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DEVELOPMENT OF ON-LINE MONITORING DEVICE TO DETECT
THE PRESENCE/ABSENCE OF SODIUM VAPOR

by

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Chemical Technology Division

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ABSTRACT

A process is being developed by the Sodium Waste Technology Program at ANL-W to remove metallic sodium from scrap and waste. The final step in the process is the removal of residual metallic sodium by evaporation at temperatures up to 482°C (900°F) and at pressures of about 10^{-2} torr (1.3 Pa). Efficient operation of this process requires that the operators have a method to indicate the completion of the evaporation. This "end point" would signify when the chamber and scrap and waste is free of metallic sodium. It was determined that a measure of the vacuum was not sufficiently sensitive, and a research effort was undertaken to select an on-line monitoring device. In this effort, three promising methods were reviewed. The use of a quadrupole mass spectrometer was recommended and an on-line device was designed for use in a Sodium Process Demonstration (SPD) Plant.

I. INTRODUCTION

A process that will remove sodium from scrap and waste and convert the resulting sodium to a compound such that both (1) the scrap and waste, and (2) the sodium compound are acceptable for disposal is being developed by the Sodium Waste Technology Program at ANL-W. In support of this development effort, a Sodium Process Demonstration (SPD) facility has been built at the Idaho site of Argonne National Laboratory (ANL). The facility is designed to melt and drain the sodium from scrap and waste and transfer this sodium to a storage tank. After draining, the residual sodium is removed from the scrap and waste by evaporation. Finally, the sodium is converted to a more easily handled waste form by controlled oxidation (calcining). The technique for removal of sodium from components by the evaporation mechanism is known;^{1,2} however, there is no effective technique available to determine the "end point" of sodium removal from the components and vessel. A research effort was initiated to define a method and then to develop an economical device, if possible, by which the "end point" of the evaporation could be determined. Without such an on-line monitor to indicate the presence or absence of sodium vapor, the process operators have no reliable indication that the cycle is complete and the components and vessel are free of metallic sodium.

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II. BACKGROUND

The melt-drain-evaporate (M/E) vessel in the SPD is a sealed, all-metal unit which operates up to 482°C (900°F). Since this is an engineering scale system, the detection-method sensors must be relatively simple, strong enough to function well with minimal maintenance, and capable of functioning remotely. The environment in which the detector (sensor) must operate is sodium vapor at pressures from $\sim 10^{-3}$ torr to 1500 torr (0.13 Pa to 200 KPa), temperatures up to 482°C (900°F), and a radiation field when radioactive materials are processed.

Sensing of sodium vapor is a capability of various analytical instruments. A review revealed several techniques that might be adapted for use in the SPD plant. Three of the most promising techniques--conductivity measurement,³ atomic absorption, and mass spectrometry--were evaluated as sensors in an environment similar to that in the SPD.

Commercial conductivity devices such as hot-cathode ion gauges appeared feasible since the filament current of these gauges varies with the vacuum system pressure and can easily be monitored. These gauges are also relatively inexpensive.

Atomic absorption is a technique which is sensitive to sodium vapor and could be adapted for use in this application. This method utilizes a hollow cathode lamp which emits light at the sodium wavelength. The beam travels across a gap and impinges on a photodetector. The light energy measured by the photodetector is dependent upon the amount of sodium vapor present in the gap.

The quadrupole mass spectrometer may also be used to monitor the presence of sodium vapor. In this unit, continuous monitoring of the 23 Atomic Mass Unit (AMU) by the mass spectrometer would indicate the presence of sodium vapor as well as the changes in the concentration level.

III. TEST APPARATUS AND PERFORMANCE OF MONITORING DEVICES

An apparatus was constructed in which sodium vapors could be generated and condensed. It also accommodated the several sensors to be tested. This initial apparatus was later modified to allow for the installation of more sensors.

A. Tests Using the Initial Test Apparatus

The configuration of the initial Sodium Evaporation System (SES) is shown in Fig. 1. It was constructed mainly of Varian Conflat[®] high-vacuum fittings, which acted as the conduit for the sodium vapor. The main features of the apparatus were a boiler, condenser, vacuum pump, and ports through which the various monitoring devices were introduced. The piping was wrapped with tape heaters; thermocouples were attached and the system was insulated by approximately two inches of blanket insulation. The boiler had a separate heater which was controlled by a proportional temperature controller.

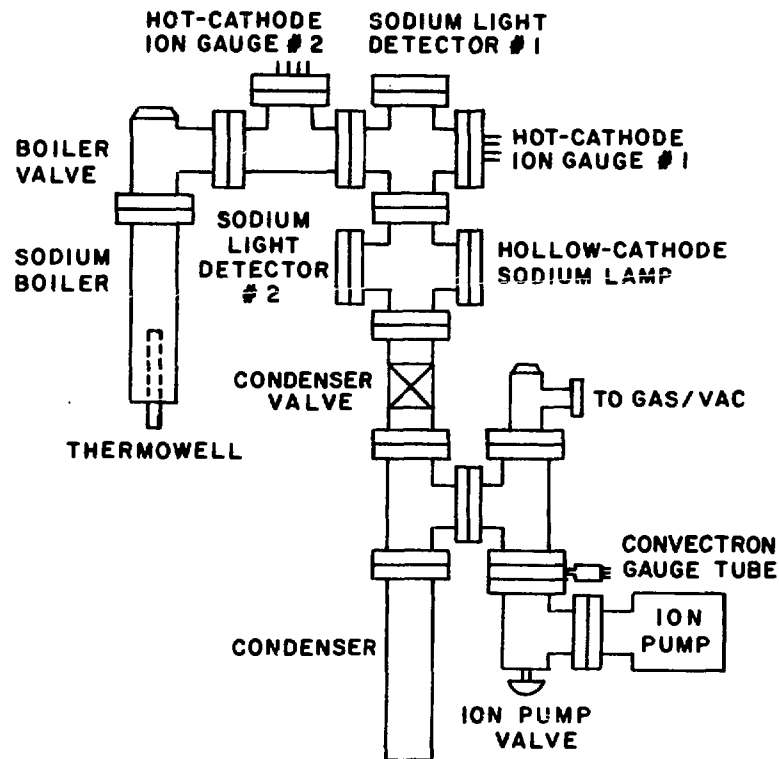


Fig. 1. Initial Configuration of the Sodium Evaporation System (SES)

Two types of hot-cathode ion gauges were installed in two of the ports-- a Varian Milli Torr[®] ion gauge and a Varian UHV[®] gauge. Two sapphire windows were mounted opposite each other directly above the condenser; a third window was mounted in line of sight with the condenser to allow a view of the vacuum space and the ion gauge.

Operation of the SES closely paralleled that of the SPD M/E vessel, which is designed to operate up to $\sim 482^{\circ}\text{C}$ ($\sim 900^{\circ}\text{F}$). The procedure for startup of the SES included heating the system piping to 450°C under a vacuum, with the condenser temperature range from 100°C at the top to 50°C at the bottom and the boiler at room temperature. The boiler temperature is then adjusted to the desired operating temperature ($300\text{--}480^{\circ}\text{C}$).

When all sections of the system reach the appropriate operating conditions, the condenser and boiler valves are opened or closed to obtain the desired test conditions. Sodium vapor is allowed to enter the system by opening the boiler isolation valve or by increasing the boiler temperature with the valve open. Adjusting the boiler temperature influences the concentration of sodium vapor present in the system. Vapor is removed from the system by opening the condenser valve, allowing sodium vapor to be drawn to the condenser, and closing the boiler valve.

1. Conductivity Measurements

a. Hot-Cathode Ion Gauges

The Varian Milli Torr[®] ion gauge was installed in its "as-received" condition. The normal operating range of this gauge is 1 to 10^{-6} torr (1.3×10^2 to 1.3×10^{-4} Pa), and the response curve⁴ of the Milli Torr gauge indicates that it may respond at pressures as high as 4 torr (5.3×10^2 Pa), though the curve appears to lose its linearity at pressures above 0.5 torr (67 Pa).

Tests indicated that the voltage applied to the grid of the ion gauge (180 V) was sufficient to cause sodium vapor in the immediate area to glow, as a plasma. The meter on the Milli Torr controller indicated a variation in current with a change in the amount of sodium vapor in the system, and the filament voltage of the Milli Torr gauge appeared to decrease to zero when the sodium vapor plasma was present.

The Varian UHV[®] gauge was designed to operate at lower pressures. A regulated power supply was connected to the UHV gauge so that the voltage applied to the filament of the gauge could be controlled. The filament current varied with the presence or absence of sodium vapor.

Unfortunately, these gauges were more delicate than expected and were very susceptible to shorting when sodium condensed on their surfaces. This shorting occurred at sections which were slightly cooler than the surrounding piping. In addition, the durability of the feedthroughs on these gauges was less than expected. Vendor recommendations limited operating temperatures to $<450^\circ\text{C}$, and the combination of the heat and sodium environment caused seals in the gauges and feedthroughs to fail in a relatively short time. The wires and sapphire windows had been brazed into the Conflat flanges. Sodium reacted at these brazed areas, and eventually leaks developed. Because of the problems in this environment, these gauges could not be used as sodium vapor detectors.

b. Two-Pin Electrical Feedthrough

A Varian ten-pin electrical feedthrough was modified so that only two pins extended into the vapor space. These two pins were connected to a power supply and a current-measuring circuit, and a constant voltage was applied. The current variations were monitored as a function of sodium concentration. The sodium concentration in the system was varied by evacuating the system or allowing it to remain static. Evacuation created a concentration gradient in the system which caused sodium vapor to transfer. As shown in Figs. 2 and 3, when a vacuum was applied to the system by opening the condenser valve (causing the sodium concentration in the piping to increase), the current measured across the two pins increased. Figure 3 is a continuation of the test in Fig. 2 after the system was isolated for about 16 h by closing boiler and/or condenser valves to retain vapor in the piping.

This modification of the feedthrough for use as a conductivity meter was found to develop the same types of failure problems at the feedthrough seals as the ion gauges. Hence, this device also could not be used as a sodium vapor detector.

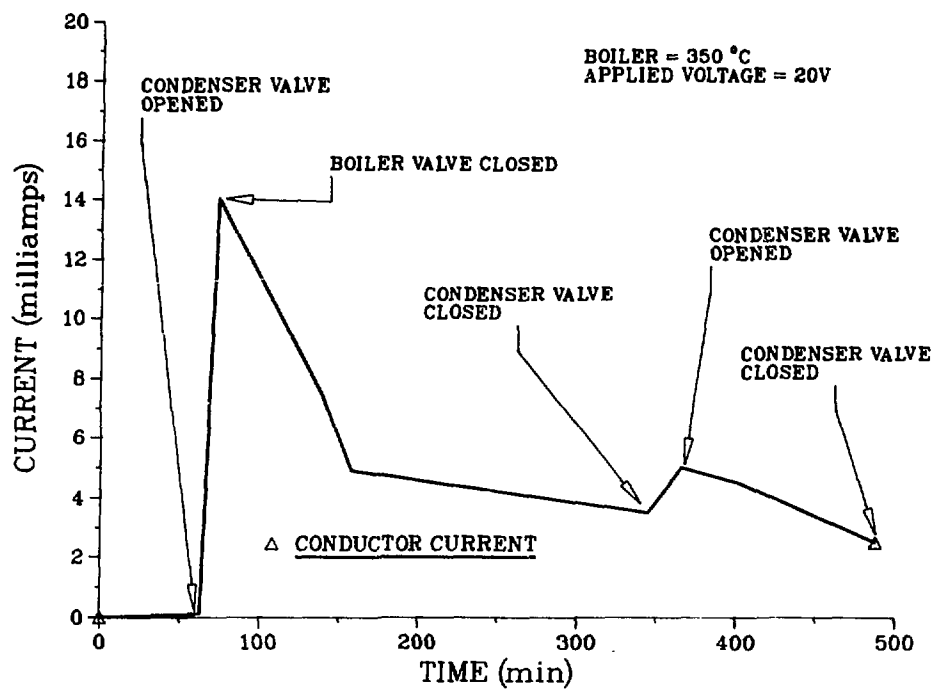


Fig. 2. Response of 2-Pin Conductor to Sodium Vapor

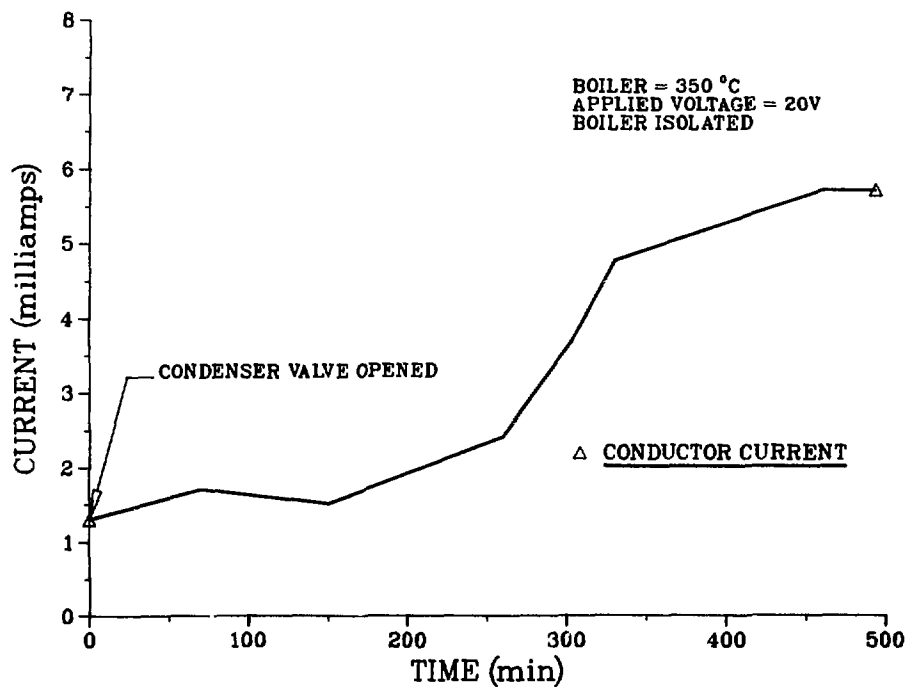


Fig. 3. Response of 2-Pin Conductor to Sodium Vapor after Isolation for ~16 h

2. Atomic Absorption

The sapphire windows in the SES were used to allow an atomic absorption technique to be used. A hollow-cathode sodium lamp was positioned so that it shone through the sodium vapor. The transmitted light was focused through a filter and onto a silicon photodetector, the output of which was measured with an electrometer. The output was found to vary with the presence of sodium vapor. As the concentration of sodium in the system was changed by opening or closing the boiler valve or changing the boiler temperature, the photodetector responded. An increase in the concentration of sodium caused a decrease in the voltage measured by the photodetector, and a decrease in sodium concentration caused an increase in the measured voltage. This effect is shown in Fig. 4. This response was encouraging.

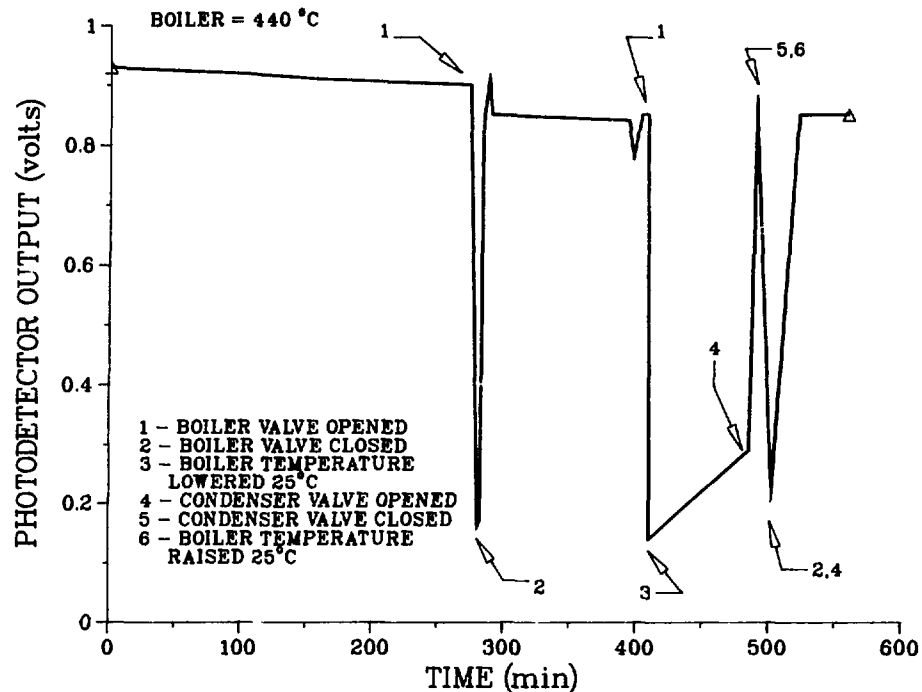


Fig. 4. Response of Photodetector to Sodium Vapor

Ultimately, the window seals proved to be a weak point because the joints were brazed, and they failed with time at the experimental conditions. Another difficulty was that the crucial alignment requirements of these components were very difficult to maintain in a remotely operated facility. The sensitivity of this technique was encouraging, and it could be used under less rigorous operating requirements.

B. Tests Using Modified Test Apparatus

Because of the problems encountered with devices in the initial configuration, the SES was modified to accommodate the installation of (1) a more durable electrical feedthrough and (2) a quadrupole mass spectrometer. This new configuration is shown in Fig. 5.

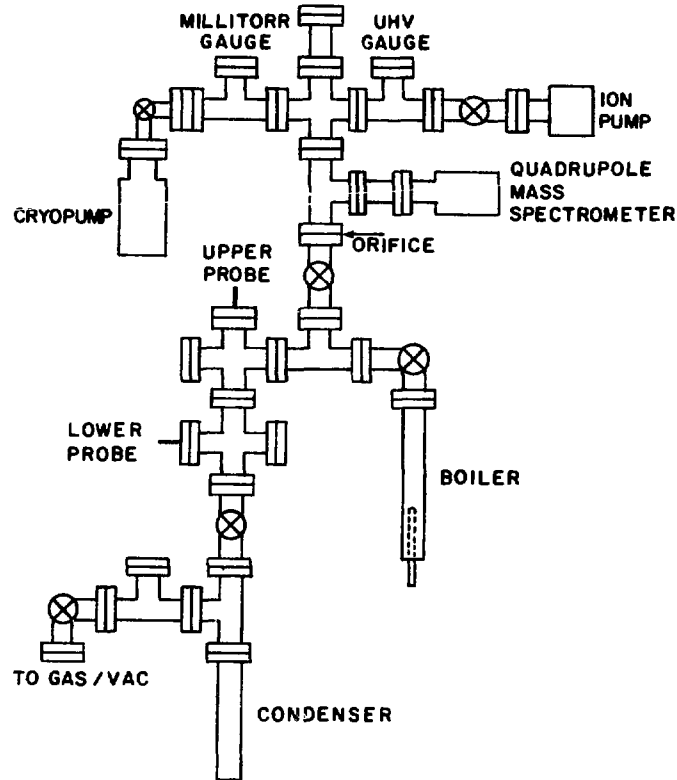


Fig. 5. Modified Sodium Evaporation System

1. Description of New Devices

a. Conductivity Probes

Since applying a constant voltage to a conductivity device and monitoring the current passed across a gap was a successful indicator of the presence of sodium vapor, the search for a more durable feedthrough continued. An electrical feedthrough* was found which was constructed of materials that were expected to withstand the extreme operating conditions. A conductivity probe design, utilizing this feedthrough, is shown in Fig. 6.

Two conductivity probes were operated in the SES with minor problems. A constant voltage of 10-100 V was applied to the probe, and the current passing from the electrode to the collector was measured. The voltage level applied was determined by the sodium concentration. As the sodium concentration increased, the current increased, and the voltage was decreased to maintain a desired current. At higher voltage levels the current exceeded the limit of the power supply, and the voltage applied was reduced to maintain the proper current. The current revealed the presence or absence of sodium vapor.

*Type HP, manufactured by ILC Technology, Sunnydale, CA.

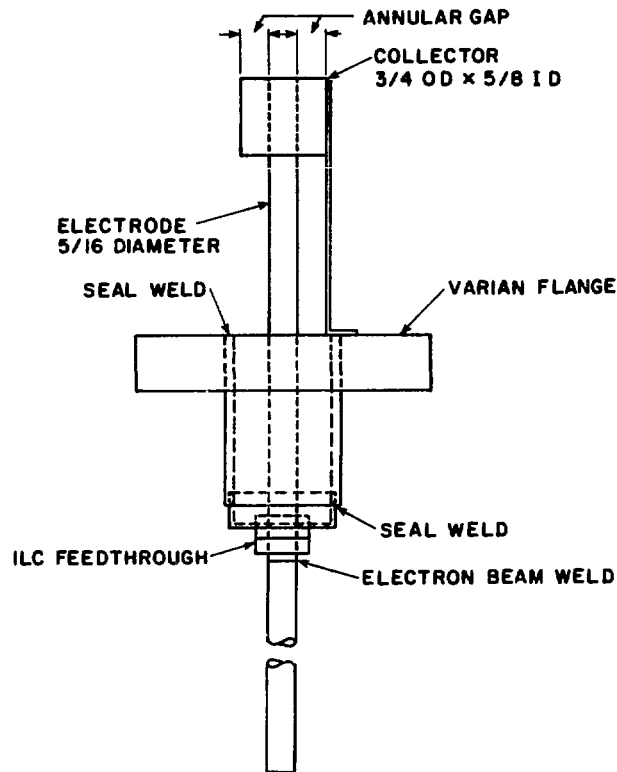


Fig. 6. Conductivity Probe
(Dimensions in
inches)

These probes responded to the admission of sodium vapor to the system but did not always respond immediately to removal of the vapor, *i.e.*, the "end point." Although feedthroughs for these probes last up to a maximum of 4800 h, after a period of operation they were susceptible to fracture on cool-down due to what appeared to be thermal shock; therefore, extreme care must be taken to prevent the feedthroughs from undergoing thermal shock. Subsequent examination of the probes after an extended period of operation indicated some undercutting and erosion of the central rod.

b. Quadrupole Mass Spectrometer

A quadrupole mass spectrometer that utilizes a Faraday cup sensing device was installed. A 0.009-cm orifice separated the main sodium piping from the high-vacuum section where the quadrupole was mounted. The purpose of this orifice was two-fold: first, with a sodium vapor pressure of 0.5 to 1 torr (67-133 Pa) in the boiler section, it prevented the pressure in the quadrupole section from rising above 10^{-4} torr (1.3×10^{-2} Pa), which would cause the quadrupole to become inoperative; second, it acted to limit the conductance of the sodium vapor stream so that, in effect, a minimal quantity of sodium vapor was exposed to the detector of the quadrupole. Experience has shown that some sodium may reach the quadrupole surfaces and condense. The quadrupole piping must be the hottest component in the area. During normal operation, the mass range from 0-50 AMU is scanned; by monitoring the peak height of the 23 AMU channel, the presence or absence of sodium vapor can be determined, as well as its relative concentration.

Response to the introduction of sodium vapor was rapid, as was the response to the decline of sodium vapor as the source was exhausted.

2. Method and Results

In a subsequent series of tests, both the new conductivity probes and quadrupole were operated in the following manner: after the desired temperature and pressure conditions had been attained, a voltage was applied to the conductivity probe and the current was monitored, while the quadrupole was operated and the 23 AMU peak was monitored. The response of these devices was observed and recorded (1) when sodium was in the system and (2) later, as the sodium vapor transfer approached the end point, when all the sodium vapor in the system had been transferred to the condenser.

Figure 7 shows the responses of the two conductivity probes and the quadrupole mass spectrometer during Exp. 1, with the boiler operating at 400°C. The monitors respond to the presence of sodium vapor shortly after the boiler heater is turned on. When the boiler heater is turned off after 6.5 h, the quadrupole mass spectrometer response is immediate, as is that of the lower conductivity probe. The upper probe, however, does not exhibit a sharp decrease. The reason for the lack of response by the upper probe to the cessation of sodium vaporization has not been determined.

Figure 8 shows the response of the probes and quadrupole mass spectrometer during another run, Exp. 2. Again, with the boiler at 400°C, response to the generation and cessation of sodium vapor is rapid. Unlike Exp. 1, the upper probe responds quickly to the boiler heater being turned

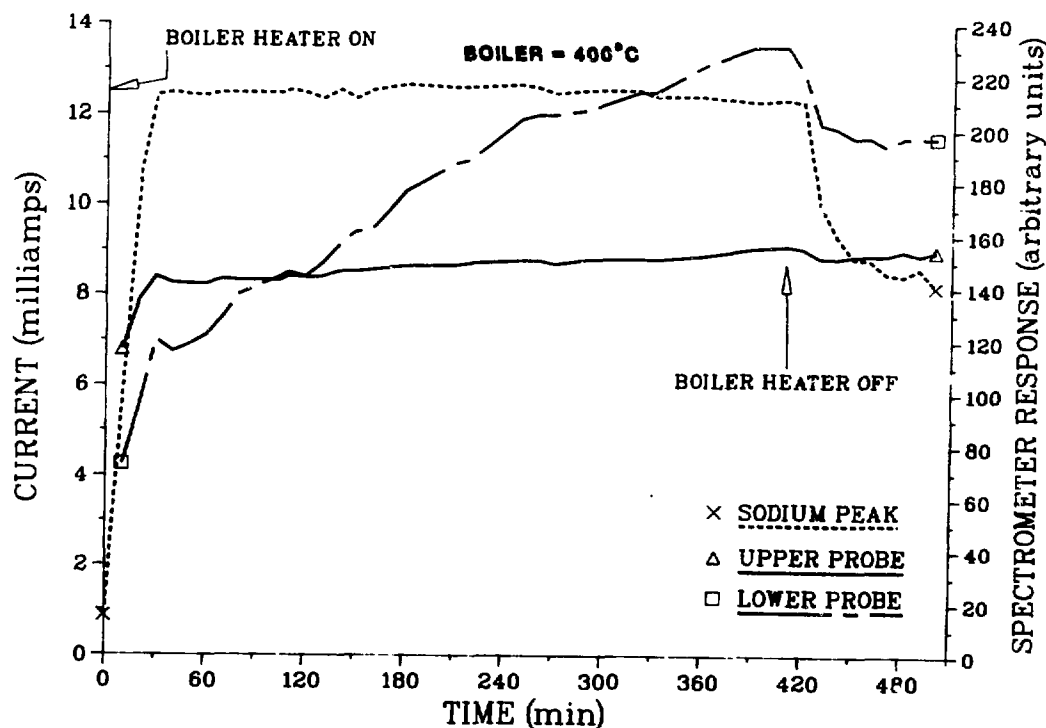


Fig. 7. Response of Conductivity Probes and Quadrupole Mass Spectrometer to Presence/Absence of Sodium Vapor (Experiment 1)

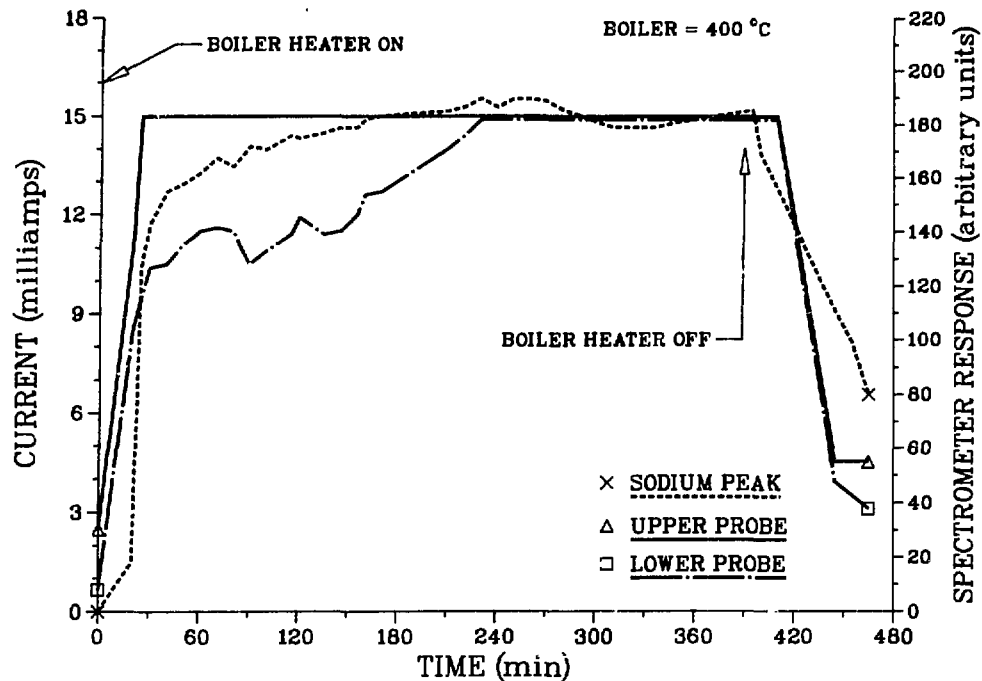


Fig. 8. Response of Conductivity Probes and Quadrupole Mass Spectrometer to Presence/Absence of Sodium Vapor (Experiment 2)

off in this experiment. The condensation of sodium vapor on some surfaces of this probe and the subsequent evaporation of this coating by treatment at a high temperature may explain its sluggish recovery in Exp. 1 and its more normal response in Exp. 2.

Experiment 3, as shown in Fig. 9, demonstrates the response of the monitors when the sodium supply in the boiler is exhausted. As desired, the response levels of each device decline, indicating that all the sodium has transferred and no sodium vapor is in the system. At the end of this experiment, the boiler was removed from the system and inspected visually; no free metallic sodium was found.

The final experiment in this series utilized a stainless steel block made up of five separable sections. Each section had one face containing an area machined to accurately simulate 0.0025- to 0.076-cm deep (1, 10, 20, and 30 mil) crevices present in components. The block was wetted in a bath of sodium at 500°C for ~48 h prior to its insertion into the boiler section of the SES. The block was heated to 450°C in the usual manner to vaporize the sodium, and the responses of the monitoring devices were recorded as the sodium was removed from the sample to the condenser.

Figure 10 shows the response of the quadrupole mass spectrometer and conductivity probes as the sodium vaporized from the block and crevices. The quadrupole responded rapidly to the vaporization of sodium. Although the probes responded to the introduction of sodium to the systems, they did not respond well to the completion of the transfer of sodium. However, they

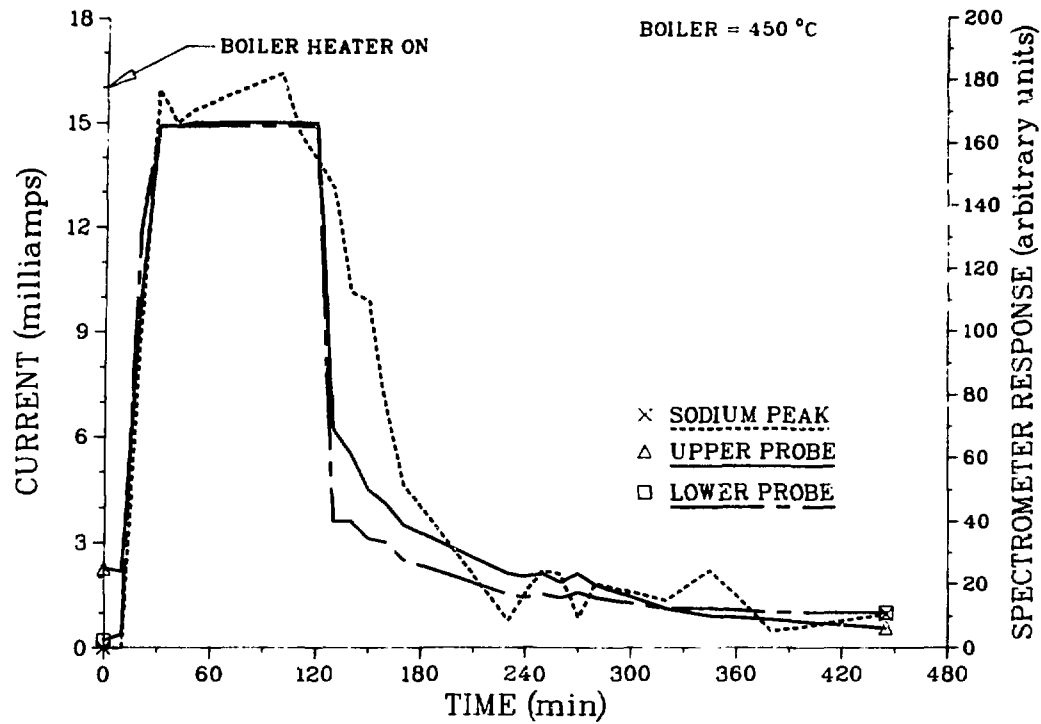


Fig. 9. Response of Conductivity Probes and Quadrupole Mass Spectrometer at Completion of Evaporative Transfer (Experiment 3)

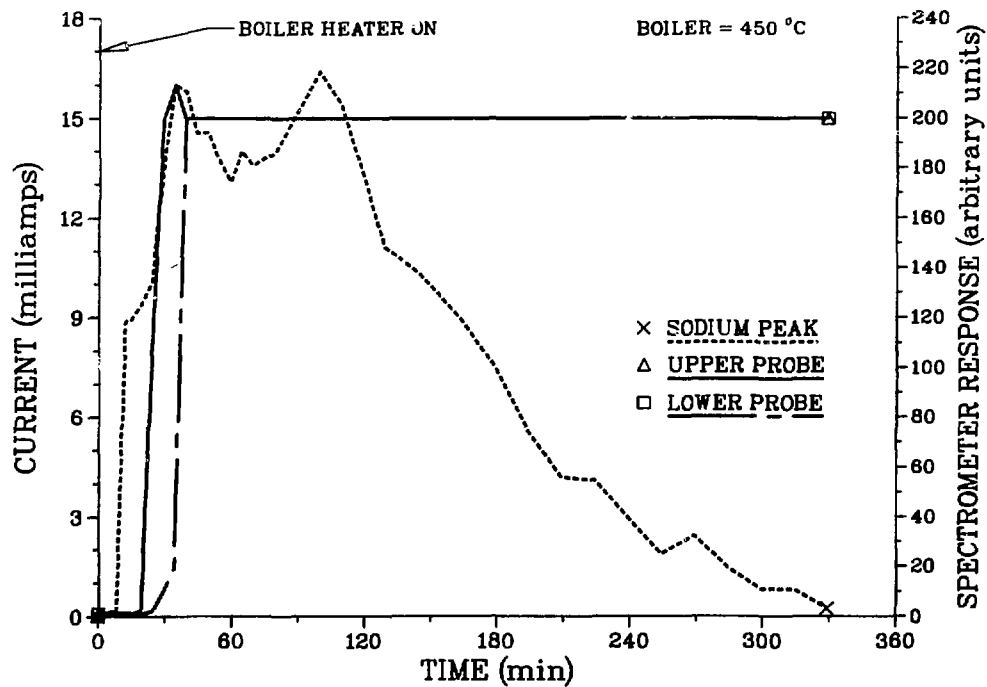


Fig. 10. Response of Conductivity Probes and Quadrupole Mass Spectrometer to Evaporative Removal of Sodium from a Crevice Test Specimen

ultimately returned to their baseline levels by the next measurement period, after 16 h had elapsed. This erratic response of the probes indicated that they are too slow for "end point" indication.

Visual observation of the block after removal from the boiler indicated no metallic sodium present. A residue was noted on the block, however, and weighing of the block indicated that at least 96.5% of the sodium had been vaporized. A portion of this residue was removed and analyzed. Results of the analysis by inductively coupled plasma spectroscopy are given in Table 1. This analysis showed the presence of iron, nickel, and chromium, indicating the residue to be a corrosion product. A small amount of sodium was also noted in this analysis, probably present as an oxide.

Table 1. Composition of Residue on Stainless Steel Crevice Test Specimen

Element	Filtrate, % in sample	Solid, % in sample
Al	0.35	0.01
B	<0.01	<0.001
Ba	<0.01	<0.01
Be	<0.01	<0.001
Ca	0.07	0.1
Cs	0.16	<0.003
Cr	1.4	10-100
Cu	0.26	0.03
Fe	70.24	1
Mg	0.03	0.005
Mn	0.84	0.5
Mo	<0.04	0.2
Na	16.33	0.1-10
Ni	10.01	0.3
Pb	<0.09	<0.001
Sn	0.05	<0.003
Sr	<0.01	<0.1
Ti	<0.01	0.01
V	<0.03	<0.01
Zn	0.05	<0.1
Zr	<0.01	<0.01

A most important discovery during these experiments was the extreme sensitivity of sodium vapor to slight temperature gradients ($\sim 10^\circ\text{C}$) within the SES. This system requires isothermal operation since the presence of a slight temperature gradient will cause the sodium to reflux in the areas of the temperature gradient rather than transfer from the boiler to the condenser.

IV. CONCLUSION

All the devices tested exhibited sensitivity to the presence of sodium vapor. Commercial conductivity devices such as hot-cathode ion gauges showed an expected sensitivity to the presence of sodium vapor, but the inability of the brazed joints in these devices to withstand the extremes of temperature and the sodium environment cause them to be eliminated for possible use.

The atomic absorption technique successfully monitored the presence of sodium vapor. Increases or decreases in the measured response of the sensing devices correlated closely with the presence or absence of sodium vapor in the system. However, the sapphire windows, used to pass light through the vapor space, utilize brazed joints which cannot withstand the combination of sodium vapor and high temperature for long periods of time. The clarity of the windows during operation was not a problem, though the windows did show signs of light etching after exposure to air.

The conductivity probes, fabricated using a specially brazed feedthrough, are able to withstand the temperature and sodium environment, but care must be taken to avoid thermal shock. The conductivity probes always responded rapidly to the introduction of sodium vapor; however, they were generally slow to respond to the removal of the sodium vapor. One possible cause may be the condensation of a layer of sodium (however thin) on the feedthrough; this layer would allow the passage of a current even though the vapor space was clear. Maintaining the temperature of the conductivity probe at the highest temperature in the system to prevent any condensation may allow more rapid recovery.

A quadrupole mass spectrometer proved to be the most effective method over a relatively long period of operation for "end point" determination. Response to the introduction or removal of sodium vapor was rapid. Unmistakable, large signals from the quadrupole were noted in minutes after sodium vapor was admitted to the piping. The signal from the quadrupole remained high while sodium was present. Finally, the signal reduced as the sodium vapor dissipated, indicating the sodium transfer was completed.

An ideal monitoring system would consist of two of these detection devices. The conductivity probe and the quadrupole mass spectrometer in tandem would comprise a sensitive, responsive sodium vapor detection device.

Based on this effort, a sodium vapor monitoring system was designed for the SPD, drawings for the installation of the sodium vapor detector were prepared, and a proposed operating procedure was written. The drawings and procedure are being used for fabrication, installation, and operation of a vapor detector on the SPD.

ACKNOWLEDGMENTS

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