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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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# CONTROL OF CONTAMINANTS IN LIQUID METAL SYSTEMS

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## SUMMARY

The contaminant controls and cleaning procedures used in the SNAP-8 simulation loop, consisting of two sodium-potassium (NaK) loops and one mercury (Hg) loop, minimized both startup and operational problems. Mercury wetting was induced in the boiler and condenser as determined by progressive improvement in the heat-transfer performance. The degree of wetting is an indication of the system cleanliness. The boiler was conditioned in approximately 250 hours of operation without wetting additives. After 384 hours of operation, analysis of a Hg sample by a distillation method indicated no contamination. Oxide plugging in the NaK loops was minimized using the loop-entry procedures developed in the investigation. No plugs occurred because of changes of repairs that required system entry, and only minor partial plugs occurred in over 1500 hours of operation of the NaK loops.

## INTRODUCTION

Past experience with liquid-metal systems has shown that more rigorous control over cleaning and purifying procedures are required than for conventional steam conversion systems. Impurities in these systems accelerate corrosion and impair the mechanical and thermal performance of equipment. They can, in some extreme cases, prevent system startup or result in such marginal performance that continued operation is of no value.

Contaminant control is a major problem in liquid-metal systems. Failure to control contaminants and the results can be found in the literature (e. g. , refs. 1 and 2). The solution is to adopt procedures based on past experiences plus a new awareness of the hydrocarbon effects on wetting in an Hg system. Consideration must be given to the consequences of the problems and the practical steps and procedures necessary for the prevention and control of contaminants.

In NaK or alkali metals in general, the major contaminant is oxygen, which can cause corrosion or plugging. Oxygen may be present in the alkali metal, in the inert cover gas, or in the system piping and equipment prior to startup. It may be introduced due to leakage during operation or during system maintenance and repairs. Experience has shown that welds are susceptible to corrosive attack unless the proper welding technique is used and each weld is inspected. The liquid metal itself becomes a contaminant for welds in making repairs or alterations.

System components and piping are susceptible to attack particularly at high temperatures. In test loops at Oak Ridge National Laboratories, wall thickness loss due to corrosion was measured in an AISI Type 505 (9M) boiler using eutectic NaK to vaporize Hg at 1100<sup>o</sup> to 1300<sup>o</sup> F (870<sup>o</sup> to 980<sup>o</sup> K) (ref. 3). These are the same materials and temperatures being used in the boiler of the system under investigation here at Lewis. Corrosion is moderate in oxidation resistant materials while plugging is a major problem since it can cause operational difficulties or even system shutdowns. Decreased solubility of oxides at lower temperatures causes them to precipitate whenever the temperature is less than the oxide saturation temperature. Precipitation occurs in the colder portions of the system and can build up to completely block the passage of flow.

Hydrogen can also cause plugging but to a much lesser degree. Water vapor in the air will react with alkali metals, forming hydrides as well as oxides; for example, with sodium the reaction produces sodium hydride which has a limited solubility in liquid sodium. The excess hydride appears as a solid and could block flow passage. Carbon, formed by a transfer process from the system materials at elevated temperatures can produce a hard and brittle scale. This scale adds resistance to the heat transfer of the components and reduces the effectiveness of the high heat-transfer coefficients of the alkali metals.

In Hg the major contaminants are oxygen and hydrocarbons. These contaminants degrade the system heat-transfer performance by prevention of "wetting." Nonwetting reduces heat transfer and, if a boiler is involved, can affect the boiler stability. The exact mechanism of this wetting phenomenon is unknown, but that a Hg system is highly sensitive to these contaminants has been proven many times. These contaminants can be present in the form of "dirt" left in the system piping and equipment prior to startup, or can be introduced during operation by leakage from the rotating machinery lubrication systems or backstreaming from vacuum pumping systems.

A study was made of a simulated SNAP-8 power system which was being used as a supporting effort in the SNAP-8 program. The system was highly complex, partially because of the need for flexibility in the test program. Recognition of the contaminant control problem led to modification of the original design before actual operation. Other solutions, although recognized as applicable, were limited by practical considerations. To meet system heat-transfer design parameters, particularly in the boiler and the con-

denser (see table I), the internal surface of the system had to be free of contaminants.

Several phases were involved in the cleaning procedures: (1) initial assembly, which included preventions and precautions before and during assembly; (2) preoperation, which included cleaning measures after assembly and prior to startup; (3) operation, including the maintenance of contaminant control during operation; and (4) repair and re-assembly of system and/or components, including precautions and techniques used during inadvertent entry or planned alterations. The cleaning methods and solvents adopted were determined by the size of and component materials in the system. Consideration is given to an accidental contamination of the loops, which allowed air into the NaK system and organics into the Hg system before any extensive operation was begun. Various practical procedures for cleaning a complex system are presented which were used to reduce system conditioning time (e. g. , oxygen removal from alkali metal systems or wetting in the case of a mercury system) and maintain contaminant control along with the techniques, precautions, and general recommendations for accomplishing these tasks.

This report is intended to serve as a guide, and many techniques pertinent to liquid metals are presented.

## APPARATUS

Contaminant problems were studied in a 35 kilowatt simulated SNAP-8 Rankine cycle power system which used mercury (Hg) as the working fluid, and a eutectic mixture of sodium and potassium (NaK) as the heat-transfer media. The complexity of the system due to the auxiliary and test support equipment can be seen in figure 1, a photograph of the partially completed boiler outlet section. A schematic of the system is shown in figure 2. The system basically consisted of two NaK loops and one Hg loop.

The primary loop used an electromagnetic pump to flow NaK through an electrical heater transmitting energy to a tube-in-shell boiler. Liquid mercury was pumped by a centrifugal pump through a filter and into the boiler where it was vaporized. The mercury vapor flowed in two parallel paths containing valves and venturies into a common vapor cooler before entering the condenser. The third or heat-rejection loop consisted of an electromagnetic pump which flows NaK through the condenser and into two air-cooled heat exchangers. Auxiliary equipment constitutes the remainder of the system and includes some of the major contaminant controls.

A common oxide control loop (OCI) for both NaK loops was incorporated to control the precipitation of any oxides that might be formed, rather than form a plug in the operating loop. A tap from each NaK loop was run to the oxide control loop (as seen in fig. 2) and the NaK was allowed to flow through an economizer and a cold trap. The economizer was a counter flow tube in tube heat exchanger used to cool the NaK entering

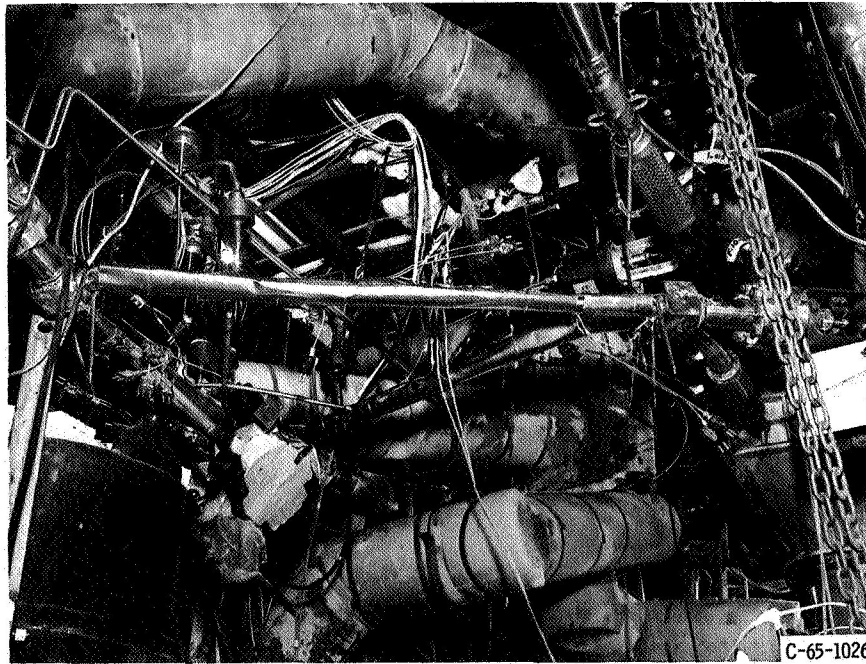


Figure 1. - Boiler outlet section.

the OCI loop and heat the NaK returning to the main system. The cold trap consisted of a stainless-steel cylinder, which was enclosed so that air could be blown over it. It was designed so that, at normal flow through the OCI loop (1 gal/min or 0.004 m<sup>3</sup>/min) there was an 8-minute stay time for the NaK. This time was considered long enough to precipitate any oxides present. A parallel plugging indicator line was used to determine the NaK oxide content of the system. This line was part of the OCI loop and consisted of a finned-tube cooler enclosed so that air could be blown over it and a calibrated serrated seat plugging valve. An electromagnetic flowmeter was used to measure total flow through the OCI loop.

Expansion tanks were used in the system to maintain suction heads on the NaK pumps, to allow for thermal expansion of the NaK, and to serve as inlets for the inert cover-gas pressurization system. Auxiliary vacuum and inert gas (argon) pressurization systems were used in conjunction with the main loops. The inert gas contained less than 5 ppm of contaminants as measured from periodic sampling.

Both NaK and Hg fill systems incorporated two operations, charging the system originally and filling the individual loops. The NaK fill system consisted of a dump tank, 20-micron filters, and several valves. The Hg fill system consisted of an expulsion tank, a dump tank, and several valves. During operation, both fill systems are isolated.

The system piping consisted primarily of AISI type 300 series stainless steels, but the boiler and condenser contained AISI type 505 (9M), AISI type 1010 mild steel and AISI type 410 stainless steel. Valves in the system had either stainless or cobalt-chromium based alloy seats with various packing and gasket materials. These materials

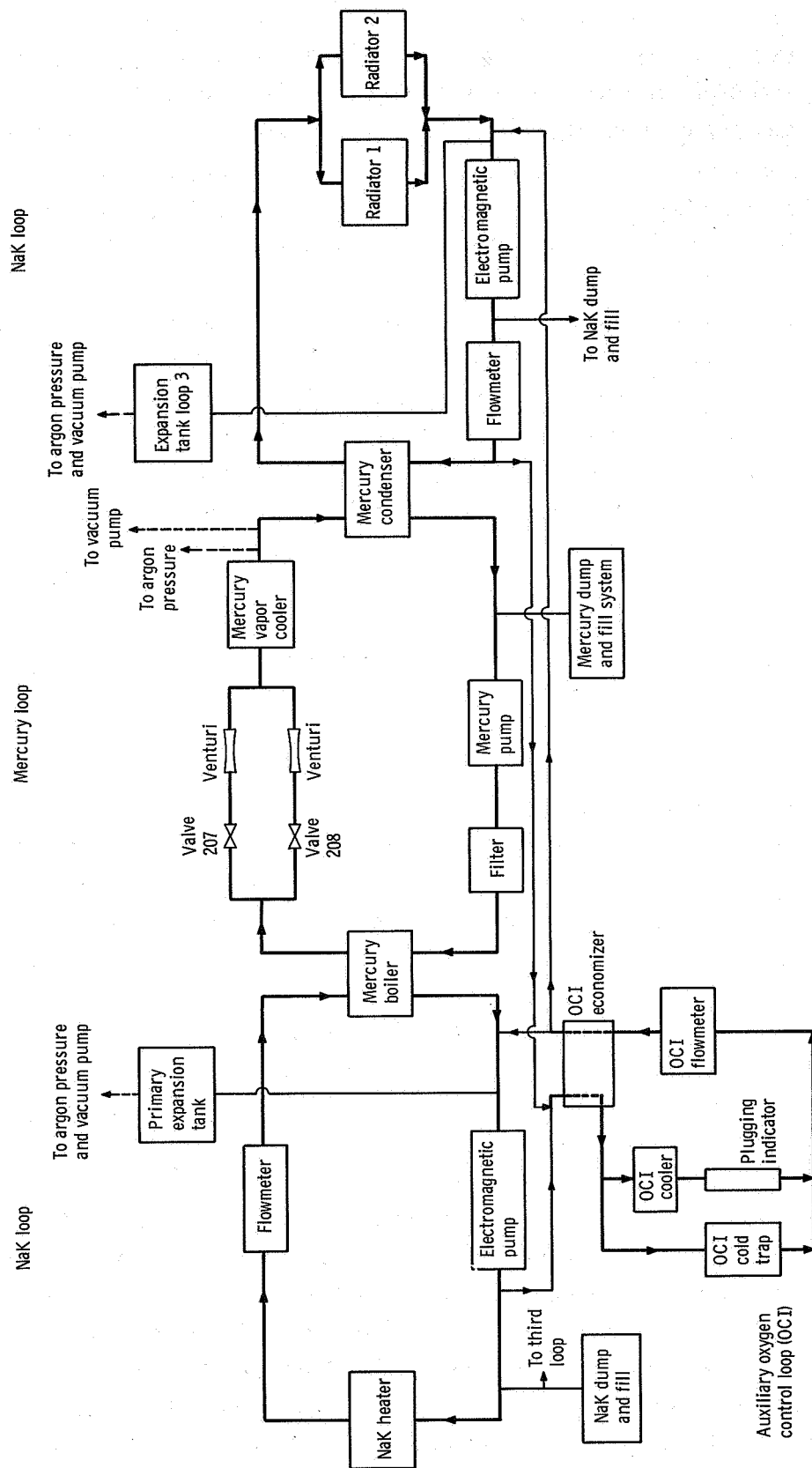


Figure 2. - Schematic of three-loop (NaK-mercury-NaK) system.

and the physical problem of cleaning associated test instrumentation having thin diaphragms, placed restrictions on the cleaning procedures that could be used.

During operation, Hg inventory was as high as 400 pounds (180 kg) with flows as high as 11 000 pounds per hour (5000 kg/hr). The as-received Hg was triple distilled and contained less than 0.025 ppm of nonvolatile material by weight. The combined NaK loops used approximately 400 pounds (180 kg) or 8 cubic feet (0.226 m<sup>3</sup>) with up to 35 000 pounds per hour (16 000 kg/hr) of flow in each loop. The NaK used was reactor grade.

The cleaning solvents used were based on the component materials in the system and their corrosion and oxidation problems. Flushing with a heated liquid presented too many physical problems because of the piping complexity, instrumentation, and, in general, the large size of the system. Acid cleaning could not be done because of the thin metal diaphragms in the pressure transducers. A compromise cleaning method was developed that took into account those problems and the various structural materials used in the system.

## CLEANING PROCEDURES

### Initial Assembly

Before initial assembly all components and system piping were vacuum checked and X-rayed to avoid installation of a defective unit. The part was then degreased with NA 500 (trichloroethane) and dried with an oil-free argon purge. This procedure is similar to Method IV of reference 4. Upon assembly completion, the system was X-rayed and vacuum leak checked using a helium leak detector and spraying each weld and fitting with helium.

Prior to each Hg loop cleaning, the rotating pump was removed, taken apart and cleaned individually to protect against damage to the pump rotating components (bearings, shafts, etc.), which could possibly be caused by the cleaning solvent. The polytetrafluoroethylene seats in some of the vacuum valves were removed during the cleaning operation to prevent any possible damage to the sealing surfaces.

The assembled system was flushed with vapor because it is more likely to reach all parts of the system than a liquid flush. Many areas, particularly instrumentation, had small size tubing or were not easily accessible to a liquid flush. As seen in figure 3, an earlier photograph of the boiler outlet section, the pressure transducers used in the system had 6- to 12-inch (15.3 to 30.5 cm) vertical standoff legs of diameters ranging from 1/4-inch (0.635 cm) tube to 1/2-inch (1.27 cm) pipe sizes. The standoffs were required because the diaphragms in the transducers could not take the elevated tempera-

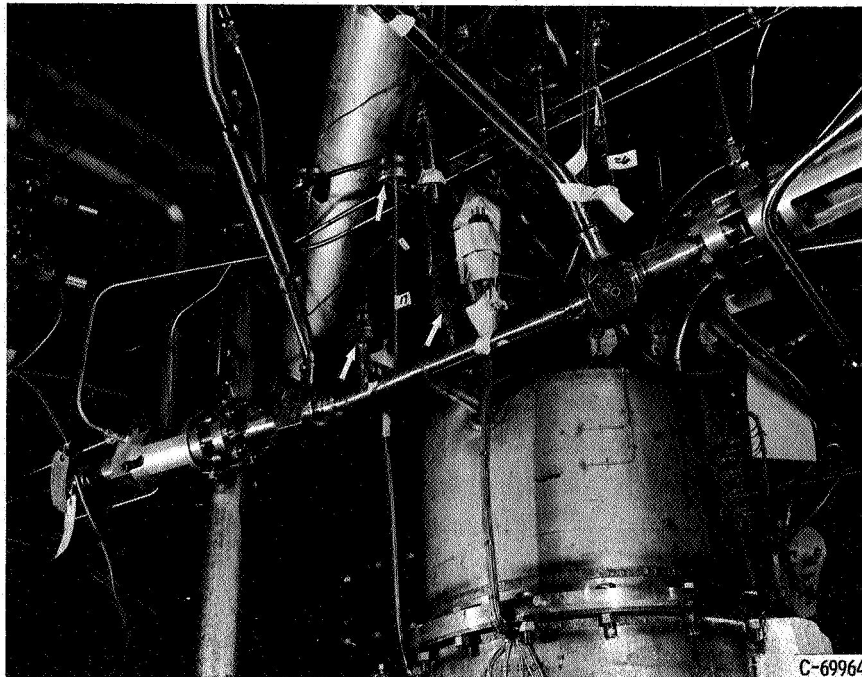


Figure 3. - Boiler outlet section showing transducer standoff legs.

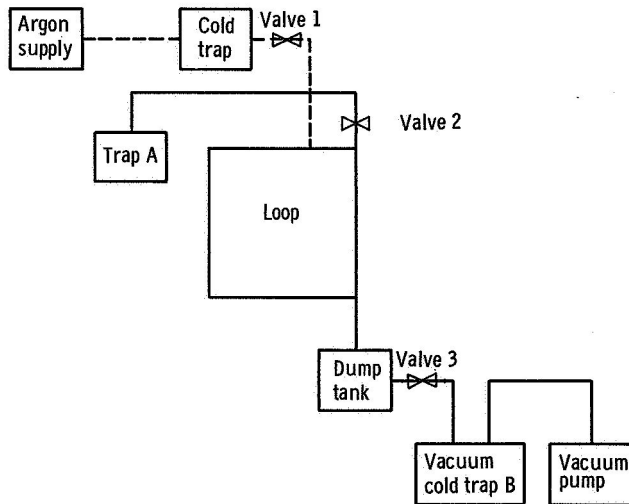


Figure 4. - Schematic diagram for loop flushing.

ture of the system fluids. These standoffs were nonflow legs and hence possible plugging sites in the NaK system. There were approximately 20 transducers in each loop. A rough vacuum ( $<1$  psia or  $7000 \text{ N/m}^2$ ) was drawn in the loop (fig. 4), and NA 500 was heated in trap A allowing NA 500 vapor to enter the loop and wash the walls. NA 500 vapor pressure is 100 millimeters of Hg ( $13\,300 \text{ N/m}^2$ ) at  $68^\circ \text{ F}$  ( $293^\circ \text{ K}$ ) (ref. 5). The NA 500 vapor was frozen in cold trap B which was then heated to obtain a sample of the fluid. This procedure was repeated two or three times or until a visual inspection



showed no discrepancies from the original.

A liquid flush followed the vapor flush using a similar flow procedure. Liquid flushing does a better job of cleaning flow through areas than a vapor flush could. A vacuum was drawn in each loop, and the NA 500 was pumped in from the low point (dump tank) to the high point (the expansion tanks in NaK loops and the condenser inlet in Hg loop, see fig. 2). When the loop was filled, it was soaked for 1/2 hour to reach the nonflow areas and then gravity drained. The residue was chemically analyzed to determine the type of system contaminants, usually a hydrocarbon (grease or oil). One liquid flush operation produced a rubber compound that was surmised to be either a small piece of rubber hose or part of a stopper that had broken off and been left in the system. At least two or three of these flushes had to be made until a chemical analysis of the solvent after the 1/2 hour soak proved to have the same composition as the original solvent. An acetone flush by gravity followed the NA 500 flush. Pure acetone was used mainly to remove any residual NA 500. Acetone, being more volatile, dries faster and leaves no residue. No soak period was used because the system was considered to be relatively clean and draining by gravity was the easiest way to remove all the solvents.

After flushing, each loop was sealed and evacuated, followed by a hot (300° F or 420° K) argon purge. This was repeated three to five times to evaporate any of the remaining solvent material and also to help outgas the system. The argon was heated using a wire heater coiled around 50 feet (15 m) of copper tubing, mainly to keep the gas dry, but also to speed up the evaporation rate of the cleaning solvents. During these gas purging periods, the flexible metal hoses in the system were heated with an electric dryer to ensure liquid removal because these hoses were potential liquid traps. Line heaters were used wherever possible when pulling a vacuum in the system to aid evaporation and outgassing.

The NaK loops were cleaned as an assembly only once. After the first fluid injection, the potential fire hazards of cleaning an entire NaK contaminated loop were too great for the testing area involved. The Hg loop, however, was cleaned three times before actual operation: an initial cleaning, a second time because of a hydrocarbon oil leak from the Hg pump lubrication system, and the third time following the installation of a different type boiler. In this latter case, the Hg loop was flushed in two halves (the final closure welds had not been made): the boiler outlet to the condenser outlet and the condenser outlet to the boiler inlet (fig. 2). The boiler itself was cleaned by three 1/2 hour soaks and gravity drains using alcohol as the solvent to remove the contaminants followed by 300° F (420° K) argon purges. An acetone flush and another hot argon purge followed. Wherever possible, several solvents were used because every solvent has preferential cleaning properties for the various organic contaminants expected. A combination of solvents will remove a wider range of contaminants (ref. 6).

## Preoperation

Filling the NaK system is a critical operation because NaK oxide formation can cause plugging in the lines. A schematic of the fill operation is shown in figure 5. A vacuum of 0.02 torr ( $2.7 \text{ N/M}^2$ ), which was close to the maximum capability of the loop was drawn before filling. The fill system was set up by use of proper valving so that all lines could be purged with argon from both ends before any NaK is transferred. The NaK is then transferred from the commercial drums into the dump tank. All fill or transfer operations are made through a type 304 sintered stainless-steel 20-micron filter in the fill system. The commercial fill system was removed and capped off before system operation. A hand valve was included in the fill line between the dump tank and the operating loops so that the loops could not be filled accidentally.

To fill the NaK operating loops, a vacuum was pulled on both loops and the NaK dump tank. The valve at the vacuum pump was then shut and the dump tank was pressurized with argon. Both the vacuum and the argon lines enter at the top of the dump tank while the NaK fill line is at the bottom of the tank. The loops were filled through a 20-micron filter to the desired level and then the fill valve was closed. The pressure in the dump tank was set at slightly above atmospheric pressure to prevent the possibility of air leaking into the system. A bypass line around the filter, entering at the top of the dump tank, is used as the dump line so that any NaK dumped would not go through the filter.

Although extreme care was used to prevent any oxygen from entering the NaK loops, a NaK hot flush was performed. This initial liquid-metal hot flush was performed to re-

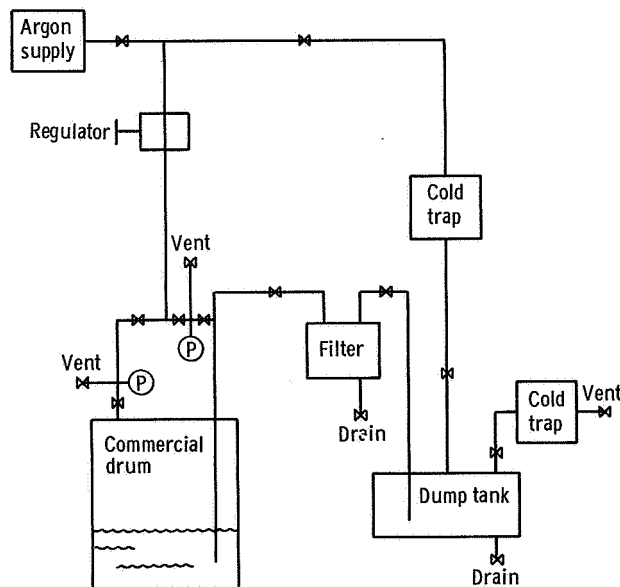


Figure 5. - Schematic diagram of NaK fill system.

move oxides from the walls of the system piping. It consisted of heating the liquid metal to temperatures that will dissolve any oxides that may have formed on the tube walls. In the system investigated, NaK was circulated for approximately 8 hours at 800° to 1000° F (700° to 800° K) and then dumped hot into the dump tank to precipitate out the dissolved oxides. The oxides either precipitated on the walls of the dump tank or floated on the free surface to the liquid metal. The NaK was allowed to cool to ambient temperature before the system was refilled. The loop was then refilled from the bottom of the dump tank, and plugging temperature runs were made. If the oxide content was greater than 50 ppm, corresponding to 400° F (480° K) on figure 6 (taken from ref. 7), another hot flush was made, but, if the level was satisfactory, data runs could be initiated. Usually only one hot flush was needed.

Charging the Hg system was done in an overly simplified manner. Mercury was poured into a funnel with a valve on the end leading to the Hg expulsion tank. After the desired amount of Hg was added this valve was closed. To prevent any chance of air entering the system, the Hg should be filled into a vacuum. Another, but not equal, method would have been to use a dip tube so that the liquid mercury would displace any

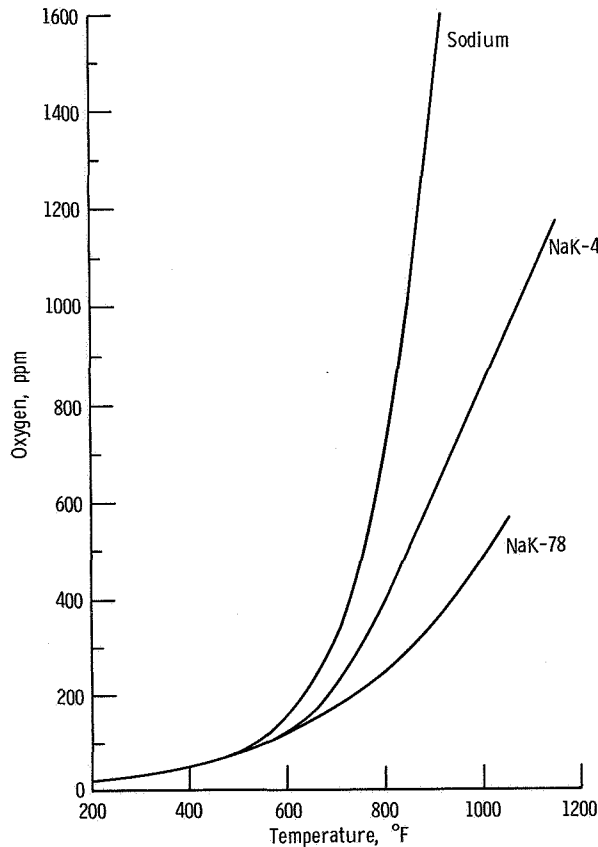


Figure 6. - NaK oxide saturation temperatures. (See fig. 2, ref. 7.)

air in the expulsion tank and prevent entrapment of air along the sides of the expulsion tank.

To fill the operating loop, a vacuum was pulled on the loop including the dump tank. The system fill valve was closed and the injection valve was opened and then closed to allow some Hg to flow into the dump tank and allow any entrapped gas to escape. The system was again evacuated and, when the maximum loop vacuum capability was reached (10 to 15 microns or  $1.3$  to  $2.0 \text{ N/m}^2$ ), Hg was forced into the operating loop by opening the fill and injection valves and pressurizing the expulsion tank. These valves were shut when the desired amount of Hg was added.

## Operation

During operation, care was taken to maintain loop cleanliness. All pressurization lines and all vacuum lines to both NaK and Hg loops had cold traps between their source and the loop, and also between the loop and the atmosphere (vent lines). These cold traps were stainless-steel cylinders immersed in liquid nitrogen and maintained at temperatures below  $-60^\circ \text{ F}$  ( $220^\circ \text{ K}$ ). Contaminants from an external source must first pass through the cold trap where they will be solidified and trapped out. On the other hand, leakage of fluids out of the system will also be prevented by these traps. In practice, these traps prevented water vapor from entering the loops and froze any that might enter the cold trap.

Alkali metal loop operation is continually plagued with plugging problems. To combat this problem, an oxide control loop was incorporated to precipitate any oxides formed. Using the theory that oxides will migrate to the coldest part of a system, it was desired to control where oxide formation could occur. A tap from each NaK loop was run to the oxide control loop (as seen in fig. 2) and the NaK was allowed to flow through a cold trap. The cold-trap temperature was maintained by air cooling at approximately  $20^\circ \text{ F}$  ( $10^\circ \text{ K}$ ) below the oxide saturation temperature as determined using the plugging indicator valve in the oxide control loop. As the oxide content of the NaK loops was lowered, the cold-trap temperature was reduced at a maximum rate of  $20^\circ \text{ F}$  ( $10^\circ \text{ K}$ ) per hour to less than  $200^\circ \text{ F}$  ( $370^\circ \text{ K}$ ), corresponding to an oxide content of 20 ppm. This plugging temperature reduction rate was kept low to ensure the precipitation of the oxides into the cold trap. Rapid cooling or large temperature differences between the oxide saturation temperature and the cold-trap temperature would cause the oxides to precipitate too rapidly and possibly cause plugging in an undesirable area. The much lower temperatures maintained in the OCI loop compared with the operating loops was responsible for its high susceptibility toward plugging (approximately 30 percent of the plugs formed in that loop).

Stagnant legs produced approximately 40 percent of the plugging problem areas. Total nonflow paths included the expansion tank lines, and all instrumentation lines, while temporary nonflow paths, included the transfer lines to the OCI loop, dependent on which loop was being cold trapped, and the OCI loop itself, if it was not in use. Line heaters were necessary on all these nonflow and potential nonflow lines so that they could be maintained at the same temperatures or slightly higher than the adjacent temperature of the flowing stream. Constant monitoring of these temperatures minimized the possibility of plugging the nonflow paths.

## Repair and Reassembly

System changes or repairs were another problem area for contaminant control. On system shutdown, a slight inert gas pressure was put in the loops and the dump valves were left open for several hours to aid in drainage of the system except for the unavoidable pockets. Before any cutting was done, the section that had to be changed or repaired was isolated by valving off the rest of the system. A small positive pressure ( $<1$  psi, or  $7000 \text{ N/m}^2$ ) was maintained on both sides of this section by means of a pressure regulator. When the cut was subsequently made, the flow of inert gas would keep air (mainly the oxygen and water vapor) from entering the system and kept the pressure at a minimum.

All preparations for changes were made before the system was cut so that the loops would be open a minimum time before reassembly, usually not more than 24 hours. In the case of Hg loop entries, the ends of the cut were capped with electrical tape. A small pinhole was made in the tape to maintain a low-pressure purge and prevent the entrance of oxygen until the system could be rewelded. Before cutting into a NaK loop, dry ice ( $\text{CO}_2$ ) was wrapped on both sides of the cut, for approximately one foot back, to freeze any NaK lying in the line and prevent it from flowing out. After the NaK loop was opened, a damp cloth was used to clean each end of the cut up to one foot into the pipe. Both ends of the cut were fitted with rubber plugs and compression fittings until the loop could be rewelded. A small positive pressure or purge was maintained at all times that the system was open or not in operation. The inert gas pressure or purge was maintained to prevent the entrance of oxygen, while the pressure was kept low so that the stagnant NaK could not be forced out of an open section of line.

Components previously used in a liquid-metal system cannot be cleaned in the same manner as an unused component. Mercury contaminated components were first washed in trichloroethane to remove any grease and oil on the surface. The part was then soaked (pickled) in pure nitric acid if no structural material compatibility problem existed and then steam cleaned. This procedure was repeated until less than 0.05 milli-

gram per cubic meter of Hg could be found on a Hg detector. The component was then flushed with water and either baked or purged with hot argon. NaK contaminated components were steam cleaned until no further reaction with the NaK or NaK oxide could be seen. The part was then washed in trichloroethane and dried using an oven bakeout or hot argon purge. After cleaning, the component was evacuated to evaporate any remaining moisture, and leak checked. Finally, the ends were capped to prevent the entrance of dust and other foreign materials.

Reinstallation of a previously used liquid-metal component created the only other major cleaning problem area. The part was prepared for welding by degreasing in trichloroethane and then acetone, and vacuum drying. As much bench welding as possible was done so that only two system welds had to be made in any area. Welding was done by a tungsten - inert gas process using an argon purge. A fine control regulator was used to combine the system purge and the welding purge.

In the Hg loop, the ends of the cuts were prepared taking precautions that no filings entered the piping. In the NaK loop, the pipes were recleaned 1 foot back using a damp cloth and the ends of the cuts were prepared as in the Hg loop. Dry ice was packed around the pipe to freeze any possible NaK in the area and prevent leakage into the weld causing contamination. The loop purge was again used as the welding purge. All liquid-metal system welds were both dye-penetrant checked and X-rayed to ensure weld quality, while noncritical area welds such as in the vacuum or argon lines were dye-penetrant checked, as a minimum test.

## DISCUSSION

The cleaning procedures described dealt mainly with the problem of loop conditioning and maintaining contaminant control in a highly complex system. A minimum delay time between fluid injection and good data acquisition, as determined by the design requirements of the boiler and the condenser (shown in table I) was the primary goal. For the system investigated this was approximately 240 to 300 hours of three-loop operation as seen in the curves of boiler parameter against time (fig. 7). In this simulated SNAP-8 system, the boiler was the critical heat-transfer component to be conditioned since the data showed the condenser to be oversized. The boiler parameters monitored to determine conditioning were quality, degree of superheat, terminal-temperature differential, and outlet enthalpy. Vapor quality was calculated using a heat balance method and agreed with the ratio of vapor flow to liquid flow as measured by venturis. The amount of superheat was calculated by subtracting the Hg saturation temperature from the Hg outlet temperature. Terminal-temperature difference is a measure of heat exchanger effectiveness and, in this case, is the NaK inlet temperature minus the Hg out-

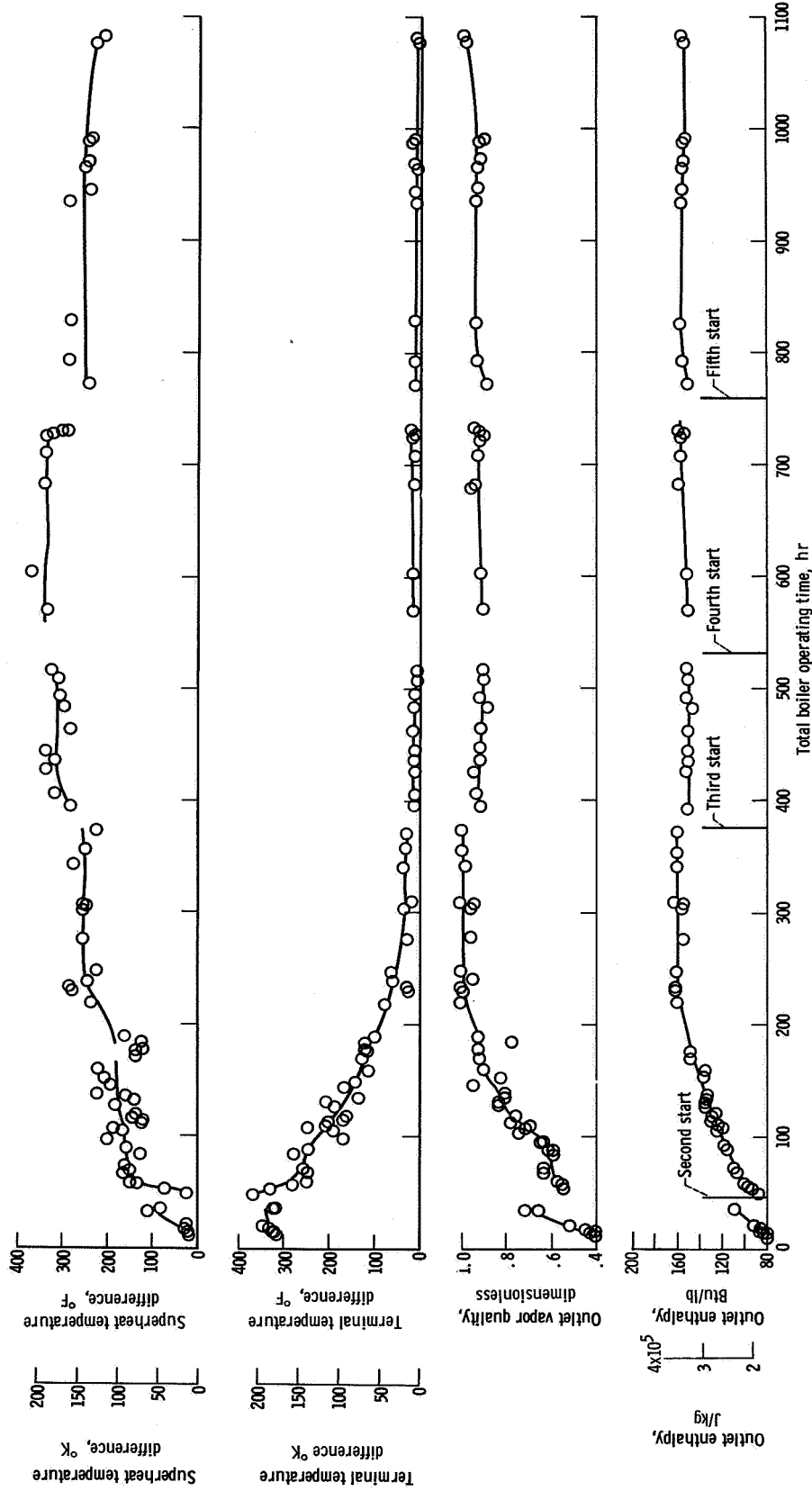


Figure 7. - Variation in boiler parameters during boiler history (taken from figs. 4 and 5 of ref. 10).

let temperature. The outlet enthalpy as calculated from a heat balance is probably the best single indication of boiler conditioning. A conditioned system produces increased heat-transfer performance. Secondary goals of the system included contaminant control during operation, contaminant control on inadvertent entry or planned alteration, and minimum turn around time from shutdown to startup.

Oxygen and hydrocarbon contaminants are the main cause for a deconditioned system (refs. 1 and 2). In a Hg system, wetting is a good indication of loop performance, and this can be monitored by progressive changes shown by temperature profiles, specifically in this case as seen by boiler thermocouples. Mercury will not wet a dirty system and quite often not even a supposedly clean system. An oil film will prevent the Hg from wetting which in turn prevents good heat transfer. Unpublished data produced at Lewis has shown that an oil film 6 angstroms thick ( $2 \times 10^{-8}$  in. or  $6 \times 10^{-10}$  m) can prevent conditioning. As seen by the data in table I the boiler reached approximately design conditions after 240 to 300 hours and the condenser was overdesigned. The condenser design overall heat-transfer coefficient was 1000 Btu per hour per square foot per  $^{\circ}\text{F}$  ( $2 \times 10^7$  J/(hr)(m<sup>2</sup>)( $^{\circ}\text{K}$ ) actual overall heat transfer coefficient was 2 to 3 times greater.

As reported in reference 8, when 25 grams of oil were injected into a similar Hg loop the effect on the boiler was noticed immediately and after 45 minutes the boiler outlet temperature dropped from  $1270^{\circ}$  to  $1100^{\circ}$  F ( $961^{\circ}$  to  $867^{\circ}$  K). Wetting additives such as rubidium can be added to the Hg system which can shorten the conditioning time considerably. In Hg samples taken from loops using rubidium additives (ref. 9), it was found that the Hg itself was clean but contained rubidium oxide residue. Additives have not been shown to have purely catalytic effects on the system. From the heat-transfer standpoint, it is desirable not to use additives if possible since their effects on the heat-transfer coefficients is unknown. In the loop investigated, the boiler conditioned without additives and a Hg analysis after 384 hours of operation (the second shutdown as seen in fig. 7) using a sample taken from the Hg filter showed no contaminants by distillation.

Prevention of the entrance of oxygen and the resultant oxide formation was the major concern in the NaK loops. The initial cleaning removed any contaminants that were present in the loop piping, while the NaK hot flush removed any oxides remaining on the walls or present in the NaK itself brought in during the filling operation. The initial and preoperation techniques worked satisfactorily as found from the lack of problems in the first startup of the NaK loops. Electrical heater problems caused the loops to be shutdown and an electrical failure while the loops were under vacuum caused the NaK loops to partially fill and also allowed air in. The protections incorporated to prevent a recurrence were to put a hand valve in the NaK fill line to isolate the dump tank and to make all valves leading to the atmosphere normally closed.

A solid oxide plug forms because too much oxide concentration builds up in one area. This type formation usually occurred in a valve seat, a flow restriction (such as venturi,



orifice, or small size tubing), a dead leg where no flow occurred, or a cold spot in the line. In the system investigated approximately 100 plugs formed, 80 to 90 of them before the first 100 hours of running due to the electrical power failure. Ten percent of the plugs occurred in valves, 10 percent in other flow restrictions, 40 percent in stagnant lines, and 40 percent in cold spots (OCI loop and radiator area). Each plug can cause up to several days of system running to dissolve the plug or an average of 200 man-hours to shut down a system, cut out the plug, reassemble the system, and start up again. Plugs in valves and flow restrictions can only be minimized by minimizing these areas in the original design. Stagnant legs should be minimized, but, as in the case of some pressure instrumentation where standoffs are necessary for temperature requirements, the legs must be provided with external line heaters and their temperature maintained at or above system temperature. The diaphragms having standoff legs were not insulated, thus, their temperature limits were not exceeded. Temporary stagnant lines such as bypass lines should be opened periodically to maintain a clear flow passage. Potential cold spot should be noticed in the original design and provided with heaters. The OCI loop, a planned cold spot, was the major cold-spot plugging area, but sufficient line heaters enabled most of the plugs to be opened shortly after inception.

An oxide plug formation could be removed in several ways. Getting flow to the plugged section was most important. If the area was not completely plugged and would allow some flow, erosion and washing away of the oxide formation could occur. The heat brought to the area by the hot NaK flow also tended to dissolve the NaK oxide and wash it away. External heating either by line heaters or a torch helped to dissolve or melt the plug if some flow could be initiated. If removing the plug from within was found impossible, the section of piping containing the plug had to be cut out of the system.

An inert gas purge was maintained in the open NaK loop until the loop could be capped so that oxygen would not enter and form a plug in another section of the system. A purge was used rather than pressure to prevent a pressure buildup with the resultant possibility of forcing stagnant NaK out of the open section. The proof of the plug removal procedures was verified after the electrical power failure which vented the NaK system to atmosphere while the loops were under a vacuum. Many plugs of different degrees of severity formed causing many nonflow areas and flow restrictions in the loops. Most of these plugs were removed after several days of applying heat and, using as high a pressure differential across the plug as possible to break it, allowing flow. The plugs that were not dissolved, were cut out and the system rewelded using a new or cleaned section of piping or valve. Upon restart, no new plugs formed and the system was subsequently operated for over 1500 hours actual running time with only minor partial plugs forming, using the oxide control loop. No oxide problems occurred, after the electrical failure, using the procedures described even when a small leak developed in the boiler area which caused the system to be exposed.

## CONCLUDING REMARKS

The solutions presented worked satisfactorily in the system investigated. Preparatory time for system operation, system conditioning time and operating problems were minimized and contamination of the system fluid materials was shown by analysis to be negligible. The factors contributing to the successful operation are enumerated as follows:

1. Cold traps were incorporated in all lines between the operating system and external sources to minimize the possibilities of impurities entering and exiting the auxiliary systems.

2. Contaminants in the pressurizing gases were monitored to keep them at a minimum. Fluid materials used in the operating system were procured as contamination free as possible, such as triple distilled mercury and reactor grade NaK. Filters of a 20-micron size were used to remove particles that could affect the system.

3. Cold spots and nonflow areas were eliminated or incorporated with heaters in the original design to prevent oxide plug buildups in undesirable areas. A deliberate cold spot was incorporated in an auxiliary oxide control loop to precipitate oxides in a controlled manner where they do not affect system operation.

4. An inert gas system was incorporated so that, in the case of failure or of loop entry at any part of the system, a purge could be maintained to prevent the entrance of air and water vapor by diffusion.

5. The cleaning method selected for contaminated parts was determined by the specific compatibility problems of each component and the contaminant to be removed. Welding is particularly susceptible to contamination by the system fluids, which may result in failure of the weld during operation unless the proper welding technique is used and each weld is inspected.

6. The wetting phenomenon in mercury is highly sensitive to contaminants, and to produce the desired heat-transfer characteristics, contaminant control is of major importance.

A system should be designed with cleaning in mind. This means elimination of incompatibilities between cleaning solvents and component materials, allowance for easy accessibility for cleaning each component and for draining the cleaning solvents from all parts of the loop. In most cases, however, this type of designing is difficult because each component has its own material, structural, and design problems; therefore, cleaning problems are relegated to secondary importance.

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National Aeronautics and Space Administration,  
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TABLE I. - BOILER AND CONDENSER HEAT-TRANSFER PARAMETERS

Boiler condition	Outlet quality, percent	Temperature differential						Outlet enthalpy	
		Superheat		Terminal		Pinch-point		Btu/lb	J/kg
		°F	°K	°F	°K	°F	°K		
Design	100	210	116	20	11	---	--	162	$3.8 \times 10^5$
After 300-hour operation	97	250	139	35	19	125	69	160	$3.7 \times 10^5$

Condenser condition	NaK inlet temperature		Mercury inlet pressure		Mercury inlet temperature		Superheat		Overall heat-transfer coefficient	
	°F	°K	psia	N/m <sup>2</sup> abs	°F	°K	°F	°K	Btu/(hr)(ft <sup>2</sup> )(°F)	J/(hr)(m <sup>2</sup> )(°K)
Design	500	533	15.5	$107 \times 10^3$	680	633	-----	-----	1000	$2 \times 10^7$
Actual	450 to 500	505 to 533	10 to 15	$(69 \text{ to } 104) \times 10^3$	<sup>a</sup> 640 to 680	<sup>a</sup> 611 to 633	0 to 100	0 to 55	2000 to 3000	$4 \times 10^7$ to $6 \times 10^7$

<sup>a</sup>Saturated.