MASTER

HISTORY OF THE WATER CHEMISTRY
FOR THE FEW TUBE TEST MODEL

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ABSTRACT

This paper describes the water chemistry activities carried out in support of the Few Tube Test. This test was conducted to provide design confirmation data for the Clinch River Breeder Reactor Project (CRBRP) steam generators. Proposed CRBRP chemistry was followed; all volatile treatment (AVT) of water was carried out with on-line monitoring capability.

HISTORY OF THE WATER CHEMISTRY
FOR THE FEW TUBE TEST MODEL

SUMMARY

The Few Tube Test Model (FTTM) program involved in the testing of a 7 tube, full length model of the evaporator and a 7 tube (3 active tubes), full length model of the superheater for the Clinch River Breeder Reactor Project (CRBRP).

Water chemistry requirements were based on CRBRP conditions. City water was pre-treated by filtration, demineralization and deaeration processes before entering the high pressure system. An all-volatile treatment (AVT) specified for CRBRP using hydrazine and ammonia was followed. Unwanted ions in the recirculation water were removed by a mixed bed of hydrogen and hydroxide resin.

On-line instrumentation provided continuous monitoring of dissolved oxygen, pH total conductivity, cation conductivity, sodium ion concentration and hydrazine concentration from various sampling points. The instrument readings were cross-checked by benchtop analyses.

The 1915 hours of FTTM steaming experience have shown that the water chemistry specifications proposed for use in the CRBRP can be maintained. There were, however, a few occurrences of certain ionic species being measured at a higher than specified concentration, but prompt corrective action always restored the total water chemistry to the proposed specification.

1.0 INTRODUCTION

The Steam Generator Few Tube Test Model program was part of an overall testing plan being implemented in support of the Clinch River Breeder Reactor Project (CRBRP) steam generator design.

The Few Tube Test Model (FTTM) program involved the testing of a full length "hockey stick" evaporator with 7 active heat transfer tubes and a superheater with 3 active heat transfer tubes. The models were prototypical to CRBRP with respect to tube size, tube support devices, weld joints and configuration. This test would generate the first thermal performance and hydraulic stability data in a multiple-tube configuration, as well as the first long term endurance data under prototypical conditions.

The overall scope of FTTM consisted of steady-state thermal performance testing, long term endurance testing, and post-test examination.

To maximize the operating time while collecting data, proper attention must be given to the water treatment in order to minimize corrosion and scaling of the system. If the heat transfer surfaces are allowed to become fouled by corrosion and scale, the dependability and efficiency of the system would be substantially decreased. In keeping with the prototypical design of FTTM, the water chemistry requirements were based on CRBRP conditions (Table 1). On July 31, 1978, steaming operations were initiated in the General Electric Company Steam Generator Test Rig (SGTR), San Jose, California. Figure 1 shows a simplified schematic of the SGTR water/steam systems.

Steaming operations, in conjunction with thermal/hydraulic testing continued until December 19, 1978, at which time the SGTR was shut down. Subsequently, in 1979, the FTTMs were removed from the SGTR and destructively examined at the manufacturer's site in support of the plant steam generator design evaluation.

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Figure 1. Flow Diagram of SGTR Water/Steam Systems

2.0 WATER TREATMENT

Pre-treatment of San Jose city water is performed by the low-pressure water system, comprised of the demineralized water system and the deaeration system. The filtering and demineralization are performed by the demineralized water supply system. The raw water is run through mixed resin beds; exiting water quality is $<1\mu\text{mho/cm}$. From the resin beds the water goes to a 450 gallon holding tank. The holding tank is part of a closed loop system where water circulated through an additional mixed bed resin. From the holding tank, loop system water $<0.3\mu\text{mho/cm}$, goes to the deaeration system comprised of three 200 gallon tanks. Each tank has a nitrogen covergas and is equipped with 18 kw of electrical heat. The tanks automatically fill, boil and then "go into service". The "on-line" tank is connected to the inlet of the makeup pump and is used until a low level signal switches to the next "ready tank". The empty tank is then filled, boiled and placed on standby. The low pressure water system is equipped with an alarm system which provides a local alarm whenever preset conductivity limits are exceeded. There are alarms for the demineralized water exiting the initial resin beds, the demineralized water in the holding tank, the total conductivity of the sample, cation conductivity of the sample and make-up water conductivity. All action taken as a result of an alarm is initiated by an operator and/or the chemist.

The following table shows the specification for the purity of the demineralized water to be produced.

Table I
Make-up Water Specification

Total Dissolved Solids	50 ppb (max)
Sodium Ion	1 ppb (max)
Conductivity	0.3 μmho (max)
Filtration	to 5 microns (ave)

A laboratory versus on-line instrumentation cross-check was periodically made on the conductivity and the sodium ion level using benchtop analytical techniques. Make-up water from the deaeration tanks was analyzed for oxygen content using grab samples prior to placing a tank "on-line".

Even though the recirculation water is of high purity, it can pick up

trace impurities which at high temperatures can be troublesome. Therefore, these impurities were removed by passing the recirculation water through a polishing system to remove unwanted ions. A mixed bed of hydrogen and hydroxide exchange resin was used. During normal operations, a hydrazine-ammonia solution was automatically pumped into the system whenever the system's make-up pump switched on. The ammonia, adjusting the pH of the recirculation water to minimize corrosion, is rapidly consumed by the cation exchange resin.

The operation was continued beyond the ammonia breakpoint (the point at which ammonia is contained in the effluent), reducing the removal of ammonia during the service cycle and greatly extending the length of the cycle.

3.0 SAMPLING LOCATIONS

Continuous monitoring of the water chemistry takes place within an instrumentation panel located in the SGTR chemistry laboratory via sampling point lines that extend from the loop area to the laboratory.

The sampling points for the continuous monitoring on the high pressure water system consist of the steam drum (saturated steam), hot well, evaporator inlet and deaeration tanks (make-up water). The sample, upon reaching the laboratory, is cooled and reheated to a controlled temperature of 75°F, then split among the water chemistry monitoring instruments. Water exiting from all the instruments is run to drain.

There is also a sampling line available for grab sample analyses enabling benchtop verification of the on-line analyses. Figure 2 depicts the plumbing layout of the sampling system.

4.0 INSTRUMENTATION

Prior to start-up all of the instrumentation underwent calibration using procedure STPI 04.50.159 (Appendix 1), and were periodically calibrated to maintain a high level of reliability in the readings. The oxygen monitor was only air calibrated at this time since a sample flow is not established until after start-up. Figure 3 shows the layout of the instrumentation as

Figure 2. Layout of Sampling System

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Figure 3. On-Line Water Chemistry Instrument Panel

observed from the front panel.

The panel instrumentation consists of:

- (1) Leeds and Northrup Sodium Ion Analyzer
- (2) Leeds and Northrup Calibrating Facility
- (3) Leeds and Northrup Sodium Monitor
- (4) Beckman Hydrazine Analyzer
- (5) Larson Lane Condensate Analyzer (Cation Conductivity)
- (6) Beckman Oxygen Monitor
- (7) Leeds and Northrup Six-point Conductivity Recorder
- (8) G.E. Single Pen Recorders (Four)
- (9) Leeds and Northrup pH Recorder/Electrode

As a means of verifying the functioning of the hydrazine analyzer and the oxygen monitor, benchtop cross-checks were performed using CHEMETS, vacuum sealed ampoules containing specific reagents for specific analyses. The chemical reactions are based on ASTM procedures thereby not compromising the quality or basis for comparison of the results.

A Beckman pH meter and Markson portable conductivity meter were used for benchtop pH and conductivity analyses. Total solids were not analyzed in this phase of the project because the evaporative sampler was not functioning.

For calibration of the on-line sodium analyzer and for bench-top sodium analyses, an Instrumentation Laboratories Atomic Absorption/Atomic Emission spectrophotometer based on flame emission technique was utilized. This instrument can also be used for benchtop analyses of copper, nickel, chromium and iron.

5.0 PROCEDURE

Corrosion is minimized when the make-up and condensate water are free of gases and maintained at the proper pH level to reduce iron or copper pickup. In addition to the deaeration process, the water is treated chemically to achieve these conditions. The all volatile treatment (AVT) system specified for CRBRP was followed for maintaining the pH and oxygen concentration. The AVT for FTTM consisted of hydrazine and ammonia injections from the chemical injection system. The hydrazine scavenges oxygen while the ammonia maintains the proper pH level.

The AVT chemicals are prepared in a five-gallon polyethylene bottle and pumped into the loop using the chemical injection system which is located in the back of Bay 1 (Figure 2). The system can be turned on manually or set to switch on/off automatically when the make-up pump turns on/off. Chemicals were injected on an "as needed" basis when a drop in pH, a slight increase in oxygen or a hydrazine level drop was noticed. In the future, however, the system will be set up to continuously inject a more dilute concentration of the chemicals at a low pump stroke into the loop area so that the pH and hydrazine level remain essentially constant at all times.

The on-line instrumentation provide a continuous measurement of residual hydrazine, pH, dissolved oxygen level, total conductivity, cation conductivity (after removal of cations) and sodium concentration. Analyzer output signals were displayed continuously on strip chart recorders and readings were recorded in a log book on the average of four times during the day shift and every four hours during off-shift hours and weekends on a log sheet. If a particular situation dictated the need for more frequent readings, this would be carried out until a "normal" situation had been achieved. Immediately after the on-line readings were taken, benchtop analyses were performed for verification during the day shift. The oxygen, pH, hydrazine, sodium and conductivities were monitored very closely. Experience has shown that it takes from 8-24 hours for the oxygen readings to stabilize. However, once stabilized, the readings remain fairly constant below the specification limit.

6.0 EXPERIENCE

Table 2, CRBRP Water Chemistry Specification, outlines the parameters that were tracked and the specifications.

Sampling was accomplished primarily from the Recirculation line although other points were periodically monitored. Table 3 shows a monthly average for the parameters analyzed. Comparing the results to Table 2, it can be seen that the water chemistry for FTTM was very much within specification.

Table 4 lists the various times that the system was down or sampling in the laboratory had to be secured. The most redundant contributor to down time was the condensate feed pump.

TABLE 2

CRBRP Water Chemistry Specifications

<u>Parameter</u>	<u>Units</u>	<u>Preheat and Operation Below 5% Power</u>	<u>Operation Above 5% Power</u>	<u>Mandatory Operator Shutdown</u>
<u>Allowable Feedwater Impurities</u>				
Solids, Suspended max.	ppb	N/A	16	N/A
Dissolved Oxygen, max.	ppb	10	7	100
Silica, max.	ppb	30	20	N/A
Iron, max.	ppb	50	10	N/A
Copper, max.	ppb	10	<1.5	N/A
Hydrazine, min.	ppb	100	5	N/A
Sodium, max.	ppb	N/A	1	N/A
pH @ 77°F, range	N/A	8.6 - 9.6	8.7 - 9.1	<8.0
Cation Conductivity @ 77°F, max.	μmho/cm	N/A	0.3	N/A
Chlorides, max.	ppb	N/A	9	N/A
<u>Allowable Recirculation Water Impurities</u>				
Chlorides, max.	ppb	100	50	150
Solids, Suspended, max.	ppb	250	90	90
Sodium, max.	ppb	60	6	30
pH, range	N/A	8.5 - 9.6	8.5 - 9.0	<8.0
Cation Conductivity @ 77°F, max.	μmho/cm	6.0	2.0	9.0

TABLE 3

Monthly Average of Water Chemistry Parameters

<u>Month</u>	<u>Hydrazine</u>	<u>Oxygen</u>	<u>Total Conductivity</u>	<u>Cation Conductivity</u>	<u>Sodium</u>	<u>pH</u>
August	100 ppb	2 ppb ⁽¹⁾	14 μ mho	2.4 μ mho	30 ppb	9.0
September	100 ppb	4 ppb	20 μ mho	1.7 μ mho	11 ppb	9.5
October	100 ppb	4 ppb	19 μ mho	1.1 μ mho	5 ppb	9.6
November	100 ppb	4 ppb	16 μ mho	1.5 μ mho	4 ppb	9.5
December	100 ppb	4 ppb ⁽²⁾	5.5 μ mho	1.4 μ mho	5 ppb	8.8

⁽¹⁾ Excludes initial high oxygen readings.

⁽²⁾ Excludes erratic readings due to deaerator problem.

TABLE 4

FTTM Down Time

<u>Date System Down</u>	<u>Date System Up</u>	<u>Down Time</u>	<u>Reason</u>
08/20/78*	08/21/78	20½ Hours	Flush DA tanks 1365/1366.
08/23/78*	08/24/78	11 Hours	Repair of coolers. HPD resin bed change.
08/27/78	09/18/78	3 Weeks	Condensate feed pump down.
09/26/78	09/26/78	5 Hours	Repacking of condensate feed pump.
10/05/78	10/16/78	10 Days	Repair of condensate feed pump.
10/20/78	11/06/78	16 Days	Hardware problem in loop area.
11/13/78	11/22/78	9 Days	Repair of condensate feed pump.
12/04/78	12/04/78	7½ Hours	Repair of condensate feed pump.
12/05/78*	12/05/78	6½ Hours	Hardware problem in loop area.
12/06/78	12/06/78	8½ Hours	Sampling secured due to electrical problem on the deaeration system.
12/07/78	12/07/78	5½ Hours	Repair of condensate feed pump.
12/11/78*	12/12/78	11 Hours	Drain and flush of DA tanks 1365/1366.
12/12/78*	12/12/78	7½ Hours	Drain and flush of DA tanks 1365/1366.
12/13/78	12/13/78	13½ Hours	System drained for orifice change. Refill in progress 8 hours after drain.
12/18/78*	12/18/78	5½ Hours	Make-up tank problems.

*Sampling secured in Chamistry Laboratory only.

Throughout the operation period from July 31, 1978 - December 19, 1978, relatively few problems were encountered in maintaining the specified water chemistry for FTTM except for higher than specified pH. The high pH resulted from the mechanics of the AVT injection system. Modifications to this system have been completed to allow for better control. Other problems that did surface are discussed below.

6.1 High Sodium Concentration

6.1.1 During the initial start-up, loop operators were requested to place two high pressure demineralizers on line. The sodium concentration in the feedwater increased to > 1000 ppb. It was discovered, after analyzing samples from various points, that the source of the problem was the demineralizer resin. The resin was sodium-form instead of hydrogen form. After replacement with the hydrogen form resin, the sodium concentration gradually came within specification. During subsequent start-ups, the sodium concentration would initially start high but drop within specification limits within 1-2 days by increased blowdown and demineralization. At no time during the high sodium concentration events was the evaporator allowed to experience departure from nucleate boiling. Power levels were controlled to less than 5%.

6.1.2 Deaeration tanks 1366 and 1367 have a propensity for sodium ion buildup because of a prior contamination problem. These tanks were periodically drained, flushed and re-filled to lower the sodium ion concentration. Deaeration tank 4000, however, did not present this problem. The problem can be controlled, however, by periodic flushing.

6.2 High Oxygen

6.2.1 Experience has shown that during start-up or after the on-line sampling has been restarted after a brief shutdown, the oxygen level tends to be high until equilibrium is established. For a start-up, this may take 8-24 hours; for a brief shutdown, 15 minutes to 8 hours.

6.2.2 There was one occurrence in which the oxygen level was erratic for over 24 hours. Hydrazine analysis showed that the water was heavily concentrated yet the oxygen level would continuously change from a 0 (zero) ppb level to an off-scale reading. It was discovered that there

was an electrical malfunction in the deaeration system and the water in the tanks was not going through a complete "boil" cycle. After this problem was corrected, the oxygen level dropped to 1-2 ppb.

6.3 Sodium Ion Analyzer

6.3.1 The sodium ion analyzer worked reasonably well for about three months until the dimethylamine gas (DMA), which is used to depress the hydrogen ion level in the sample to allow a sodium reading, eroded a screw cap at the top of the flowmeter in the assembly, allowing the gas to escape into the air. A replacement was ordered and installed, but the gas still escaped. The instrument was taken off line and sodium analyses were conducted by flame emission only. Since the sodium analyzer is calibrated from the results of flame emission analysis, this practice did not effect the reliability of the sodium analysis--only the frequency.

6.4 pH Control

6.4.1 During the last few weeks of operation, only hydrazine was added to the water to maintain the proper chemistry. This procedure worked fine at low power levels, but during high power conditions the hydrazine thermally decomposed into ammonia, nitrogen and hydrogen--the ammonia causing the pH level to rise rapidly. This problem was solved by a combination of a continuous blowdown (turn sampling line on continuously), venting, and placing additional high pressure demineralizers on line.

6.5 Start-up Control

During each start-up, a "black-water" condition was experienced. The conductivity readings (total and cation), sodium concentration and suspended solids increased until the sample temperature reached a 400°F to 500°F range. This condition lasted about 1-2 hours until the system reached equilibrium.

6.5.1 For the benefit of the loop operators, who log in the chemistry readings during off-shift hours or weekends, a chart was prepared to enable the operator to attempt to solve the problem of getting a particular parameter into specification if it appeared to be "approaching an out-of-spec. region." In the event that the chemistry support personnel or the Operations Engineer could not be immediately contacted, the operator did have an alternative to troubleshoot, using the chart as a guide. (Appendix II)

7.0 FUTURE PLANS

Monitoring for chloride and copper was not carried out in this test, but will be performed in any future testing, utilizing ion chromatography, a new analytical technique.

Ion chromatography combines conductivity detection with ion exchange separation. It involves using a system of two columns in series, the first column separates the ions of interest, and the second column serves to remove the background reference of the eluting ions while converting sample species to a common, highly conductive form which can be directly monitored with a conductivity detector.

This analytical tool will also be used to measure Cl^- , F^- , SO_4^{2-} , NO_3^- , PO_4^{3-} , Na^+ , K^+ , Cu^{++} , and Ca^{++} in the makeup and recirculation water.

8.0 CONCLUSION

The FTTM water chemistry has been maintained within the desired specifications proposed for use in the CRBRP.

There were several short periods during startup when some ionic impurities measured at higher concentration levels over those specified. Prompt corrective action was taken, and in all cases, the correct water chemistry purity was restored. The analytical techniques and instrumentation used for on-line monitoring of selected ionic species and pH levels performed well for the 1915 hours of steaming in this first phase.

SODIUM TECHNOLOGY PROCESS INSTRUCTION

PREPARED BY:

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APPROVED-MGR-STE:

J.M. Scott

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TITLE:

SGFTT WATER CHEMISTRY INSTRUMENTATION CALIBRATION and CHECK-OUT

1. PURPOSE

This document outlines the procedures to be used in calibrating the instrumentation in the Water Chemistry Laboratory. The calibration will include the hydrazine analyzer, dissolved oxygen monitor, sodium-ion analyzer, pH meter and conductivity meter on-line instruments and the laboratory atomic absorption/flame-emission spectrophotometer.

2. REFERENCE DOCUMENTS

2.1 Book of ASTM Standards - Part 31

2.1.1 ASTM D1385-67 (Standard Method of Test for Hydrazine in Water)

2.1.2 ASTM D888-66 (Standard Method of Test for Dissolved Oxygen in Water -- Colorimetric Indigo Carmine)

2.1.3 ASTM D1125-64 (Standard Method of Test for Electrical Conductivity in Water).

2.1.4 ASTM D1293-65 (Standard Method of Test for pH of water).

2.2 Instrumentation Laboratories Instruction Manual for 151 Atomic Absorptive/Emission Spectrophotometer.

2.3 Beckman Oxygen Monitor Manual Model 7001.

2.4 Beckman Hydrazine Analyzer Manual Model HM-2.

2.5 Leeds & Northrup Sodium-Ion Analyzer Direction Sheet #177625, Issue 4.

2.6 Leeds & Northrup Electrolytic Conductivity Cells Direction Sheet #177666, Issue 2.

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3. REQUIRED EQUIPMENT

3.1 Benchtop pH meter

3.2 Measuring Circuit (such as Wheatstone Bridge)

3.3 Demineralized Water System

3.3.1 Demineralized (deionized) water shall meet the requirements as outlined in RDT F5-IT for Grade A water. Table 1 lists the specifications.

TABLE 1

PROPERTY	VALUE
Chloride, ppm max.	0.1
Fluoride, ppm max.	0.1
Conductivity, μ mhos/cm max.	2.5
pH	6.0-8.0
pH (CO ₂ compensation)	5.8 min.
Turbidity, Jackson Candle	1.0
Units, max.	

3.3.2 Subsequent references in this document to the terms "DI water" or "water", shall mean Grade A demineralized (deionized) water.

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4. PROCEDURE

4.1 Calibration of Hydrazine Analyzer

- 4.1.1 Connect power to signal monitor.
- 4.1.2 Connect signal monitor and cell leads to terminal block in wet chemical section.
- 4.1.3 Open door to wet chemical section and pull out slide tray to the service position.
- 4.1.4 Insert the cell assembly into the sample cup by pushing straight down while rotating slightly. A few drops of water on the O-ring will facilitate assembly. NOTE: Care must be taken to keep electrode shield in place in order to avoid damage to the fragile ceramic thimble. Attach the black wire from the cell to Position 1 on the terminal block at the rear of the case.
- 4.1.5 Carefully unscrew the reservoir cap and set aside. Fill reservoir with 10% sodium hydroxide (NaOH) until the level reaches 5/8" below the top of the side arm. The reference electrode assembly is threaded into the side arm on the electrode assembly. (Fig. 1)
- 4.1.6 Install reference electrode assembly and continue filling the reservoir with caustic to about 1/2" from the top. Replace cap. Make sure the rubber insert and mating flange are clean and free from solid caustic soda to insure an airtight seal.
- 4.1.7 Attach red wire from reference electrode to Position 3 on the terminal block at the rear of the case.
- 4.1.8 Connect the left sample inlet to a source of DI water and right valve to the hydrazine solution. The hydrazine solution may be an on-line sample whose hydrazine concentration has been determined by ASTM 1385-67 (Standard Method of Test for Hydrazine in Water) or it may be a standard prepared per 4.1.30.

Part Description
for Figure 1

1. Slide tray
2. Reference electrode assembly
3. Slide arm on the electrode assembly
4. Reservoir cap
5. Reservoir
6. Sample stop valve
7. Sample chamber
8. Overflow tube
9. Flowmeter control valve
10. Flowmeter

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- 4.1.9 Open the left sample stop valve to the full open position, allowing sample chamber to fill and purge the overflow tube.
- 4.1.10 Open the flowmeter control valve fully to allow air to purge through the flowmeter and connecting tubing. Check tubing for air bubbles; purge further if necessary by squeezing the flexible tubing attached to the sample chamber.
- 4.1.11 After about 10 minutes of full flow, cut the flowmeter control valve down to obtain a reading of 1.0 GPH and partially close the sample stop valve to obtain approximately the same or somewhat less flow from the overflow tube.
- 4.1.12 Rinse down 4-6 hours in order to wash away any foreign material and to equilibrate flow between the caustic and sample reservoirs.
- 4.1.13 Turn range switch in wet chemical section to setting which will not be exceeded by expected hydrazine concentration.

<u>Range Switch Setting</u>	<u>Hydrazine Concentration Range</u>	<u>Instrument Meter Scale</u>
X3	0 - 30 ppb	0 - 3
X1	0 - 10 ppb	0 - 10
X10	0 - 100 ppb	0 - 10

INTERCONNECTION DIAGRAM
HYDRAZINE ANALYZER

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- 4.1.14 Check the mechanical zero of the meter with POWER OFF. If not at zero, set it with the screw on the face of the meter.
- 4.1.15 Turn the power switch on and the MODE switch to the ZERO position. (Red pilot light will come on.) Allow 2 minutes for stabilization. If meter reads other than zero, insert small screwdriver into the ZERO opening and turn the meter to zero.
- 4.1.16 Set Range switch in wet chemical section to appropriate setting.
- 4.1.17 Turn MODE switch to the CHECK position. If necessary, insert screwdriver into CHK opening on the monitor and turn to bring the meter pointer to 10 on the scale.
- 4.1.18 Check the flowmeter in the wet chemical compartment. If it has drifted from 1.0 GPH, adjust to 1.0 GPH.
- 4.1.19 Rotate MODE switch to the READ position. With DI water flowing, the meter will drop to zero. Close left valve and open right valve containing hydrazine solution (sample stream or standard solution prepared per 4.1.30 whose concentration is known).
- 4.1.20 Readjust flowmeter to 1.0 GPH.
- 4.1.21 Adjust the CALIBRATE control on the monitor TO THE CONCENTRATION OF THE HYDRAZINE SOLUTION.
- 4.1.22 Allow 10 minutes for stabilization.
- 4.1.23 Check flowmeter and adjust if necessary.
- 4.1.24 Lock the CALIBRATE control with the lock ring.
- 4.1.25 Close right valve and open left valve (DI water). The meter will drop to zero.
- 4.1.26 Close left valve. Reopen right valve. Meter should drive to value of hydrazine solution.
- 4.1.27 If necessary, readjust the CALIBRATE control and relock.

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4.1.28 Disconnect the DI water at the rear of the wet chemical section and connect sample stream. When right valve (sample stream or standard) is closed and the left valve is opened, the meter will indicate the hydrazine concentration in PPB.

4.1.29 Instead of using the sample stream, a solution of known hydrazine concentration may be connected to the right sample port for an external standard. Connect port with flexible tubing to a drain plastic container. Elevate container to allow the solution to flow by gravity. Prepare standard solution from the 100 ppm hydrazine solution supplied with the analyzer. To make up 3 liters of hydrazine solution, use the indicated amount of 100 ppm standard and dilute to 3 liters with hydrazine-free water.

<u>Volume of 100 PPM Standard</u>		<u>Final Concentration of Hydrazine Standard</u>
3.0 ml	} Diluted to 3 liters	100 ppb
1.5 ml		50 ppb
0.6 ml		20 ppb
0.3 ml		10 ppb

Only one concentration is needed for calibration.

4.1.30 Re-standardize once a week.

4.1.31 Keep caustic reservoir filled. Refill when level drops to 3/4" from bottom.

4.2 Calibration of Oxygen Monitor

4.2.1 Initial Calibration of Oxygen Monitor

4.2.1.1 Turn on line power

4.2.1.2 Verify that sensor membrane is dry. Expose sensor to ambient air at atmospheric pressure.

4.2.1.3 Set RANGE switch at AIR CALIBRATE and wait about one hour or less for a stable meter reading.

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- 4.2.1.4 Adjust CALIBRATE Control so that reading on the 0-100 scale of the front panel meter is equal to 1/10 the barometric pressure in mm Hg. Assume 760 mm Hg at sea level.
- 4.2.1.5 Install sensor in flow chamber. Set RANGE switch at 200 p/10⁹. Set water flow at 250 ml/min. and allow system to purge for 24 hours in order to clear all oxygen from the flow system and to consume all oxygen in the sensor electrolyte.
- 4.2.1.6 When meter reading on 200 p/10⁹ appears to be zero, move RANGE switch to lowest setting that provides an on-scale reading. If system is leak-free and thoroughly purged, the range selected will be 0 to 50 p/10⁹ (if the dissolved oxygen content of the sample does not exceed 50 p/10⁹).
- 4.2.1.7 The oxygen monitor is now approximately calibrated. Before the system is put into operation, exact calibration should be carried out per 4.2.2.

4.2.2 Exact Calibration of Oxygen Monitor

- 4.2.2.1 Calibration of the oxygen meter should be performed in conjunction with indigo carmine analysis (ASTM D 888-66 Standard Method of Test for Dissolved Oxygen in Water --Method A -- Colorimetric Indigo Carmine) of a grab sample taken under steady state conditions to insure stability of oxygen content.
- 4.2.2.2 Set RANGE SWITCH at lowest setting that provides an on-scale reading. Wait for reading to stabilize.
- 4.2.2.3 If initial calibration with air (4.2.1) has been performed, the CALIBRATE control should be very close to the correct setting. If CALIBRATE control has accidentally been moved out of adjustment, set it so the front panel meter indicates the "estimated" dissolved oxygen content of the sample.

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4.2.2.4 Take sample for chemical analysis, making a note of the meter reading. Make analysis as soon as possible.

4.2.2.5 Note meter reading. If there is no change from 4.2.2.3, adjust CALIBRATE Control so that the meter reading agrees with the analytical value obtained in 4.2.2.4. If there is a change in the reading from 4.2.2.3, use the following equation to determine the required meter setting.

$$\text{Required Meter Setting} = \frac{\text{Analytical Dissolved O}_2}{\text{Sampling Meter Reading}} \times \text{Meter Reading at Calibration}$$

4.2.3 Frequency of Calibration

4.2.3.1 Air calibrate the instrument daily for the first 3-5 days of operation until sensor has stabilized. After this, calibration is performed weekly until an appropriate interval is determined.

4.2.3.2 If instrument reading varies by more than 3 ppb, readjust the CALIBRATE control per 4.2.2.3 - 4.2.2.5.

4.3 Calibration of IL 151 Flame Emission Spectrophotometer

4.3.1 Unplug the hollow cathode lamps.

4.3.2 Insert nitrous oxide burner (See Manual).

4.3.3 Ignite flame.

4.3.3.1 At the air cylinder, set external oxidant pressure to 40 psig.

4.3.3.2 At the acetylene cylinder, set the external fuel pressure to 15 psig.

4.3.3.3 At the nitrous oxide cylinder, set the N₂O pressure to 40 psig.

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- 4.3.3.4 Place the gas control selector switch at the AIR position.
- 4.3.3.5 Use the oxidant flow control to reduce the oxidant pressure until the ball in the oxidant flowmeter stops dripping.
- 4.3.3.6 Place the aspirator tube in a graduated flask containing water.
- 4.3.3.7 Adjust aspiration rate control until the oxidant flowmeter reads 12 to 14 SCFH. A clockwise turn lowers the flow while a counterclockwise turn increases the flow. The aspiration rate, at this setting, should be 4-6 ml/minute. Check with graduated cylinder.
- 4.3.3.8 Place the gas flow selector to the AIR/FUEL position.
- 4.3.3.9 Set the fuel flowmeter to a setting of 3-5 SCFH with the fuel flow control. This causes the flow to increase to 10-14 SCFH when FUEL/N₂O is selected.
- 4.3.3.10 With gas selector at AIR/FUEL, press the pilot switch to ignite the air/acetylene flame. DO NOT ASPIRATE WATER.
- 4.3.4 Set the Readout Mode Selector switch to FE.
- 4.3.5 Refer to Appendix A for Standard Preparation.
- 4.3.6 Aspirate solution containing sodium. Use the Wavelength Selector to set monochromator to approximate emission peak on the Wavelength Indicator (589.0 nm for sodium).
- 4.3.7 Aspirate a blank (DI water). Press the Auto Zero Button. If the display does not go to zero, reduce the photomultiplier voltage.
- 4.3.8 Aspirate a "high standard". During aspiration, adjust the slit width and PM voltage until a workable readout is obtained.

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4.3.9 Select the Integration Mode and Period.

4.3.10 Aspirate another standard one-half as concentrated as that in 4.3.8 and check for linearity. Linearity correction may be obtained by (1) dilution of sample, (2) use of less sensitive spectral lines and (3) aspiration rate adjustment (reduction).

4.3.11 Set the Digital Display to the analytical concentration of the element in the Standard using the Scale Expansion Switch and the Decimal Position Button.

4.3.12 Aspirate Sample from loop and read concentration on the Digital Display. Repeat.

4.3.13 Record readings.

4.3.14 Using the concentrations determined by flame emission, calibrate the Sodium Ion Analyzer.

4.4 Calibration of Sodium Ion Analyzer

4.4.1 Startup

4.4.1.1 Close the valve at the top of the absorber column and the two valves BLANK and SPAN in the calibration facility. Set the selector in the calibration facility to MEASURE SPAN and the selector in the analyzer to MEASURE SAMPLE. Turn the SAMPLE REGULATOR knob counterclockwise about 12 turns.

4.4.1.2 Start the sample-water flow into the analyzer and adjust SAMPLE REGULATOR until the sample-flow rate indicated by the left hand flowmeter is 100 cc/minute.

4.4.1.3 Adjust the regulator on the dimethylamine cylinder for a delivery pressure of 3 psig and open the needle valve to supply the gas to the analyzer. Open the valve at the top of the absorber column until the gas flow indicated by the right hand flowmeter is 60 cc/minute.

4.4.1.4 Operate the analyzer "on stream" for several hours to purge the sample lines and analyzer of all incidental sodium ions.

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- 4.4.1.5 Perform the standardizing procedure after the analyzer has been on stream. On initial startup, make a slope adjustment.

IMPORTANT: Make certain that the sample water flows at all times to prevent escape of unabsorbed dimethylamine gas.

- 4.4.2 Startup Calibration - Slope Adjustment (Recommended for Initial Startup or Renewal of Measuring Electrode)

- 4.4.2.1 The slope adjustment expands or contracts the monitor span to match the slope of the electrode's response curve; i.e., the change of emf for a given change of sodium-ion activity. The span is factory adjusted for a Nernstian response of 59.16 millivolts for a ten-fold change of sodium-ion concentration at 25°C. A slight correction is usually required for a particular electrode system. This requires monitor readings from two known solutions having a one-decade difference in sodium-ion concentration either within the monitor range or slightly above it.
- 4.4.2.2 The background sodium-ion concentration of the dilution water used for the reference solutions has a negligible effect on the accuracy of the slope adjustment if both reference solutions contain at least 100 times more sodium than the dilution water.
- 4.4.2.3 Set the selector valve in the analyzer to MEASURE BLANK-SPAN and the selector in the calibration facility to MEASURE SPAN. Close the SPAN valve.
- 4.4.2.4 Prepare or obtain two reference solutions, the one with a sodium-ion content ten times the other and selected so that the two concentrations bracket the high-end on the monitor scale. See Appendix A.
- 4.4.2.5 Pour one liter (10 minute supply) of the lower concentration sodium chloride solution, reference solution A, into the smaller lower reservoir marked SPAN. Open the SPAN valve and, if necessary, re-adjust the SAMPLE REGULATOR to obtain a flow of 100 cc/min on the left flowmeter. (Increase the flow briefly to flush out and fill the system.) Reference solution A now flows from the SPAN reservoir through the regulator, flowmeter, absorber, and the electrode mounting block to drain. Gas must be flowing at 60 cc/minute.

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- 4.4.2.6 Switch the monitor to MEASURE, wait for measurement of reference solution A to stabilize; then, adjust STANDARDIZE on the monitor (and, if necessary, the ZERO dial) to bring the meter pointer to 50 on the linear scale. Make this critical adjustment very carefully. The mechanical meter zero must also be accurate. If necessary, turn off monitor power for 30 seconds and make certain the pointer is at 50 on the linear scale.
- 4.4.2.7 Close the SPAN valve and remove the SPAN reservoir. Save any remaining solution and rinse out the reservoir thoroughly with low-sodium-ion water. Then pour in one liter of reference solution B and replace the reservoir in the facility.
- 4.4.2.8 Open the SPAN valve to permit solution B to flow through the system; then wait for the monitor reading to stabilize and note the reading. If necessary, adjust SLOPE to bring the meter pointer to 83.3 on the linear (lower) scale, exactly one decade of sodium-ion concentration above the reading for reference solution A. (If necessary, make coarse adjustment with SPAN dial.)
- 4.4.2.9 Close the SPAN valve and again remove and rinse the SPAN reservoir with low-sodium-ion water. Now, pour in a liter of reference solution A and again make a reading when the measurement has stabilized. The meter pointer should return to the center of the meter scale after the measurement stabilizes. (If it does not, the adjustment in 4.2.6 was inaccurate. Check the meter mechanical zero and repeat 4.2.6 to 4.2.9.)
- 4.4.2.10 Adjust STANDARDIZE (and, if necessary, the ZERO adjust) on the monitor to bring the meter pointer to the value of sodium-ion concentration of reference solution A on the log scale. Startup calibration is now complete. Drain, then rinse the reservoir with low-sodium-ion water.
- 4.4.2.11 The monitor has been calibrated for the slope of the response curve for the electrode system used.
- 4.4.2.12 Return the analyzer to the sample stream by setting its selector to MEASURE SAMPLE. Adjust sample regulator for a sample flow of 125 cc/minute.

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4.4.2.13 When sample has been measured for about 10 minutes; read concentration.

4.4.2.14 Adjust STANDARDIZE on monitor to bring the meter pointer to the value of the sodium ion concentration of the sample as determined by flame emission photometry.

4.5 Calibration of Conductivity Cell

4.5.1 Apparatus

4.5.1.1 Conductivity cells (reference and test cells).

4.5.1.2 Measuring circuit-instrument may be a manually operated Wheatstone Bridge, a deflection meter, or a self-balancing recorder or indicator.

4.5.1.3 Thermometer

4.5.2 Reagents

NOTE: In making up KCl solution, use DI water stabilized to the laboratory atmosphere by aspirating air through the water until conductivity readings remain constant.

4.5.2.1 Potassium Chloride Reference Solution - Dissolve 0.7440 g KCl (weighed in air) in water and dilute to 1 liter at $20 \pm 2^\circ\text{C}$.

4.5.2.2 Shortly before using, dilute 100 ml of reference solution (4.5.2.1) to 1 liter with water at $20 \pm 2^\circ\text{C}$. Store in a glass stoppered bottle of chemical resistant glass.

4.5.3 Determination of Cell Constant

4.5.3.1 Rinse reference cell and cell under test several times with DI water.

4.5.3.2 Control the solution temperature to $25 \pm 0.1^\circ\text{C}$.

4.5.3.3 Immerse both cells to the same depth in water. The level of the solution should cover cross-channels of both cells.

4.5.3.4 Connect each cell to a measuring instrument.

4.5.3.5 Measure the resistance (or conductance) across the bridge terminal for each cell.

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4.5.3.6 Repeat the measurement on additional portions of DI water until the value obtained does not differ by more than 1 percent.

4.5.3.7 The electrical conductivity value assigned to the KCl reference solution is 146.93 $\mu\text{mhos/cm}$ (ASTM D1125-Electrical Conductivity of Water). This value excludes the conductivity of the water.

4.5.3.8 Repeat 4.5.3.1-4.5.3.6, substituting the KCl reference solution.

4.5.3.9 Calculate cell constant per instructions 4.5.3.10-4.5.3.13.

4.5.3.10 For instruments reading measured conductance, in mhos , calculate the cell constant, J , as follows and record:

$$J = (K_1 + K_2) / 1,000,000 \times K_x$$

where:

K_1 = conductivity, $\mu\text{mhos/cm}$, of the KCl in the reference solution at the temperature of measurement (Table 2).

K_2 = conductivity, $\mu\text{mhos/cm}$, at the same temperature of measurement, of the water used to prepare the reference solution, and

K_x = measured conductance, mhos .

4.5.3.11 For instruments reading measured resistance in ohms, calculate the cell constant, J , as follows and record:

$$J = (R / 1,000,000) \times (K_1 + K_2)$$

where:

R = measured resistance of the cell.

K_1 = conductivity, $\mu\text{mhos/cm}$, of the KCl in the reference solution at the temperature of measurement (Table 2), and

K_2 = conductivity, $\mu\text{mhos/cm}$, at the same temperature of measurement of the water used to prepare the reference solution.

GENERAL ELECTRIC**SODIUM TECHNOLOGY PROCESS INSTRUCTION**

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TABLE 2

Approx. Normality of Reference Solution	Method of Preparation	Temp. °C	Electrical Conductivity μmho/cm
0.01	0.7440g KCl weighed	0	773.6
	in air/1000ml of	18	1220.5
	solution at 20°C	25	1408.8

- 4.5.3.12 The cell constant has now been calculated and cannot be altered because the value is determined by the cell dimensions. This constant is related to resistance and conductivity by the following formula:

$$\text{Specific Conductance} = \text{cell constant} \times \frac{1}{\text{Resistance}}$$

- 4.5.3.13 A gradual lag in the recorder action is almost always identified with dirty conductivity cells.

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4.6 Checkout of pH Electrodes

4.6.1 Reference Buffer Solutions

- 4.6.1.1 Borax Reference Buffer Solution - Dissolve 3.80 g of sodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) in water and dilute to 1 liter. The pH at 25°C is 9.18.
- 4.6.1.2 Calcium Hydroxide Reference Buffer Solution (pH=12.45 at 25°C) - Prepare pure calcium hydroxide [$\text{Ca}(\text{OH})_2$] from well-washed calcium carbonate (CaCO_3) of low-alkali grade by slowly heating the carbonate in a platinum dish at 1000°C and igniting for at least 45 minutes at that temperature. Cool. Slowly add the calcined product to water while stirring. Heat the resulting suspension to boiling. Cool. Filter through a funnel having a fritted-glass dish of medium porosity. Collect the solid from the filter. Dry in an oven at 110°C. Crush to a uniform granular state. Vigorously shake a considerable excess of the finely granular product with water at 25°C in a stoppered polyethylene bottle. Allow excess to settle. Filter solution with suction through fritted-glass funnel of medium porosity. The filtrate is the reference buffer. Contamination of the solution with atmospheric CO_2 renders it turbid and should be replaced.
- 4.6.1.3 Other Reference Solutions - Commercially prepared buffers which have been certified by the U.S. National Bureau of Standards may be used as control standards instead of laboratory prepared reference solutions.
- 4.6.2 Select two reference solutions whose pH will bracket the expected range of the sample stream. For example, pH7 and pH10 bracket the allowed pH range of the water (8.5-9.0) at the evaporator inlet.
- 4.6.3 Pour first reference buffer into a beaker large enough to contain the electrodes.
- 4.6.4 Measure the temperature of the solution. It should be approximately 25°C. Warm or cool as necessary.

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- 4.6.5 Rinse electrodes well with DI water. Lightly pat dry with a Kimwipe.
- 4.6.6 Immerse bench top electrodes into solution and read pH. Adjust reading with standardize knob if necessary. Repeat until two successive readings agree without changing the setting.
- 4.6.7 Wash electrodes 3 times with DI water.
- 4.6.8 Immerse electrodes in second reference buffer. Read pH from meter. Do not change knob. Repeat readings until two successive readings differ by not more than 0.02pH.
- 4.6.9 The electrodes are operating satisfactorily if the pH reading agrees with its assigned value within 0.05 pH unit.
- 4.6.10 Allow on-line electrodes to sit in DI water overnight before calibration.
- 4.6.11 Calibrate the on-line electrodes against the bench top pH meter per instruction 4.6.12-4.6.18.
- 4.6.12 Pour first reference buffer (4.6.3) into a beaker.
- 4.6.13 Measure temperature of solution. It should be approximately 25°C.
- 4.6.14 Remove electrode from DI water and pat dry.
- 4.6.15 Immerse electrodes into buffer solution.
- 4.6.16 Read pH from Speedmax recorder.
- 4.6.17 If pH does not agree with the assigned value of this buffer, adjust the coarse and fine span adjusters on the component side of the main amplifier until the correct reading is obtained.
- 4.6.18 Read the second reference buffer as a check.
- 4.6.19 The on-line electrodes have now been calibrated to the bench top pH meter.

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APPENDIX A

Preparation of Sodium Standard Solution

Dissolve 2.542 grams of dried sodium chloride (NaCl) in deionized water and dilute quantitatively to a volume of 1 liter. The final concentration is 1000 $\mu\text{g/ml}$ Na. DO NOT USE GLASS CONTAINERS!

Sodium Analyzer

1. To a 1 liter polyethylene or Nalgene volumetric flask add 0.5 ml of the sodium standard. Dilute to 1 liter. The final concentration is 500 $\mu\text{g/l}$ Na or 500 ppb Na. This is reference solution B.
2. Take 100 ml of the 500 $\mu\text{g/l}$ Na solution and add to a 1 liter polyethylene or nalgene volumetric flask. Dilute to 1 liter. The final concentration is 50 $\mu\text{g/l}$ Na or 50 ppb Na. This is reference solution A.

AA/FE Spectrophotometer

1. Prepare 50 ppb Na solution as in Step 2 for Sodium Analyzer above.
2. Measure 200 ml of the 50 ppb into a 1 liter Nalgene volumetric flask. Dilute to 1 liter. Concentration is 10 ppb. This is the "HIGH" standard.
3. Measure 100 ml of the 50 ppb Na solution into a 1 liter Nalgene volumetric flask. Dilute to 1 liter. Concentration is 5 ppb Na. This is the "LOW" standard for the spectrophotometer.

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Approved by: 1. <i>[Signature]</i> 2. <i>[Signature]</i> 3. <i>[Signature]</i> 4. <i>[Signature]</i> 5. <i>Ed Weilmus</i>	Date: 4-18-78 4-22-78 4-18-78 4-18-78 4-18-78	Procedure No.: 04.50.159 Revision No.: 0

for 40 ppb for low

APPENDIX II

Parameter	Specification at Pre-heat and Below 5% Power	Specification Above 5% Power	HIGH READINGS		LOW READINGS	
			Possible Reason	Action	Possible Reason	Action
Dissolved Oxygen	10 ppb max.	7 ppb max.	Incorrect flowrate	Adjust flowrate to 8 ml/min. on the oxygen flow-meter.	N/A	N/A
			Oxygen probe dis-assembled for cleaning introduces air into the on-line system.	Purge system. Takes 1-4 hours for stabilization.		
			Need to inject more hydrazine.	Inject hydrazine via chemical injection system.		
			Air accidentally introduced into the on-line system.	Increase flowrate for about 1 hour to purge system. Reduce to correct flowrate and allow stabilization.		
Hydrazine	100 ppb min.	5 ppb (no max.)	Over-injected hydrazine-ammonia solution.	Ask Operations Engineer to drain a few gallons of water and add make-up water. If the hydrazine concentration is less than 500 ppb, but greater than 100 ppb at operation <u>below</u> 5% power, wait 1 hr., check hydrazine concentration to (continued)	Hydrazine has been consumed.	Inject N ₂ H ₄ solution for 5 min. Check concentration 30 min. after injection.

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APPENDIX II (continued)

Parameter	Specification at Pre-heat and Below 5% Power	Specification Above 5% Power	HIGH READINGS		LOW READINGS	
			Possible Reason	Action	Possible Reason	Action
Hydrazine (continued)				see if reading has dropped to the 100 ppb range before requesting a drain.		
pH	8.5-9.6	8.5-9.0	Injected too much hydrazine-ammonia solution.	Vent hotwell. Put high pressure demineralizer on line.	Need hydrazine- ammonia solution injection.	Add N ₂ H ₄ /NH ₃ solution in 0.5 liter increments. Check the pH about 20 min. after addition.
Cation Conductivity 77°F	6.0µmho/cm	2.0µmho/cm	Injected too much hydrazine-ammonia solution. Bad DI water High Na	Put high pressure demineralizers on line. Drain few gallons of water and re- place with make-up water. Check total conduct- ivity reading on the DI water at the Raw Water Treatment Area. Change resin beds.	N/A	N/A

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