different TC's. The entering Dowanol PM was at a temperature of about 56°F as noted from TC-34. Total filling of the MSG took about 27 minutes, and circulation was initiated about 4 minutes later. The initiation of filling can be noted from TC-34 by a sharp drop in temperature of about 5°F. The temperature increased followed by an erratic decrease to the Dowanol PM temperature. This behavior is felt to have been caused by a combination of valve sequencing during the initiation of fill and the reaction of sodium in the heel area and on the surface of the thermocouple. The termination of filling can be noted by the sharp increase in temperature of TC-34, occurring from the heat of reaction of the sodium in the heel region and from heat being transferred from the feedwater. The initiation of solvent circulation can be noted from the sharp decrease in temperature occurring when the cooler solvent immerses the thermocouple. Temperatures then gradually increased to the feedwater temperature. The temperatures indicated by TC-35, located in the solid heel region, were affected very little during filling. A decrease of only a few degrees was observed, resulting from the cooler solvent absorbing some of the heat from the sodium heel.

The time at which the solvent contacted a thermocouple at a given elevation can be noted from Figure VI-1. At the lower thermocouples (TC-32, 30), a temperature drop was noted when the solvent reached the TC. This drop was apparently the result of the cooler solvent overpowering the heat of reaction of the sodium on the thermocouple surface. As filling progressed, a temperature increase was noted (starting with TC-28). This is the result of the upper surface of the solvent being heated by the reaction

FIG. VI-1
INTERNAL TEMPERATURES DURING DOWANOL PM FILLING



of sodium on vertical surfaces. The maximum temperature increase (on TC-6) was only 10°F. After the temperature increase, temperatures dropped as the upper heated solvent zone passed the thermocouple elevation. The temperature level after the drop increased with elevation as the solvent was heated by the feedwater. During the entire filling period the maximum temperature observed was only 72°F.

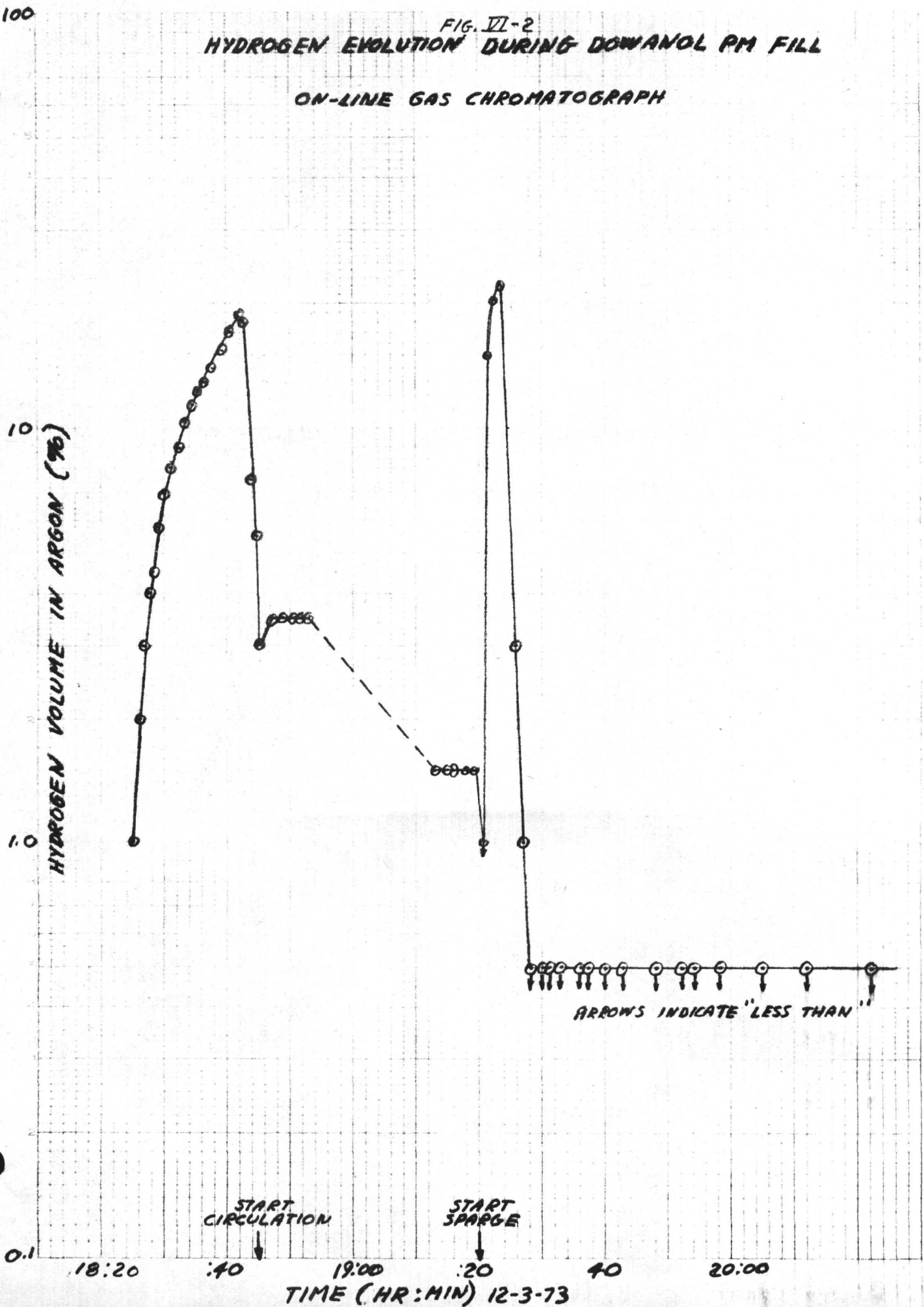
The results of hydrogen gas concentration, as obtained from the on-line gas chromatograph, are presented in Figure VI-2. During fill, the hydrogen gas evolution increased to a peak level of 19% and then dropped rapidly. This peak value agrees reasonably well with the predicted value of 35% (section IV-C) considering the dilution of the hydrogen with the argon gas in the MSG and surge tank free volumes. A sudden increase in the hydrogen concentration occurred when the sparge gas flow (at the MSG drain line) was initiated. This increase apparently resulted from sweeping the hydrogen from small pockets in the MSG and that accumulated at the top of the surge tank. The level quickly dropped to below 0.5%, the lower limit of the gas chromatograph.

D. Dowanol PM Flush

About 3 hours after Dowanol PM fill, samples of the solvent were obtained periodically and analyzed for sodium concentration. The initial results of chemistry analysis indicated a sodium concentration of slightly over 1900 μg of Na per ml of solvent. After several hours (approximate time for the bulk sodium in the heel to react), the concentration reached a value of 2100 μg of Na per ml of solvent. From this value, the estimated sodium mass reacted was determined to be (based on a solvent volume of 130 ft^3):

$$\begin{aligned}\text{Sodium Mass} &= 130 \text{ ft}^3 \times 62.4 \frac{\text{lb/ft}^3}{\text{g/ml}} \times 10^{-6} \frac{\text{g}}{\mu\text{g}} \times 2100 \frac{\mu\text{g}}{\text{ml}} \\ &= 17.0 \text{ lb}\end{aligned}$$

FIG. VII-2
HYDROGEN EVOLUTION DURING DOWANOL PM FILL
ON-LINE GAS CHROMATOGRAPH



Plans were to continue tracking the reaction rate using the results of the sodium concentration analysis. The chemistry analysis, however, only had a precision of plus or minus 5%, or a concentration of plus or minus 100 $\mu\text{g/ml}$ at the indicated value.

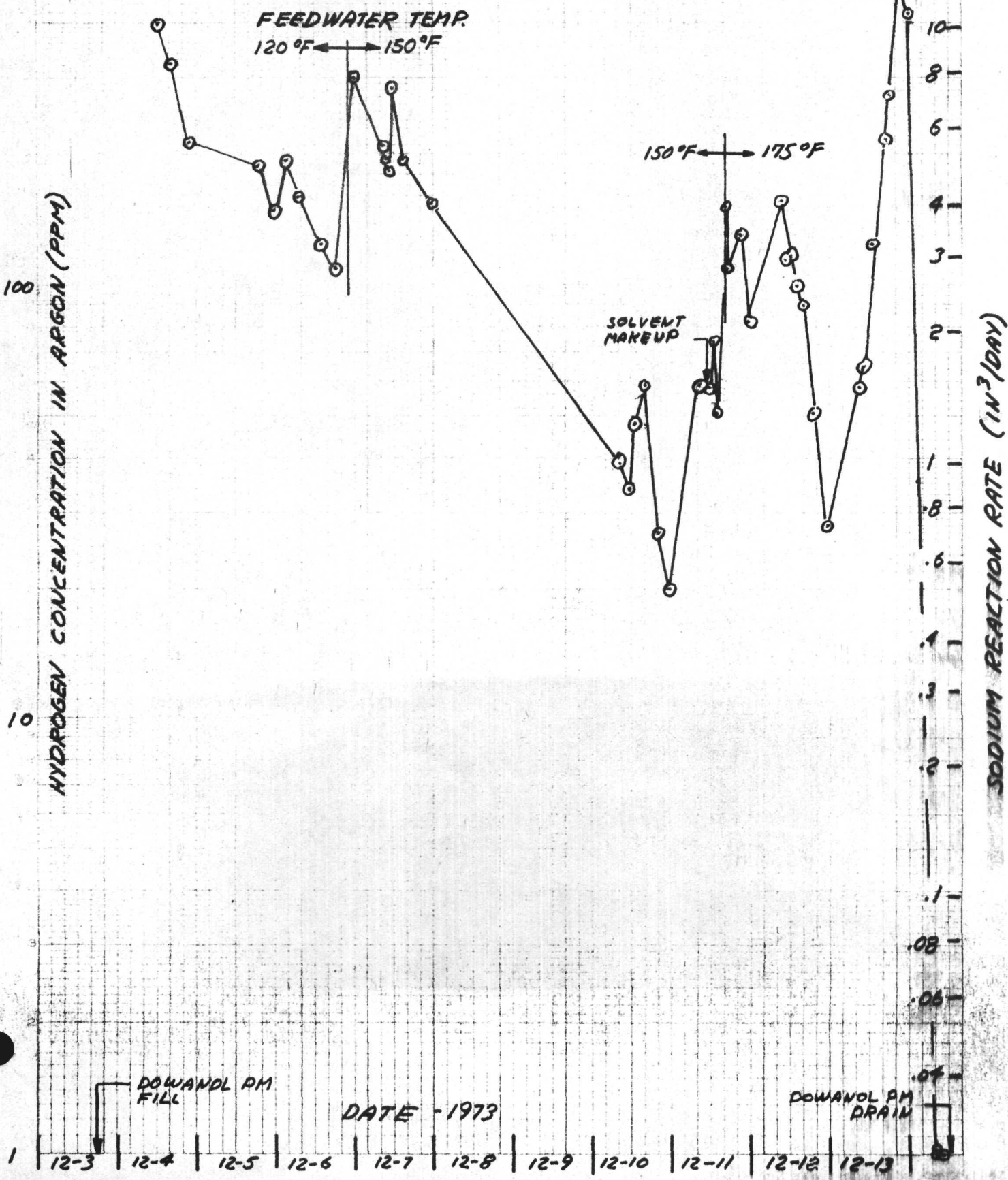
To provide a more accurate evaluation of the reaction rate, samples of the effluent gas were taken and analyzed in the laboratory for hydrogen content. The results of these analyses obtained during the Dowanol PM flush are given in Figure VI-3. The left hand scale shows the hydrogen content in ppm and the right hand scale gives the equivalent reaction rate in cubic inches of sodium per day. The concentration showed somewhat of an erratic behavior, possibly as the result of crevices opening up and being exposed to the solvent.

The temperature was increased twice during the Dowanol PM flush, once from 120 to 150°F and then to 175°F. During both changes the reaction rate increased by about a factor of 3. It is not known whether this change represents the result of increased reaction rate with temperature, opening up of crevices, or if the higher temperature released some of the dissolved hydrogen gas. It is not expected that the reaction rate would increase so rapidly with temperature since the bulk of the reactions are in crevices which are more diffusion than reaction rate controlled. Preliminary calculations indicate that the observed reaction change during the temperature increase is about the right magnitude as would be expected from gas solubility considerations. Prior to Dowanol PM drain, the temperature was reduced to 85°F. At this time the indicated reaction rate dropped by almost 3 orders of magnitude. Such a reduction tends to confirm that indicated reaction rates during and after temperature changes are mainly the result of hydrogen gas being released or dissolved from the solvent.

The curve in Figure VI-3 was integrated to estimate the total amount of sodium reacted during the Dowanol PM flush. This integration yielded a sodium mass of 1.3 lb. Combining this value with the 17 lb obtained from the sodium concentration obtained several hours after fill yields a total mass of 18.3 lb of sodium.

FIG. VI-3
MSG CLEANING
HYDROGEN SAMPLE RESULTS

SPARGE GAS: ARGON @ 5.0 SCFM
SOLVENT: PROPYLENE GLYCOL METHYL ETHER
(DOWANOL PM)



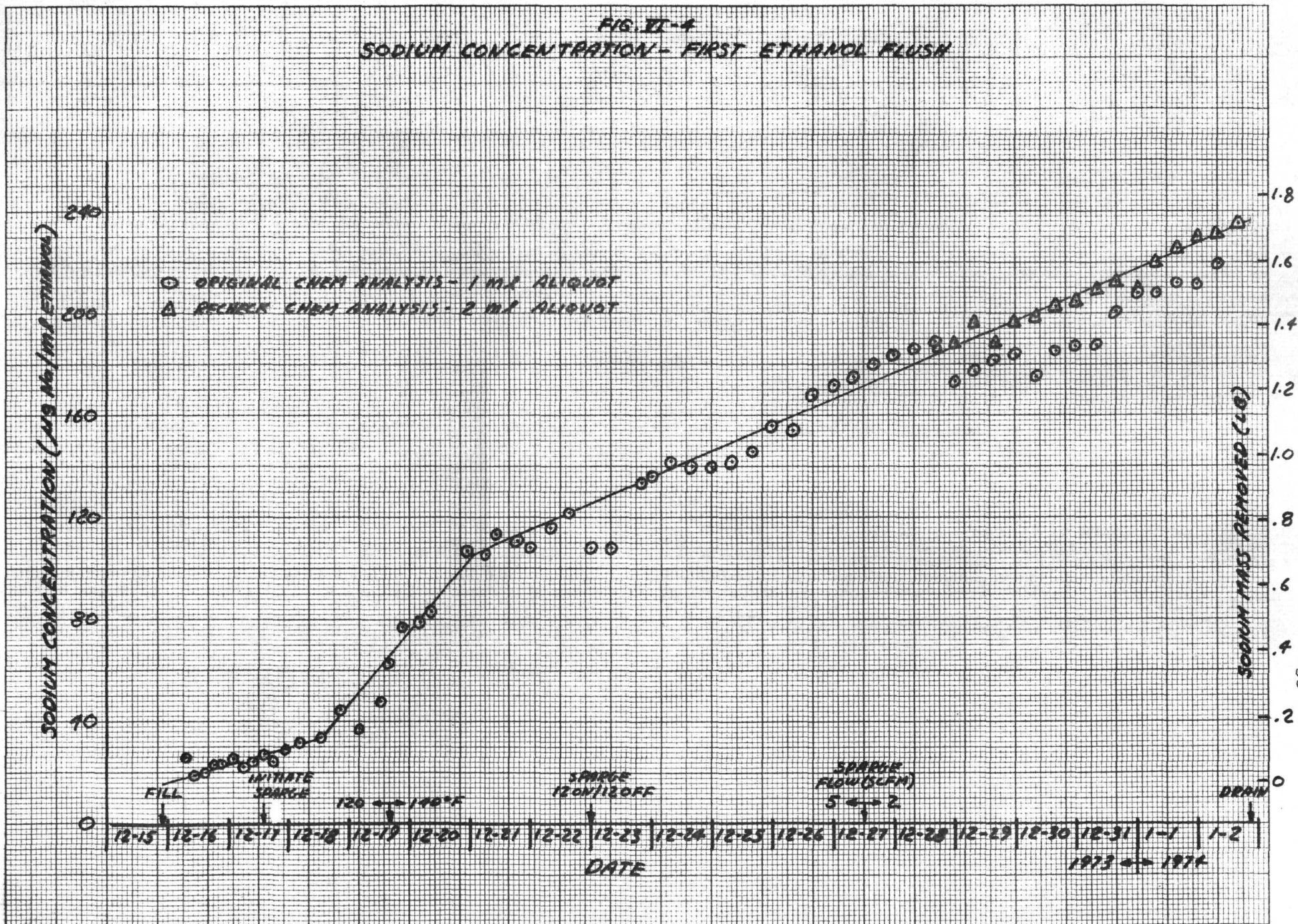
E. First Ethanol Flush

Cleaning was continued using ethanol in order to provide a better resolution of the reaction rate from sodium concentration analysis. The results of the sodium concentration analysis are presented in Figure VI-4. The left hand scale gives the actual sodium concentration in μg of sodium per ml of ethanol. The right hand scale gives the equivalent mass of sodium reacted. Sparge gas flow was initiated after two days of ethanol circulation, and after an additional two days, the feedwater temperature was increased from 120°F to 140°F . Eliminating sparging for the first two days was performed to determine the effect of sparging on reaction rate. During the ethanol flush, evaporation of the ethanol reduced the available makeup supply at an alarming rate (a fresh supply was not available at that time). To conserve the available supply, the sparge gas flow was changed from continuous to a 12-hr on and 12-hr off cycle. In addition, the sparge gas flow was reduced from 5 scfm to 2 scfm. The ethanol condensate was also collected and recycled back to the loop. Water content was shown to be acceptably low as indicated in Table VI-1. In addition, a condensate sample was analyzed for sodium content. Results of this analysis showed the sodium concentration to be less than one $\mu\text{g}/\text{ml}$, indicating that the sodium ethoxide formed is not evaporated along with the ethanol at the operating temperature.

It can be noted from Figure VI-4 that there appears to have been three distinct reaction rates that occurred during the ethanol flush (The slope of the concentration curve is proportional to the sodium reaction rate.). The initial slope changes are postulated to be the result of the initiation of the sparge gas flow. Since the reaction is primarily in crevice regions and, therefore, controlled by diffusion, the reaction products formed take a period of time to reach the main solvent stream where samples are obtained. Sparging would tend to decrease this time, thus initially increasing the apparent reaction rate.

The chemical analysis of the solvent for sodium concentration was performed in batches accumulated over a several-day period. Trends within a single batch appeared consistent. However, there was a noticeable change

FIG. VI-4
 SODIUM CONCENTRATION - FIRST ETHANOL FLUSH



from batch-to-batch. To evaluate this effect, several batches were reanalyzed using a 2 ml aliquot. The recheck of the chemical analyses are shown by the triangles in Figure VI-4, indicating a more consistent trend. It should be noted that all chemical analysis data points are within plus or minus 5%, the estimated precision of the process.

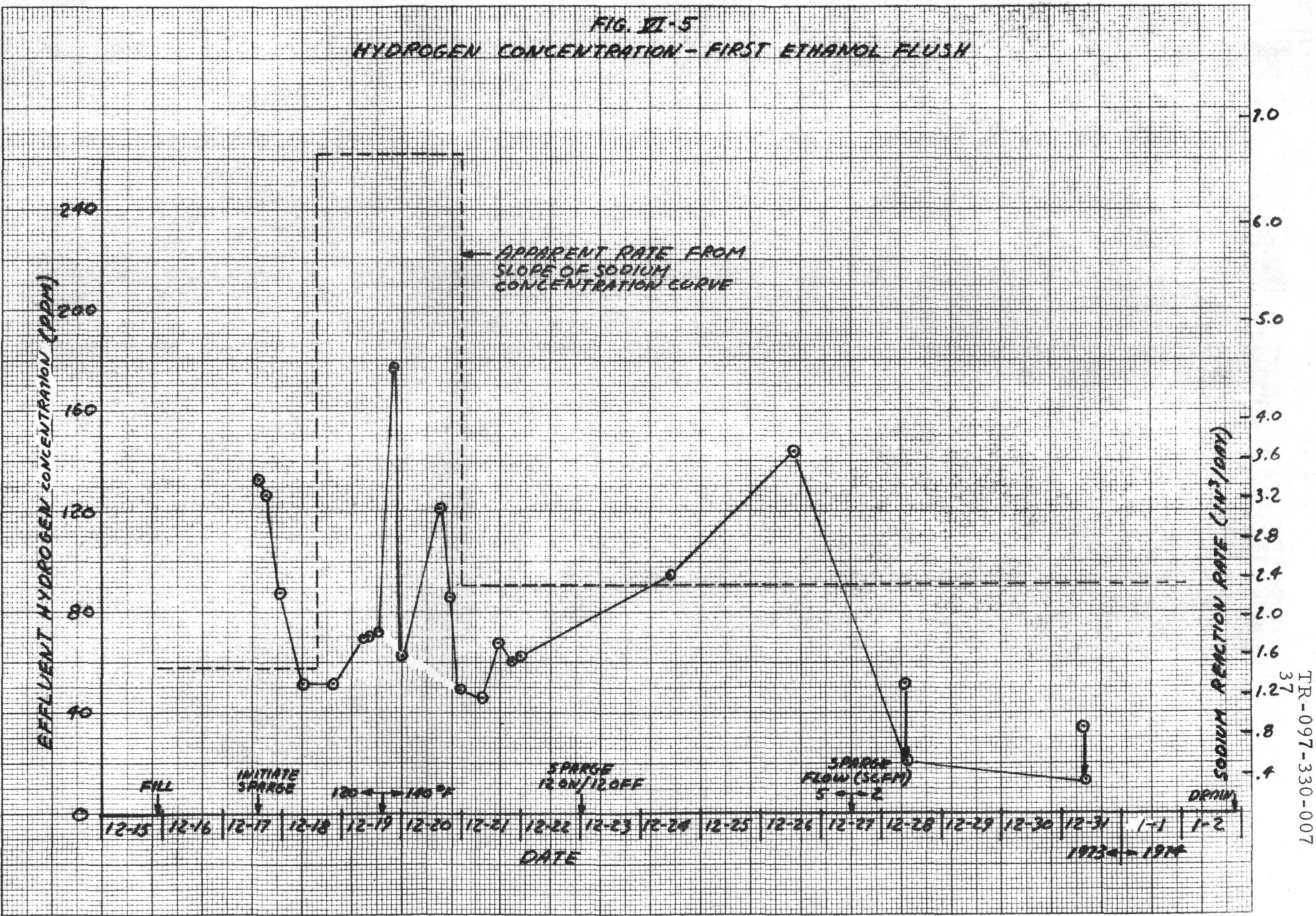
Gas samples were also taken during the ethanol flush and analyzed for hydrogen concentration. The results of these analysis are presented in Figure VI-5. The left hand scale gives the actual hydrogen concentration in ppm, and the right hand scale the equivalent reaction rate. The last two data points were obtained during a sparge gas flow of 2 scfm, and were corrected to equivalent values at a flow of 5 scfm. The dashed line shows the apparent reaction rate obtained from the slope of the sodium concentration curve (Figure VI-4). Several gas samples were taken during a period of no sparging (not shown in Figure VI-5). These show an extremely erratic behavior, giving either very high or very low readings. As can be noted from Figure VI-5, there was a marked increase in the apparent reaction rate when the temperature was increased. Again, as with the Dowanol PM flush, this behavior is thought to be caused by hydrogen gas solubility effects.

There appears to be general agreement between the reaction rate obtained from the sodium concentration analyses and the gas samples. Because of the small number of gas samples obtained, it was not possible to accurately determine the absolute mass of sodium reacted. The sodium concentration analysis is a more accurate estimate of the sodium reacted. From Figure VI-4, the sodium mass reacted during the ethanol flush was estimated to be 1.7 lb. Combining this value with the 18.3 lb from the Dowanol flush, yields a total estimated sodium mass reacted of 20.0 lb.

F. Second Ethanol Flush

After a fresh charge of ethanol was procured, the used ethanol was drained and the fresh supply added to the cleaning loop and MSG. The second ethanol flush was performed at a temperature of 140°F, and a two scfm sparge gas flow was operated for 12 hours per day. Sodium reaction

FIG. VI-5
 HYDROGEN CONCENTRATION - FIRST ETHANOL FLUSH



was monitored as before by means of sodium concentration analysis and effluent hydrogen gas analysis. Figure VI-6 shows the results of the sodium concentration analysis. It can be noted that there was a gradual, but significant reduction in the reaction rate. A similar, but more dramatic trend was noted from the effluent hydrogen gas analysis. As shown in Figure VI-7, hydrogen gas evolution decreased to a negligible rate after about 5 days of circulation. The dashed line in Figure VI-7 represents the apparent rate obtained from the slope of the sodium concentration curve (Figure VI-6). The agreement between these two methods of reaction rate determination is better than that obtained during the previous ethanol flush. There appears to be a several day delay in the reaction rate as determined from sodium concentration and that determined from the gas sample data. This is postulated to be because of the hydrogen gas that is released from the sodium reaction enters the main flow stream and is swept to the effluent by the sparge gas in a very short time period. The sodium ethoxide formed in small crevices, however, takes a much longer time to diffuse into the main solvent stream.

Based upon the low reaction rate observed from the sodium concentration analysis and the negligible rate from the hydrogen concentration, the decision was made to terminate the cleaning. The second ethanol charge was drained and the MSG dried at a temperature of 180°F (later reduced to 150°F to preclude any caustic stress corrosion problems).

Based on the sodium concentration (Figure VI-6), the total sodium removed during the second ethanol flush was 0.3 lb. Combining this value with the 20.0 lb obtained from previous flushes gives a total estimated mass of sodium removed during the cleaning process of 20.3. This total value agrees closely with the predicted residual sodium mass of 20.8 lb following sodium drain.

G. Residue

During cleaning with Dowanol PM, samples procured for chemical analysis were observed to have a fine black particulate matter suspended in the solvent that would settle to the bottom of the sample container. Inspection of the filter in the cleaning loop revealed it to be caked with a similar fine black powder.

FIG. VI-6
SODIUM CONCENTRATION - SECOND ETHANOL FLUSH

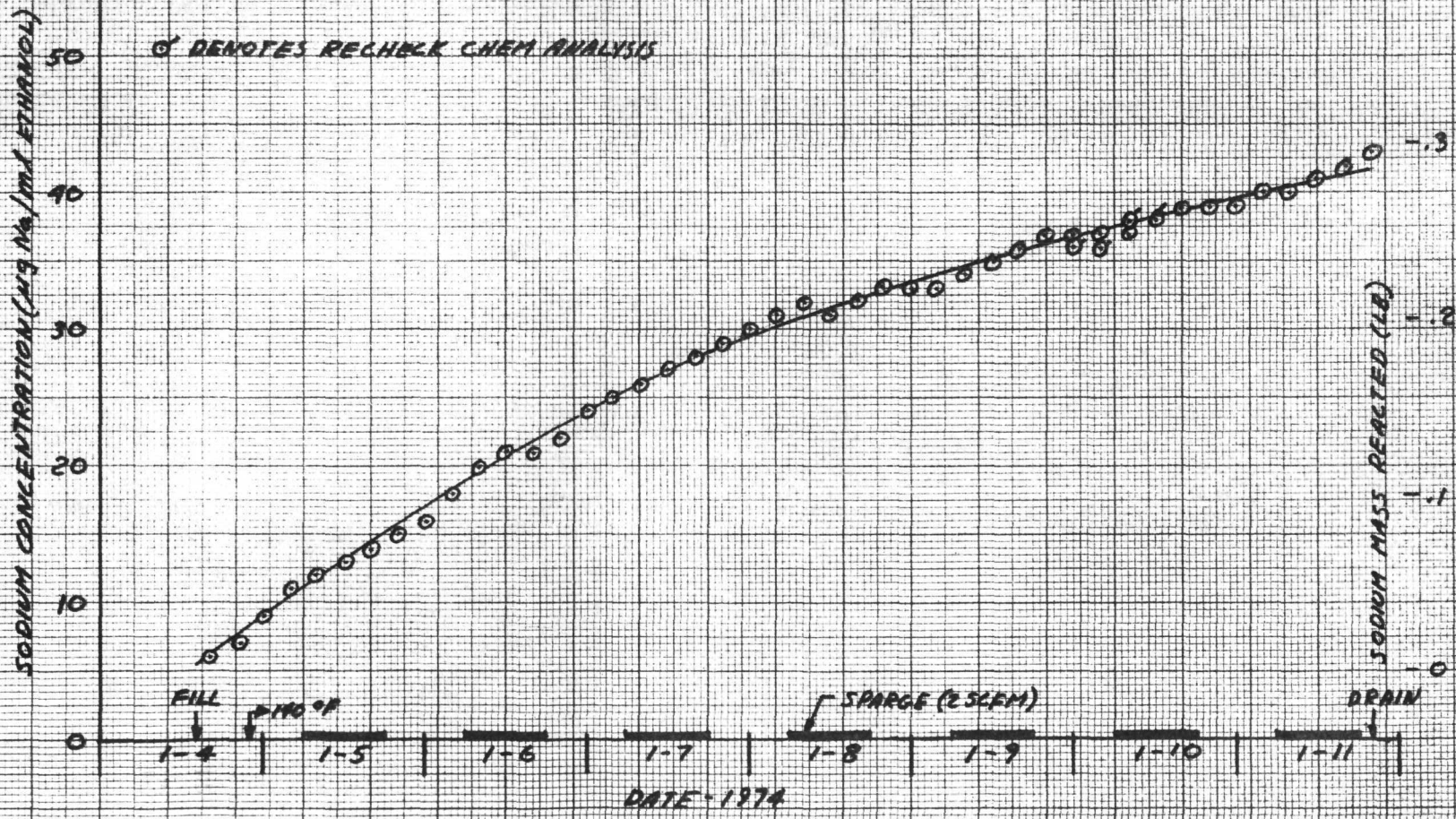
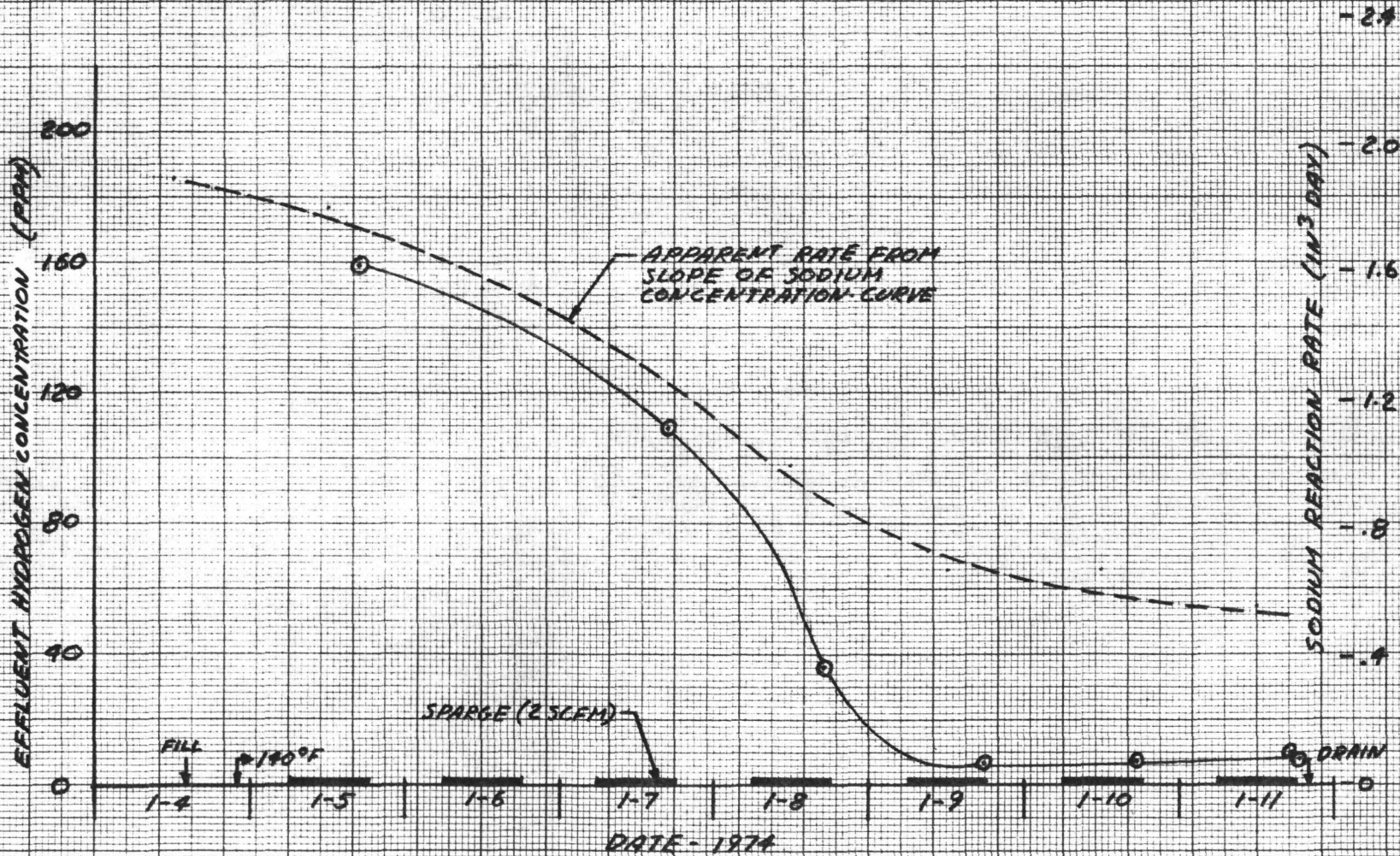


FIG. VI-7
 HYDROGEN CONCENTRATION - SECOND ETHANOL FLUSH



Analyses of the black powder were performed on the material taken from the filter. An x-ray fluorescence scan of the sample showed the presence of Fe(major), Cr(first minor), Ni(second minor), and Mo(third minor). X-ray diffraction of the deposit showed it to be alpha Fe which is isostructural with Croloy. From the x-ray fluorescence, the estimate of the composition of the residue is as follows:

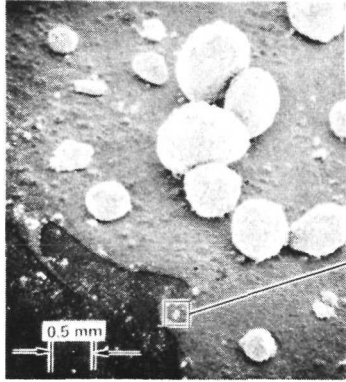
Fe	-	60-80%
Cr	-	4-5%
Ni	-	1-2%
Mo	-	~1 %

In addition, emission spectroscopic examination showed about 2% sodium, which was judged to be a sodium salt surface contaminant.

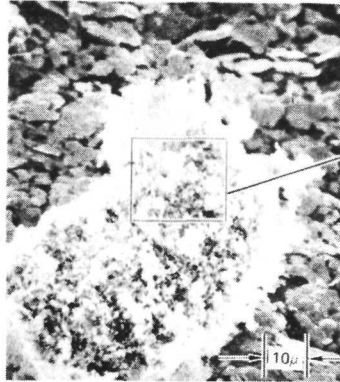
Samples of the residue were viewed in a scanning electron microscope (SEM). Various micrographs obtained from the SEM analysis are shown in Figure VI-8. Photo d shows a surface contour which appears to be glassy in appearance. Limited resolution of the SEM at the indicated magnification, however, would tend to indicate the particle viewed is an agglomerate of much smaller particles. Photo f shows particles as small as 0.2 microns in size.

The above observations and supporting analysis indicates the observed deposits are nearly identical with the base metal (2-1/4 Cr - 1 Mo). Some of the internals are fabricated from Incoloy 800 (wastage baffles) and 304 stainless steel (thermal liner), accounting for the nickel. The formation of the residue is believed to be caused by the formation of an oxide scale on the internals during fabrication and post-weld heat treatment. During sodium service, the oxides are chemically reduced leaving a residue of the observed composition. The results observed by the SEM examination are consistent with this theory. Similar observations of a black residue were noted in Ref. 6 after alcohol cleaning of a steam generator. Conclusions as to formation and composition were similar to those presented here.

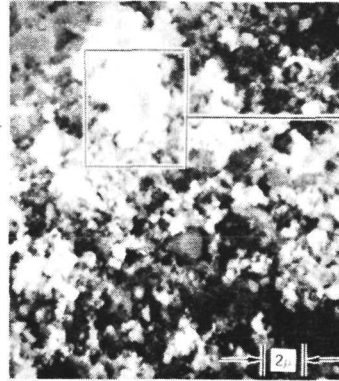
During cleaning of the filter element, an estimate of the residue volume was made (16 in³). In addition, the bulk density of the residue was measured (1.1 g/ml). If it is assumed that there is twice as much residue remaining on the MSG internal surfaces as collected in the filter, the total



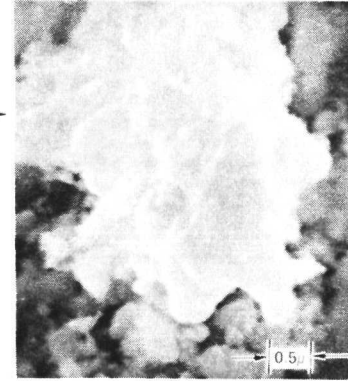
20X
a. TYPICAL AGGLOMERATED POWDER



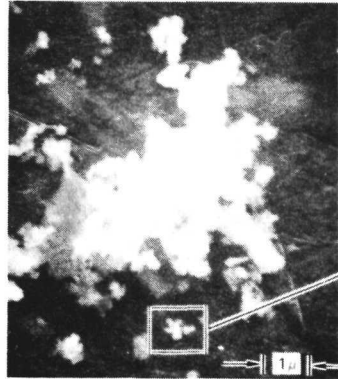
1000X
b. SMALL AGGLOMERATE IN SAMPLE



5000X
c. DETAIL OF LARGE AND SMALL AGGLOMERATES



20,000 X
d. SURFACE CONTOUR OF ONE OF THE TWO PARTICLE SHAPES



10,000X
e. SMALL FLECK FROM AGGLOMERATES



50,000X
f. DETAIL OF SMALL PARTICLES

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DEPOSITS

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mass of residue formed would be about 1.9 lbs. This amount represents a uniform corrosion of the internal surface of only 0.02 mils.

H. Visual Examination

Following cleaning, the MSG was removed from the SCTI and moved to another location for post test examination and modification as the first test article in the Large Leak Test Rig (LLTR). Detailed results of post test examination will be covered in a subsequent report. This section presents a summary of the visual inspection conducted of selected internal surfaces viewed during disassembly. Figure VI-9 shows a sketch of the MSG and the disassembly performed for visual inspection. A summary of the internal surfaces that could be viewed during the various disassembly steps is presented in Table VI-2.

Inspection of internal surfaces showed a general coating of a black residue. In areas where the solvent flow was nearly stagnant and not accessible to sparge gas flow, the residue was much thicker. These thicker regions were in the form of a cake with thicknesses up to about 1/8-in. The black deposits were analyzed and found to be the same composition as that obtained from the filter during cleaning. Wet chemistry analysis showed that the sodium content of the residue was between 1 and 2%. X-ray defraction analyses indicated the crystalline structure to be that of sodium ethoxide and sodium hydroxide. The sodium ethoxide, the reaction product of the sodium-ethanol reaction, remains as a surface contamination after drying. The sodium hydroxide was apparently formed from the reaction of sodium-ethoxide and moist air after the MSG was exposed to the atmosphere. In areas where the cake residue occurred, a reddish-black deposit was also noted. These deposits were shown to contain, in addition to alpha-ion as observed in all other residue samples, the compound Fe_3O_4 (magnetite). Only small amounts of this compound were noted on the MSG internals,

Figure VI-10 shows various photographs of the MSG internals. Figure VI-10c shows an overall view of the MSG during disassembly and inspection. At the time of this photo, the feedwater and steam heads and rupture disc nozzle had been removed. Work is proceeding on the removal of elbow section and sodium inlet nozzle cap.

FIG. VI-9
MSG DISASSEMBLY FOR VISUAL INSPECTION

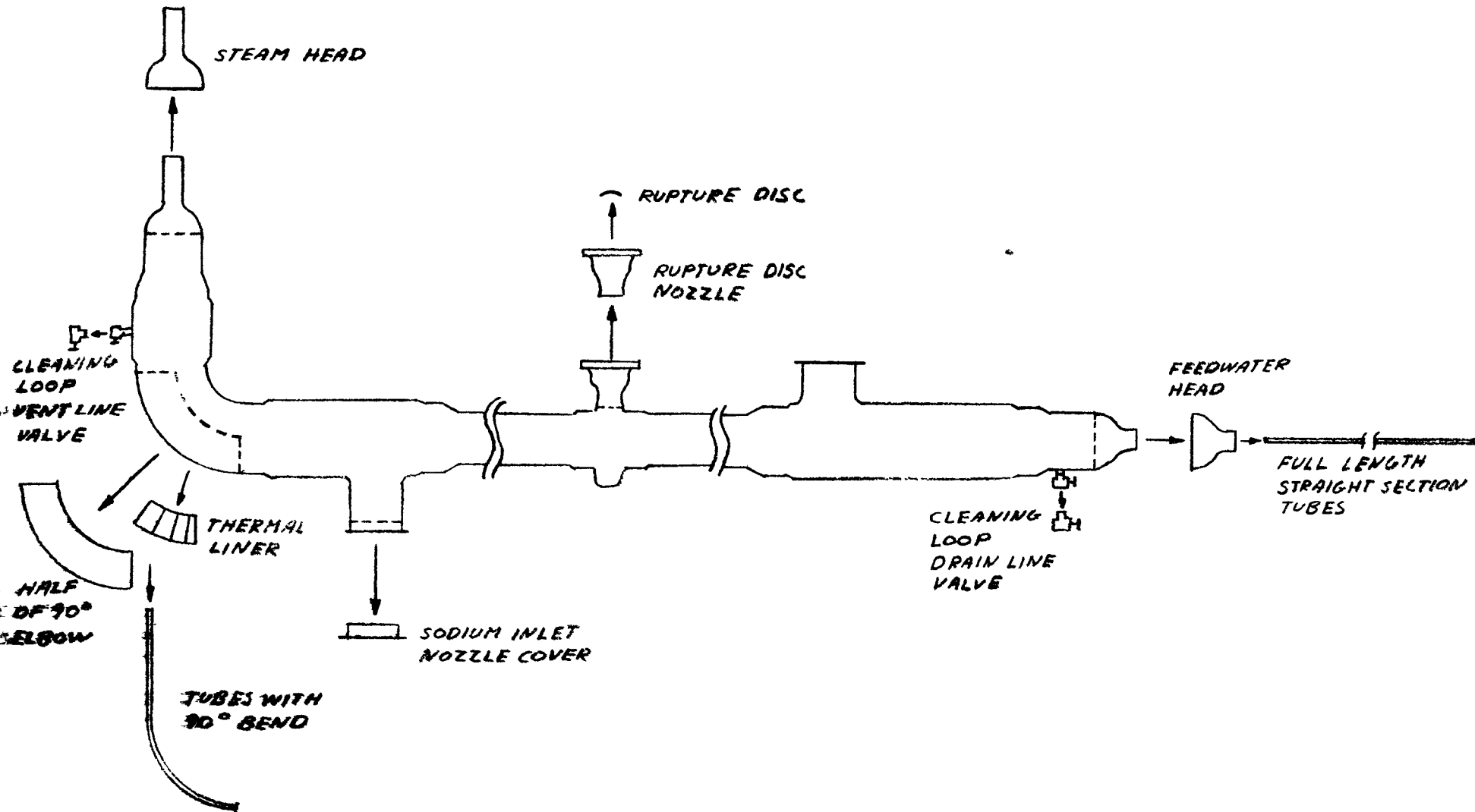
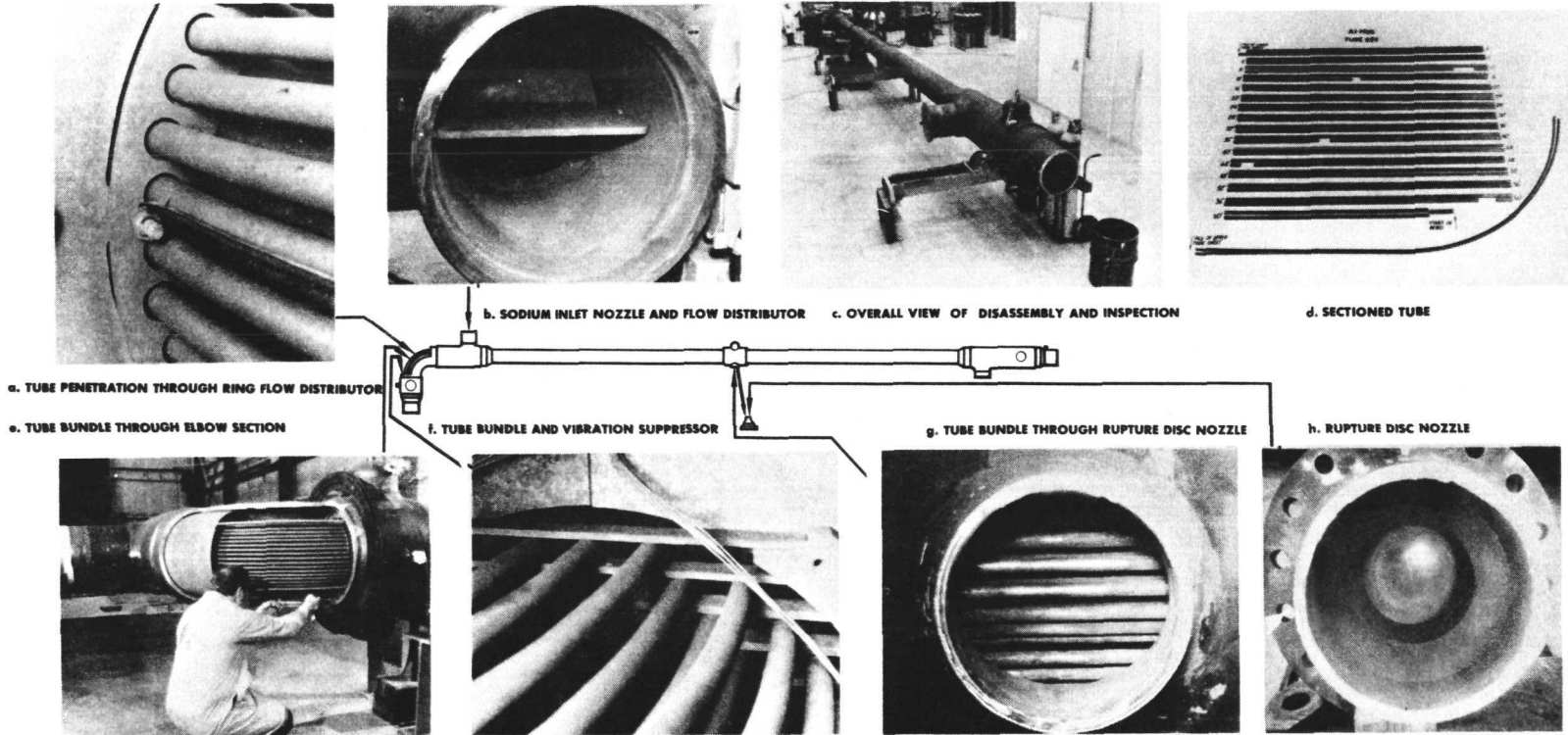


TABLE VI-2

Internal Surface Visual Inspection

<u>Disassembly</u>	<u>Surfaces Inspected</u>
Cleaning Loop Drain Line Valve	Limited viewing by borescope of tube external surfaces and upper face of thermal baffle plates
Cleaning Loop Vent Line Valve	Limited viewing by borescope of tube external surfaces
Rupture Disc Nozzle	Internal surface of nozzle, tube bundle and tie rod external surface, lower face of one and upper face of another tube spacer by borescope
Rupture Disc	External surfaces and crevice formed during fabrication
Sodium Inlet Nozzle Cover	Internal surface of nozzle and external surface of a limited portion of the upper flow distributor
Half of 90° Elbow and Thermal Liner	External surface of tube bundle in bend region, complete thermal liner including bolt connections, upper surface of ring flow restricter and tube spacer, upper surface of support ring, vibration suppressor, internal surfaces of shell, limited viewing of face to upper tube sheet
Tubes	Full length external surface at tubes. Internal surface inspection to be covered in separate report
Feedwater Head	Internal surface of holes through tube sheet by borescope - to be covered in separate report
Steam Head	Same as above

MSG SODIUM SIDE INSPECTION



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Photo VI-10g shows a view looking into the tube bundle at the rupture disc. The thin black residue film, described above, can be noted on the surfaces. Several sections were wiped with acetone exposing the shiny base metal. A borescope examination of two adjacent tube spacers (not shown) was made. No signs of unreacted sodium were observed, even in the 0.006 in radial gap between the tubes and tube spacer holes. The tubes could be moved in this radial gap giving a noticeable metal to metal contact sound. A view looking into the sodium side of the rupture disc nozzle is shown in Photo VI-10h. Again, the thin black residue film is visible, but no signs of unreacted sodium were observed. The shiny surface on the rupture disc is an area wiped with acetone exposing the base metal. The cross marks noted are the back side of the grooves on the disc for providing the proper rupture pressure. The disc was removed from the nozzle by grinding the lip formed when the disc was welded to the nozzle. A rather deep crevice was exposed showing no signs of unreacted sodium.

Photo VI-10b shows a view looking into the sodium inlet nozzle after the weld cap was removed. The flow baffle plate and the outside surface of the upper flow distributor can be seen. The thicker residue cake (1/16 to 1/8 inch) can be observed on the lower surface (right side of photo) of the nozzle. The thicker layer on the lower surface is postulated to have occurred, as discussed above, as a result of the solvent flow characteristics in the nozzle region during cleaning. There was no solvent flow through the sodium nozzle during cleaning, which results in essentially stagnant flow conditions between the flow distributor and nozzle. Also sparge gas flow could not agitate the solvent in this region. Because of these stagnant conditions, the black residue picked up by the solvent would tend to settle out on the lower surface of the nozzle. During cutting of the nozzle to remove the weld cap, sparks were observed, indicating the possibility of unreacted sodium. Inspection of the internals revealed no signs of unreacted sodium. The sparks that were observed are postulated to have occurred from residual ethanol which condensed on the cold uninsulated sodium nozzle.

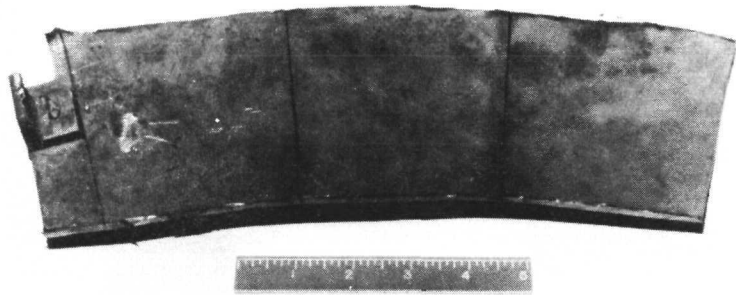
Photo IV-10e shows a view of the tube bundle after the elbow section was removed. The thermal liner assembly can be seen, which was later removed. A portion of the vibration suppressor is visible as well as some of the internal thermocouples. The photo shows residue samples being taken for chemical analysis. Photo IV-10f shows a closeup of the vibration suppressor. The metallic particles noted on top of the suppressor are cutting chips from the elbow removal operation. A bolt hole can also be noted which was completely free of sodium. Photo IV-10a shows a closeup of the tubes as they pass through the ring flow restrictor after the thermal liner was removed. A portion of a tube spacer is visible behind the flow restrictor. Also shown is one of the eight tie rod hex nuts and a thermocouple. One of the thermocouple bands, that hold the TC's to the tubes, was removed and inspected for unreacted sodium. No trace of sodium was found under the band as well as any of the areas accessible for inspection in the upper bend region.

The final photo (VI-10d) shows the external surface of a full length tube which had been split and sectioned into about four-foot lengths. The black residue was noted only on certain portions of the tube length. These portions corresponded to the lower region, rupture disc nozzle region, and up bend region. These regions in turn correspond to areas that were subjected to post-weld heat treatment after the tubes were installed. This observation substantiates the theory that the residue originated from an oxide layer formed during post-weld heat treatment, and then chemically reduced during sodium service. Since the residue is magnetic, it tends to adhere to the metal surface. Close inspection of the tube surface revealed no signs of unreacted sodium. Location of areas where the tube passed through the tube spacer proved to be very difficult, because of clean appearance of the tube surface.

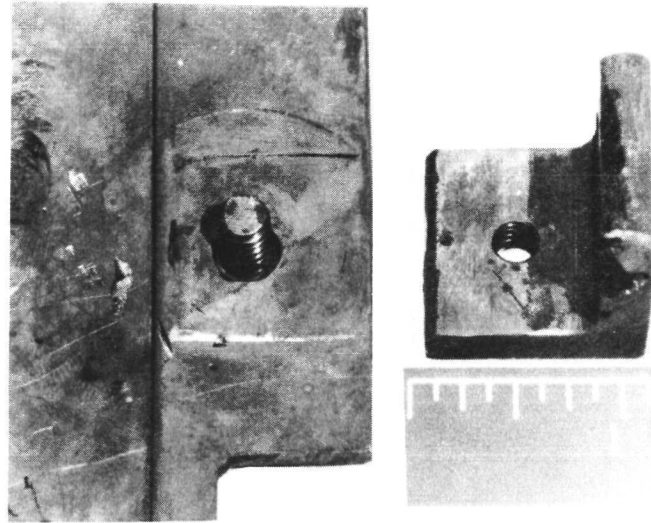
After disassembly of the thermal liner (shown in Figure VI-10e), several bolt connections were inspected. Figure VI-11 shows photos of one such bolt connection to a support bracket. Photo VI-11a shows a section of the thermal liner with the bracket bolt connection. Photo VI-11b

MSG SODIUM REMOVAL - POST CLEANING CONDITION OF BOLT THREADS AND CREVICES

Figure VI-II



A. THERMAL LINER SECTION AND BRACKET



B. EXPOSED BOLT THREAD

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shows the exposed bolt threads with the bracket removed. The underside of the bolt head (not shown) was completely (360°) seal welded to the liner. No unreacted sodium was found either in the opening between the bolt and hole in the liner or on any of the threads.

During borescope viewing through the one-inch vent and drain lines, only the thin residue film could be observed. No unreacted sodium in any area was found. Viewing of the top surface of the lower thermal baffle plates revealed no signs of any cake deposits. The crevices between the holes in the plates and the tubes were completely clean, with no indication of any sodium or other residue deposits.

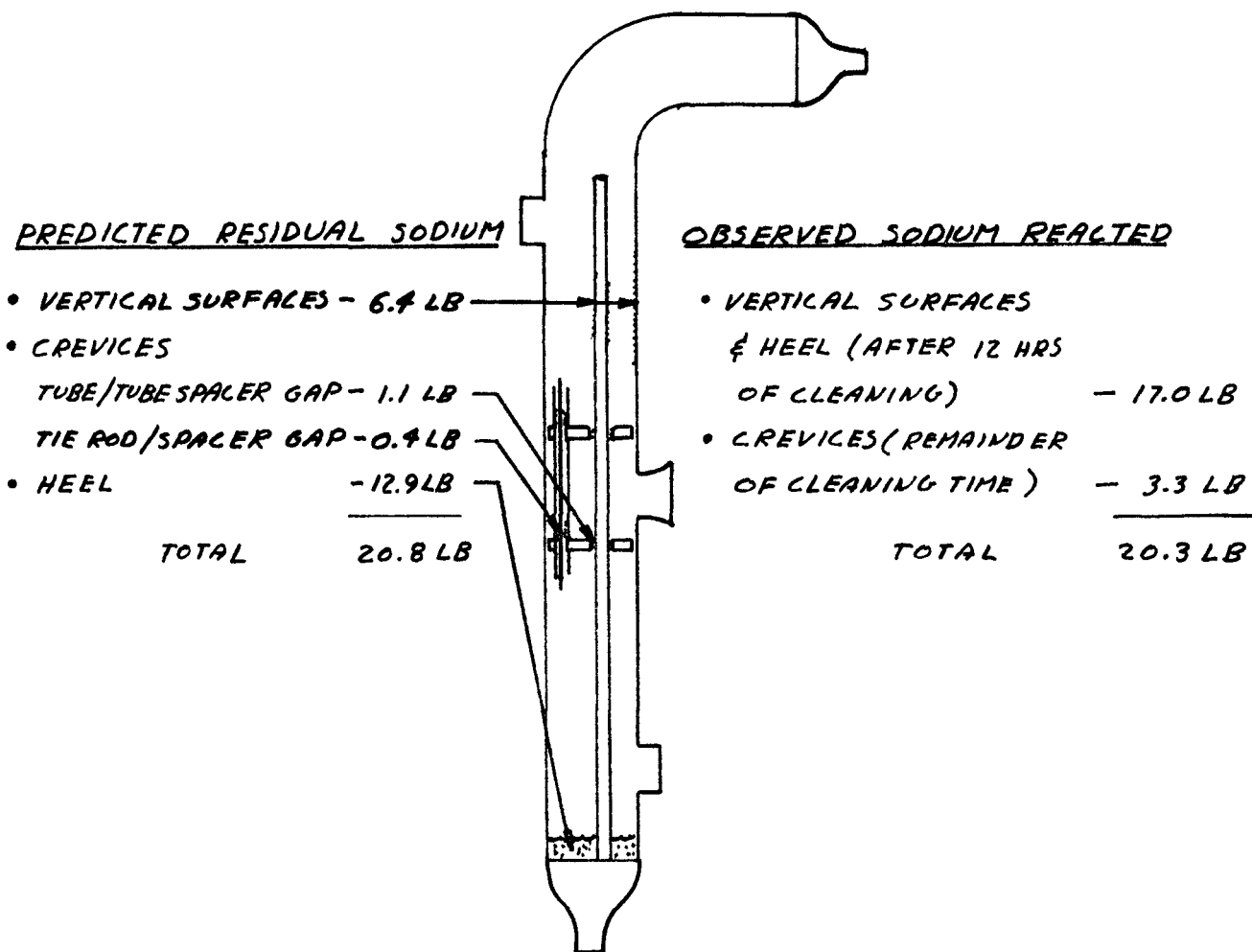
I. CLEANING RESULTS SUMMARY

A temporary cleaning loop was installed and the residual sodium was removed while the MSG remained located in place at the SCTI. Cleaning was performed using three solvent flushes; one Dowanol PM, and two ethanol. A summary of the sodium removed along with the predicted residual sodium is given in Figure VI-12. A total of 20.3 lb of sodium was estimated to have been removed during the cleaning process. This value compares to a predicted initial value of 20.8 lb remaining after sodium draining.

Visual inspection of internal surfaces showed no evidence of any unreacted sodium, even in crevice and bolt thread regions. A black residue was observed on internal surfaces and also collected in a filter during the initial cleaning stages. The residue was identified to be primarily the base metal, 2-1/4 Cr - 1 Mo ferritic steel, formed by chemical reduction of a surface oxide coating during sodium service.

The alcohol process used for cleaning the MSG proved to be very safe and highly effective in removing the residual sodium from sodium coated surfaces. The long cleaning time required to remove the sodium remaining after draining was caused by the many small crevices within the MSG, which resulted in diffusion controlled reaction rates. The design of the steam generators for the demonstration plate incorporates features that minimizes the crevices, and should result in a significantly reduced cleaning time.

FIG. VI-12
MSG CLEANING SUMMARY



CLEANING HISTORY

<u>SOLVENT</u>	<u>CLEANING TIME (DAYS)</u>	<u>SODIUM REACTED (LB)</u>
DOWANOL PM		
INITIAL	0.5	17.0
REMAINDER	10.5	1.3
FIRST ETHANOL	18	1.7
SECOND ETHANOL	7	0.3
TOTAL	36	20.3

VII REFERENCES

1. T. G. Spraul, "MSG Cleaning Instructions," TI-097-830-001, Nov. 8, 1973.
2. Informal Communication with A. E. Stewart, October 1973.
3. "Sodium Components Test Installation Special Procedure - AI Modular Steam Generator Cleaning System Cleaning," SCTI-SP-318, Rev. B, Nov. 28, 1973.
4. "Sodium Components Test Installation Special Procedure - Removal of Residual Sodium from AI Modular Steam Generator," SCTI-SP-312, or PP-04-PE-001, Rev. A, Dec. 3, 1973.
5. "RDT Standard - Cleaning and Cleanliness Requirements for Nuclear Components," RDT F 5-1T, Feb. 1972, Including Amendment 1 (April 1972) and Amendment 2 (March 1973).
6. J. M. Scott, et al., "Sodium-Heated Steam Generator Model Tests at SGTR, Volume 1, Test Rig Operating Experience and Cleaning, Disassembly, and Inspection of the Steam Generator Test Models," GEAP-10580-1, June 1972.