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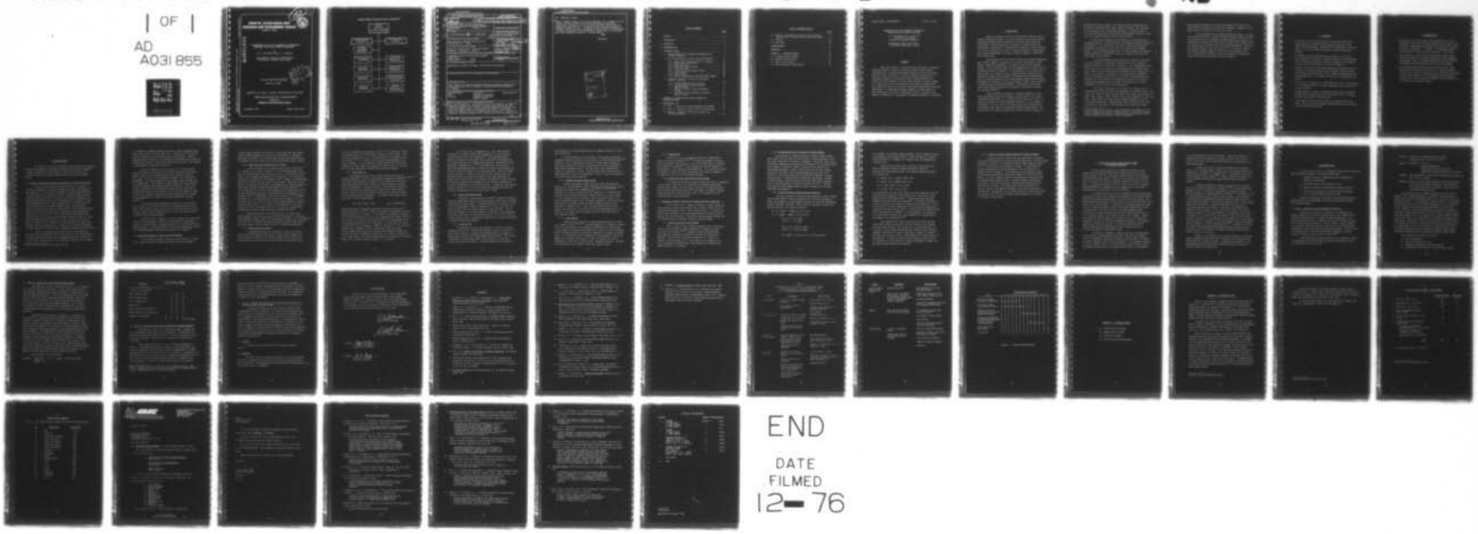

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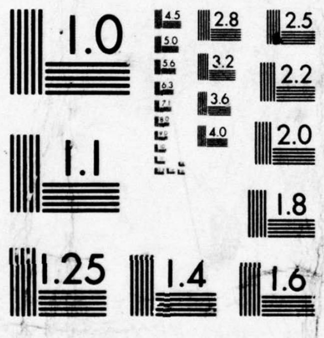
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TECHNIQUES FOR IN-SITU CLEANING OF HOMOPOLAR MACHINES WITH NaK CURRENT COLLECTORS

by
R. E. Witkowski and F. G. Arcella

Westinghouse Research Laboratories
Pittsburgh, Pennsylvania 15235

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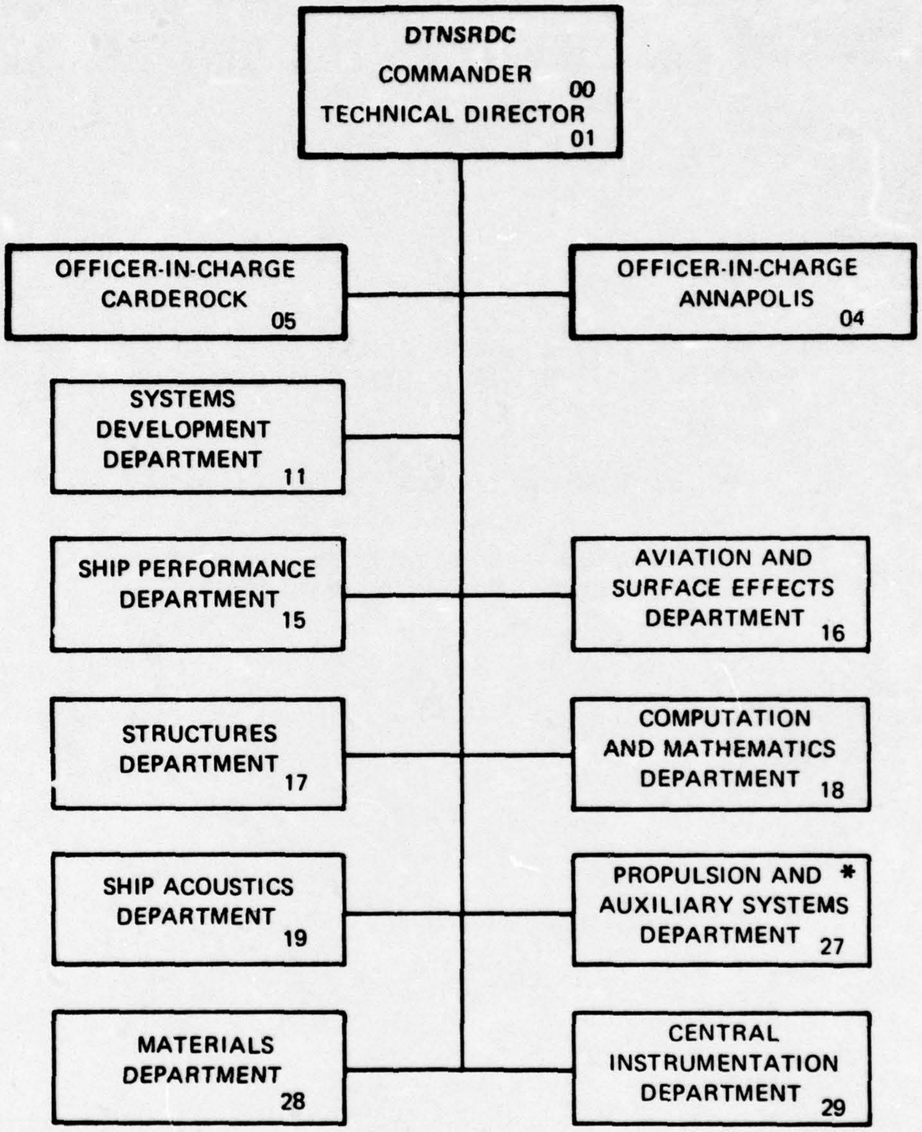

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(Authors)

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ABSTRACT

This report details a literature survey carried out to identify potential methods for periodic cleaning and removal of NaK, sodium-potassium alloy, from shipboard homopolar machines that use this liquid metal for current collection. The survey aided the definition of an experimental program for evaluating proposed cleaning methods to be carried out with the machine in place, either sealed or partially opened. As a result of this study a primary method and alternate methods have been identified, based on the specific constraints established by shipboard applications. An experimental program designed to further define the exact operations, which must be implemented in order to achieve a high degree of reliability in cleaning NaK from homopolar machines, is described.

1. INTRODUCTION

Homopolar machines that utilize liquid metal sliprings, such as sodium-potassium alloy (NaK), for current collection will require periodic shipboard maintenance when the machines are used for naval propulsion applications. The maintenance may be required for regular inspection, refurbishment, malfunction, or contamination of the NaK. In many instances it will be too impractical or costly to remove the machine for maintenance. Thus, on-board maintenance will require in-place cleaning to remove NaK from internal parts.

These machines will be located on board ship in engine compartment spaces where a number of constraints, not normally encountered in land-based or laboratory environments, are placed on possible NaK cleaning methods. From a personnel safety and environmental point of view, the following points must be considered: (1) the machine will be located within a closed space, (2) venting of toxic or explosive fumes and gases must be absolute, (3) safety and reliability of the method must be demonstrated, and (4) an applied shipboard alkali metal machine cleanup method should be performable by personnel who are only moderately trained, under conditions of limited space and available equipment. Additionally, the proposed alkali metal cleanup scheme must be compatible with machine structural and component materials.

When considering the design of a homopolar machine for shipboard applications, detailed consideration must be given to the cleanup requirements established; i.e., at some time the machine will need to be opened on board ship for some maintenance operation -- be it planned or unscheduled. If all or most of the alkali metal can be removed simply by draining, then cleanup of the machine internals becomes easier. This dictates that all internal piping should allow for gravity draining by a minimum of 3% slope in piping. The internal machine configuration must be such that the alkali metal cannot collect in pockets. The avoidance of cracks and crevices is considered essential. If possible, where cracks and drevices cannot be prevented, vacuum/pressure filling of these voids with compatible epoxies would be most desirable.

Even though these machine requirements are considered during the design stages of a homopolar machine, there may be instances when gross contamination of the alkali metal will lead to blocked or semiblocked passageways, giving rise to a much more severe alkali metal cleanup problem. Thus, blocked passageways must be opened through some mechanical means even before, or concurrently with, the initiation of an in-situ* alkali metal cleanup scheme.

A two-step program has been initiated as a means of arriving at the desired objective of defining a technique for in-situ cleaning of homopolar machines with NaK current collectors: (1) a comprehensive literature review has been carried out, surveying presently available alkali metal cleanup schemes and detailing an experimental plan designed either to adapt or to establish a new approach to shipboard homopolar machines, (2) this experimental plan is then to be utilized to define primary and alternate NaK cleanup schemes, to demonstrate feasibility by actually cleaning NaK-contaminated test specimens, and eventually to clean an experimental current collector or actual machine.

This report completes the first step of the program, that of documenting the literature review and presenting the experimental plan. The scope of the review includes accepted present-day methods and practices for alkali metal cleanup of many systems, such as large experimental liquid metal loops, liquid metal fast breeder reactor (LMFBR) components, and land-based homopolar machines. Although alkali metal cleaning is given prime consideration, other aspects such as general safety practices, alkali

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*In the context of this report, "in-situ" means that the machine is to be cleaned of NaK within the engine compartment space, preferentially sealed from the immediate environment. Depending upon the NaK cleanup method, partial opening of the machine may also be considered.

metal handling and disposal, cleanup fluid disposal, and eventual requalification of machines for use are also discussed. Appendix A of this report details the scope of the literature review.

The experimental plan includes four main task areas: (1) small scale laboratory studies to evaluate selected solvents, fluids, and gases for NaK reactivity and to determine reaction temperatures, products and mechanisms; (2) quantitative small-scale prototype cleanup studies, which will include small tubing with open and blocked passage ways, capillaries and possibly simulated cracks and crevices; (3) the design of primary and alternate cleanup schemes for this particular application; and (4) the quantitative evaluation of these two methods by cleaning NaK from a current collector system or an actual machine.

2. CONCLUSIONS

1. As a result of this literature survey, the proposed primary NaK cleanup method for homopolar machine current collectors is the moist inert gas technique. Modifications and adaptation of the basic method to an in-situ shipboard application require experimental testing.

2. An alternate cleanup scheme should be considered. The one that looks favorable and potentially feasible is the use of aqueous foams and/or emulsions coupled with ultrasonic techniques for the generation and stability maintenance of the foam or emulsion.

3. Because of the potential problems associated with explosive reactions occurring with potassium super-oxides and organic cleaning fluids, those methods which utilize pure (neat) organic compounds should only be considered when it is absolutely certain that no super-oxides are present.

4. Air or CO_2 may be employed as a passivating gas prior to the initiation of a NaK cleanup method; the machine temperature must be below 150°C, if CO_2 is employed.

5. Plugged channels or tubing, contaminated with NaK and/or compounds, may possibly be opened by the use of ultrasonics prior to or during the NaK cleanup methods discussed above.

6. If the machine can be partially opened and isolated from the NaK supply, then the controlled aqueous swabbing method may be considered to clean the machine internals.

3. RECOMMENDATIONS

1. Many methods have been discussed as being adaptable to in-situ cleaning of current collectors employing NaK as the current collection fluid. Experimental testing and verification of the primary and alternate approaches are necessary. An experimental plan to accomplish this has been written and made part of this report.
2. Once laboratory verification has been obtained, actual testing should be carried out on prototype machines in typical shipboard environments. Prime consideration must be given to the safety of operating personnel and associated facilities and equipment.
3. The literature review has shown that most alkali metal cleanup methods are presented and/or discussed in a qualitative manner. In order that a realistic analysis may be provided for extrapolation of methods and techniques to actual machines on board ships, an attempt must be made to quantize experiments and data generated in any subsequent experimental programs.

4. LITERATURE REVIEW

The overall scope of the literature review, detailing the documents searched and providing an annotated bibliography of pertinent references, is given in Appendix A of this report. A discussion of present day accepted alkali metal cleanup considerations and methods is presented below.

4.1 General Alkali Metal Cleaning Considerations for Homopolar Machines

Alkali metals require specialized equipment for handling, cleanup, and disposal as discussed by Mausteller, Tepper, and Rodgers.(1) This equipment is for the safety and comfort of the personnel performing the work, as well as to facilitate cleanup. Although the amount of NaK inside a machine may be small (10-100 cc), the confined environment of a shipboard application places strict requirements on all aspects of alkali metal handling and safety. Thus, personnel must be provided with protective clothing such as jackets, leggings, gloves, and face masks specifically designed for use with alkali metals. Self-contained breathing apparatus must be available, and fire extinguishers containing Metal-X* powder must be close at hand. Doors to the machine space should open outward and be of the push bar type (automatic closing); access to a high velocity overhead shower (fresh or salt water) is essential to remove NaK in the event that an individual is badly spattered with alkali metal. Personnel should be instructed in the proper use of protective equipment, and regular drills and practice runs of emergency procedures should be made.

Since the machine space on board ship will be confined area and alkali metal cleaning will take place in such an area, room ventilation systems equipped with filters must be considered essential and made part of the overall cleanup system. The ventilation system must be so designed

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*From Mine Safety Appliance Co., Evans City, PA.

to be capable of removing hydrogen gas and also caustic sodium/potassium smoke in the event of an alkali metal fire. Filters must be of the type that would remove this caustic smoke from the effluent gas. Ambient hydrogen monitors should also be considered, since hydrogen may be generated during machine cleanup. These should emit an audible alarm if the hydrogen level approaches the explosive point in air.

Specific considerations must be given to the actual machine system with regard to the selection of the most desirable alkali metal cleanup scheme. Since a homopolar machine will generally be placed back in use with most of its components unchanged after cleanup and maintenance, its requalification for use becomes a most important area for consideration. Such problems as caustic stress corrosion cracking, related to stainless steel components, must be addressed. Recent work by James and Borish have shown that precracked and preloaded solution-annealed 304 stainless steel specimens, which have been exposed to high purity liquid sodium and cleaned with a moist nitrogen process, showed no crack extension as a result of the sodium exposure and cleaning process.(2)

At machine operating temperatures (25-150°C), most structural metals such as copper and rotor iron will be compatible with the high purity liquid NaK environment and potential cleanup fluids, such as water, alcohol, organic fluids, or gases, such as air, CO_2, and CO, which may be considered as NaK passivating media.

Finally, machine retention of fluids or vapors after alkali metal cleanup must be prevented. Therefore, facilities must be available to carry out a hot gas purge or vacuum bake cycle prior to NaK injection and machine re-start. A technique such as this has been described for startup of a land-based homopolar machine after alkali metal cleanup.(3)

4.2 Na and NaK Cleaning of Large Loops and Components

This section critically reviews the state-of-the-art of alkali metal cleanup as related to large liquid metal loops and components currently being considered and utilized by the liquid metal fast breeder reactor (LMFBR) programs, both national and international. Although significant differences exist between these systems and homopolar machine requirements, the basic alkali metal cleanup requirements remain similar.

4.2.1 Alkali Metal Passivation Prior to Cleanup

In many instances, before sodium or NaK cleanup is initiated, the component to be cleaned is exposed to a passivating gas which reacts with the residual alkali metal and converts it to a less chemically active state. This is especially important if the component is to be inspected or opened to the ambient air environment prior to cleanup. Such passivating gases as air and CO_2 have been used for this purpose. While CO_2 has been employed to passivate Na and NaK in large LMFBR reactor components prior to alkali metal cleanup (see Enrico Fermi Atomic Power Plant Report),(4) air has been used in at least two NaK homopolar machine applications.(5,6) The reaction between sodium and carbon dioxide is self-limiting by crust formation until the temperature is raised to at least 250°C,(4) although Cordfunke, et al. have indicated that sodium reacts rapidly with CO_2 at temperatures as low as 175°C, the reaction rate being dependent on temperature and surface area.(7) For sodium at room temperature, the reaction with CO_2 is slow; approximately 100 hours for 0.085 inch of penetration, asymptoting at 0.16 inch after 250 hours of exposure to CO_2.(4)

Carbon monoxide has the potential to act as an alkali metal passivating gas, but because of its toxicity(8) and ability to form alkali metal carbonyls, $(NaCO)_n$, $(KCO)_6$,(9) and acetylene diolates; e.g., $NaOC{\equiv}CONa$,(10) it should not be considered as a passivating gas for alkali metal systems requiring cleanup. This is especially true for shipboard applications.

4.2.2 Alkali Metal Distillation

A technique which has been considered as a means of removing alkali metal from components and systems is distillation (evaporation). From a theoretical point of view, the distillation process offers some advantages, such as there being no reactive fluids or gases employed and no toxic or flammable gases generated. Also, it is a simple and reliable (controllable) process for the complete removal of alkali metals. From a practical point of view, it is a process difficult to implement and is not economic.(11,12) A number of other practical disadvantages can be cited, including: (1) the inability to remove alkali metal oxides and compounds, (2) the slowness of the process is slow, and (3) the requirement for high temperature (750-800°F) and isothermal heating.

4.2.3 Moist Inert Gas

This method appears to be a reliable technique and has been utilized extensively to clean up alkali metal contaminated components.(11,13-15) It has been used extensively to remove both the alkali metals and their associated compounds from many systems of complex geometry, including those containing small cracks and crevices. A most important consideration, when employing this technique as an alkali metal cleanup method, is the control of the chemical reaction involved; the reaction products and heat generated may be detrimental. For example, when sodium reacts with water in the following fashion

$$Na + H_2O \rightarrow NaOH + 1/2H_2 \uparrow \qquad -\Delta H_f = 34 \text{ Kcal/g-mole}$$

sodium hydroxide and hydrogen are generated as reaction products and a significant amount of heat is evolved; with NaK, the corresponding potassium hydroxide would also be formed. If the reaction is permitted to proceed in an uncontrolled manner, local hot spot temperatures may exceed 1100°C.(13) Thus, control of the reaction is essential and may be accomplished by the slow addition of moisture to the alkali metal. A recent study by Borisch and Maffei(11,13) has attempted to quantize the ability of the controlled steam-nitrogen technique to remove sodium from cracks and crevices. Results indicate that sodium can be reliably and repeatedly removed from annular crevices with diametrical widths from 2 to 58 mils, and depths of six to eight inches. The cleanups required moisture contents in the humidity range of 30 to 100% and exposure times of 4 to > 18 hours.

In an earlier paper, Bruggeman et al., have also cited the advantages of utilizing a moist inert gas mixture over pure steam for cleaning alkali metals from components.(14) When considering this method for systems which have many alkali metal paths, the elimination of parallel loops must be considered and prevented. Experimental results generated by Bruggeman et al.,(14) have dramatically shown that the moist inert gas cleaning method is superior to the pure steam method from the heat of evolution aspect of the reacting species. Since the temperature rise noted is a function of the rate of heat evolution due to the chemical reactions involved, the presence of the inert gas serves to remove some of this heat and also decreases the quantity of water reacting. The inert gas probably also contributes to the diffusion resistance of the water in migrating to the sodium interface. In addition, the increased volume of the gas due to dilution increases the residence time and thus decreases the opportunity for the water vapor to react.

4.2.4 Water Mist-Atomized Spray

A technique or method detailed by Pomie(9) consists of atomizing water with a gas under pressure and having this mist react under controlled conditions with alkali metals. A primary advantage of this method is that it can be accomplished at ambient temperature, is easy to use, has very good efficiency, and the overall quantity of liquid is small. From a practical standpoint, this method appears to offer many of the advantages of the moist inert gas method discussed in Section 4.2.3, but it requires further studies to gain a quantitative insight into its direct applicability to homopolar machine requirements.

4.2.5 Inert Gas Foam

As detailed previously, see Sections 4.2.3 and 4.2.4, aqueous cleaning of alkali metal contaminated components appears to have had the most interest and use; a suitable dilution medium must be provided to prevent rapid reactions and extreme temperature excursions. This has been accomplished by using either steam or water aerosols in conjunction with an inert gas as a dilutant. A further extension of this method is the utilization of a high expansion foam, the bubbles of which are filled with an inert gas.

A foam technique for the cleaning of alkali metal contaminated components has been detailed by Bray and Donn in a recent British patent.(16) Basically, a 5% by weight aqueous solution of sodium lauryl sulphate is foamed with nitrogen, so that the nitrogen-filled foam can be sprayed on alkali metal contaminated parts. The used foam is then collected and allowed to collapse into a drain. The disclosure further indicates that, where potassium is one of the contaminants, there is a very low probability of potassium super-oxide reactions.

4.2.6 Alcohols and Other Organic Fluids

Alcohols and organic fluids with active -OH or -OR functional groups; e.g., 2-butoxyethanol, (C_4H_9-O-CH_2CH_2OH) have been used and studied extensively as alkali metal cleaning fluids.(3,9,11,12,14,15,17-20) Basically, the reaction rate, and thus the temperature of reaction, can be controlled by the proper selection of organic fluids; i.e., the blocking or steric hinderance effect supplied by inert functional groups to the reactive functional group can be selectively determined. The basic problem which exists with organic fluids as sodium cleaning solutions is the potential for chemical fires. A more crucial problem exists for NaK; namely, the potential for the reaction of the potassium super-oxide. The reaction of the super-oxide with organic fluids is rapid and can occur with explosive violence.

4.2.7 Liquid Ammonia

Alkali metals are soluble in liquid ammonia, which may also serve as a cleaning fluid.(3,10,12,21) Although suggested as a potential cleaning fluid for large components and assemblies contaminated with alkali metal, to date ammonia has primarily been used only on laboratory scale experiments. The use of ammonia suffers from two basic drawbacks as a potential cleaning fluid: (1) it requires either refrigeration or pressurization to keep the ammonia in the liquid state, and (2) it does not dissolve the oxides, hydroxides, or other salts of alkali metals. Equipment which has contained Na and ammonia may have residues of Na, Na_2O, $NaNH_2$ and NaOH.

4.2.8 Liquid Metals

Liquid metals have been suggested and used to remove alkali metals from reactor components.(12,17) Although successful for the particular application involved, such as the use of molten lead to clean and seal faulted fuel elements,(17) the technique has had only limited use and acceptance. The principal disadvantages are: (1) an additional method is required to remove the second liquid metal film from the component, (2) high cost, and (3) difficult disposal problems.

4.2.9 Special Problems Associated with NaK Cleaning

The cleaning of NaK alloys requires special care since, in addition to their greater reactivity, the possibility of potassium super-oxide reactions with organic materials leads to the risk of violent explosions. These criteria are detailed by Davis(9) and suggest that organic cleaning fluids should only be considered when there is no possibility of the potassium super-oxide being present.

4.3 Cleaning of Homopolar Machines Utilizing NaK Current Collectors

At the present time, a number of laboratory cleanup schemes are being employed to remove alkali metal from a variety of small scale and plant-sized land-based systems. These schemes include: (1) aqueous submersion and/or washing. (2) nitrogen/steam lancing, followed by aqueous washing, (3) alcohol reaction, followed by steam and aqueous washing, and (4) controlled aqueous swabbing, followed by aqueous washing.

4.3.1 Aqueous Submersion and/or Washing

Small or large current collector components can be effectively cleaned or alkali metals by simple submersion or washing with water, followed by oven drying.(3,5,6) Although a highly reliable methods, this technique is inherently difficult to control once cleaning has commenced. Reaction rates are rapid, hydrogen gas is evolved, and vigorous reactions usually occur. This method is only employed in an open, uncontained environment where remote handling of components and cleaning fluids can be achieved.

4.3.2 Nitrogen/Steam Lancing Followed by Aqueous Washing

This technique is presently employed to remove residual alkali metals (NaK) from both small and large current collector components which have small, physically inaccessible, passageways. Basically, alkali metal contaminated components are preoxidized with ambient air, and then a purge of pure nitrogen is initiated through a steam lance with venting to the surrounding environment. Steam is then slowly bled into the nitrogen purge gas of the lance at ever increasing amounts, until only pure steam is fed through the alkali metal contaminated component or system. Generally, this is followed by water washing and oven drying. Although a reliable method, reaction rates are only semi-controllable, hydrogen gas is a reaction product, and vigorous reactions can occur. Likewise, this method of alkali metal cleanup is usually carried out in an open, uncontained environment.(6)

4.3.3 Alcohol Followed by Steam and Aqueous Washing

The use of alcohols is presently being employed to remove residual alkali metals (NaK-56) from both externally and internally contaminated piping and components of large homopolar machines at the Arnold Engineering Station, Tullahoma, Tennessee. Ethyl alcohol is employed; the following equations describe the reactions involved:

a) $Na + C_2H_5OH \rightarrow NaOC_2H_5 + 1/2\ H_2 + \text{heat}$

b) $K + C_2H_5OH \rightarrow KOC_2H_5 + 1/2\ H_2 + \text{heat}$

$23\ \text{lb. Na} \rightarrow 1\ \text{lb. } H_2(g)$

$39\ \text{lb. K} \rightarrow 1\ \text{lb. } H_2(g)$

$0.44\ \text{lb. Na} \rightarrow 0.019\ \text{lb. } H_2(g)$

$0.56\ \text{lb. K} \rightarrow 0.014\ \text{lb. } H_2(g)$

$1\ \text{lb. NaK-56} \rightarrow 0.033\ \text{lb. } H_2 = 9\ \text{ft.}^3\ H_2 \text{ at } 100°C$

As an example, the Tullahoma machine requires a 300 lb. charge of alcohol to decontaminate the homopolar machine. After the alcohol is drained from the machine, it is pumped into disposal tanks and transported to a disposal area.

After the pure alcohol wash, a mixture of 75% alcohol, 25% water is employed to remove the remaining NaK plus salts produced from the alcohol/NaK reaction. The probable chemical reactions are outlined below:

c) $NaOC_2H_5 + H_2O \rightarrow C_2H_5OH + NaOH + \text{heat}$

d) $KOC_2H_5 + H_2O \rightarrow C_2H_5OH + KOH + \text{heat}$

e) $Na + H_2O \rightarrow NaOH + 1/2\ H_2 + \text{heat}$

f) $K + H_2O \rightarrow KOH + 1/2\ H_2 + \text{heat}$

The alcohol/water solution, after reaction, is then pumped into the disposal tank and transported to the disposal area. Live steam is then passed through the Tullahoma machine. During this entire operation, hydrogen is liberated and heat is generated. To minimize the chance of a fire or explosion, the hydrogen is purged from the generator. This is accomplished by allowing nitrogen gas to flow through the machine, thus purging the hydrogen from the system. The machine is then dismantled and repairs and maintenance performed. After maintenance, all components are requalified for machine use. Upon completion of requalifications, the machine is then reassembled.(22)

This technique also relies on the fact that slower and more controllable reaction rates may be achieved through the use of higher molecular weight alcohols; the reaction rate, as noted previously, is a function of chain length and branching for a given alcohol structure. Component temperatures are usually monitored as a means of following the alkali metal cleanup process. Steam lancing and/or water washing is then employed. Although this method of NaK cleanup has been successfully employed in closed systems in confined environments, extreme care and highly trained personnel are required.

4.3.4 Controlled Aqueous Swabbing Followed by Aqueous Washing

A highly successful technique has been employed by the Westinghouse Research Laboratories to clean up NaK from the current collector region and machine internals of an experimental 3000 hp SEGMAG homopolar generator.(5) This method may be described as follows: (1) The machine is isolated, through valves, from the NaK supply loops and ambient air is allowed to fill the machine. (2) The machine is then physically opened and residual, oxidized NaK is mechanically removed from the internal components. (3) Water dampened swabs are then employed to react and remove the remaining small amounts of NaK. Although hydrogen is generated by this process, only small amounts are involved, thus explosive environments are not a problem. This method does require the system to be initially opened to the ambient environment, thus exposing personnel to oxidized NaK. Although they have not been encountered, alkali metal burns are possible and, to date, only highly trained personnel have carried out this NaK cleanup process.

5. DISCUSSION OF ALKALI METAL CLEANING SCHEMES FOR HOMOPOLAR MACHINES

For the existing application of homopolar machines to naval propulsion, where shipboard alkali metal cleanup offers its own set of unique problems, none of the individual methods covered in the literature review appears entirely adequate. Rather, a hybrid method must be devised that will satisfy the specific requirements detailed in Section 1 of this report. Each method discussed in Section 4 has its own set of advantages and disadvantages when compared with other methods, but at best these relationships are qualitative in nature, see Table I.

As indicated previously, the one technique that has received wide acceptance and use, and where quantitative information is available regarding alkali metal cleaning effectiveness, is the moist inert gas process. For homopolar machine applications, modifications of the basic method may yield a prime in-situ cleanup scheme. In addition to this method being used extensively at present, quantitative data are available regarding the removal of alkali metals from cracks, crevices, and small diameter piping. The potential problems associated with NaK and potassium super-oxide are not a principal concern, since organic fluids are not involved. Finally, this method is readily adaptable to shipboard use since the necessary components of steam and inert gas are generally available. The important aspect in utilizing this method is the required modifications that must be made and proven experimentally before shipboard use can be realized (see Section 6, Experimental Plan).

In addition to defining a primary alkali metal cleanup method in this report, alternates and/or new methods must also be considered, such as (1) hydraulic flushing, (2) aqueous or alcohol foams, (3) aqueous or alcohol emulsions, and (4) use of ultrasonics. As previously discussed, the use of organic fluids to flush NaK from the current collector region of homopolar machines must only be employed when there is no possibility of the potassium super-oxide being present. Since this is difficult to determine, even when components are directly visible, from a safety point of view, pure (neat) organic fluids should not be employed in the NaK cleanup scheme.

A method which offers the greatest potential for becoming a viable in-situ cleaning method for NaK is the use of aqueous foams and/or emulsions as a secondary or alternate method. A modification of the previously discussed foam technique(16) to make it directly applicable to an in-situ homopolar machine NaK cleaning technique appears potentially feasible.

A further consideration would be to couple the foam emulsion method with ultrasonic generation techniques,(23,24) since ultrasonic NaK wetting of current collectors, as discussed by Arcella and Witkowski, may be considered for shipboard homopolar machines.(25) Ultrasonic methods of emulsion generation have a special feature; namely, the possibility of obtaining concentrated, highly-dispersed emulsions without the use of surface activating agents. This would be especially true of a NaK cleanup method where the addition of unwanted additional materials, such as activating agents, must be minimized. In general, low frequencies are required for emulsification. At a frequency of 5 Mc/sec, very intense beams are required to cause emulsification. Alternatively, sound waves of a frequency of less than 20 kc/sec are audible. Thus, the use of high intensity sound waves of low frequency is objectionable both physiologically and sociologically. Therefore, 20-50 kc/sec appears to be the recommended frequency range for efficient emulsification.(24) In addition to serving as a method for generating and maintaining aqueous foams and emulsions, ultrasonics have the potential for aiding in breaking up oxide plugs in tubing, channels, and crevices.

Finally, if the machine can be partially opened, the controlled aqueous swabbing technique may also be considered as an alternative NaK cleanup method for machine internals. Although used successfully at the Westinghouse Research Laboratories, additional testing is required to adapt the method for shipboard use.

6. EXPERIMENTAL PLAN

To carry on this effort, an experimental program will be initiated and carried out according to four separate task areas:

1. Laboratory studies - fundamental reaction temperatures, products and mechanisms.
2. Quantitative small scale prototype studies.
3. Design of primary and alternate NaK cleaning techniques.
4. Quantitative evaluation of the NaK cleaning techniques on a prototype current collection system.

A program schedule and a discussion of each individual task is presented below. All experimental data obtained during this program will be made available.

6.1 Task I - Laboratory Studies-Fundamental Experiments

Laboratory studies will be carried out to investigate the chemical interactions of passivating gases and selected cleanup fluids, including those necessary for foaming and emulsion generation. The chemical compounds necessary to maintain and cause the eventual collapse of the foam or emulsion system will also be included. Special emphasis will be placed on possible reaction products and their influence and/or interaction on machine materials and associated equipment. This task will also interface with a concurrent program task designed to study NaK wetting of homopolar machine current collectors.(25)

Selected NaK-wetted copper specimens will be chosen and cleaned by reacting the NaK wetted surfaces with specific gases and/or fluids. Test conditions will be as follows:

Apparatus: Argon-filled cleaning chamber (isothermal)
Temperature control range, 25 to 150°C

Fluids to be Evaluated: Air, CO_2
Moist nitrogen/argon
Aqueous foam (5% lauryl sulfate)
Experimental aqueous emulsion (to be defined)

Variables: Time, temperature, cleaning fluid composition/concentration, gas flow rate, pressure

Evaluation: Visual, reaction product characterization and identification by gas phase mass spectrometry, x-ray diffraction and infrared spectroscopy techniques, cleaned surfaces by scanning electron microscopy

An inerted gas reaction chamber, equipped with a gas sampling manifold, will be employed initially to evaluate cleanup fluids and to obtain chemical kinetic information which will be required to extend the laboratory studies to a viable machine oriented technique. The specimens which have been used in the NaK wetting experiments will be transferred to this chamber and selected NaK passivating gases (air and/or CO_2) will be introduced. Following passivation, cleaning will be initiated with moist nitrogen/argon mixtures or an aqueous foam at selected temperatures and periods of time. Gas samples of the cleaning chamber environment will be taken as a function of time and analyzed by gas phase mass spectrometry. Dehydration of the reaction products will follow; analysis of the products will be by x-ray diffraction and infrared spectroscopy. The cleaned copper tabs will be examined by scanning electron microscopy, with associated electron excited x-ray analysis (EDAX) yielding information regarding surface morphology and elemental compositions.

Data presentation will be in the form of:

a) visual observations
b) cover gas composition vs. time
c) compound identification (reaction products)
d) micrographs and EDAX spectra of cleaned copper surfaces.

6.2 Task II - Quantitative Small Scale Prototype Studies

This task will function concurrent with Task I. The transition from chemical laboratory experiments to larger scale studies will aid the definition of methods and equipment necessary to design primary and alternate NaK cleaning methods. Small scale experiments will be carried out with small bore tubing (capillaries) and/or simulated cracks and crevices. These test pieces will be wetted with NaK and prepared in such a manner that both open and plugged passageways will be tested. An attempt will be made to quantify the data generated by this effort in such a manner that extrapolation to actual machine systems might be predicted.

Copper and 304 stainless steel tubing (1, 3, and 6 mm I.D.) will be wetted by heating and filled with NaK and either drained (gas pressure or vacuum) or exposed to the ambient air environment so that "plugs" will form. Employing the same inert gas-filled chamber, these tubes (100 mm in length) will be exposed to a passivating gas and either moist nitrogen or an aqueous emulsion or foam; ultrasonic agitation will also be employed. All studies with the moist nitrogen method will be carried out at 110°C or lower, while foam cleaning will be initially attempted at room temperature and adjusted if necessary. A dependent variable will be time, wherein initial exposures will be made for 30, 60, and 120 minutes. Acid/base titration of the remaining alkali metal (converted to hydroxide by reaction with water) and/or volumetric measurements of the amount of hydrogen evolved during this reaction, will be employed to obtain quantitative reaction rates and kinetics.

The test matrix is detailed below.

Specimens: 2 materials, 3 I.D.'s, 1 length, 2 conditions (opened, plugged) = 12.

Conditions	Exposure Time, Minutes			
	0	30	60	120
Air or CO_2 Passivate (selection from Task 1) all specimens				
Moist Nitrogen 30/70		12	12	12
Moist Nitrogen 50/50		12	12	12
Moist nitrogen 70/30		12	12	12
Aqueous Foam		12	12	12
Aqueous Foam With Ultrasonics		12	12	12
Aqueous Emulsion With Ultrasonics		12	12	12
Archives, No Cleaning	12			
	12	72	72	72 = 228 tests

6.3 Task III - Design of Primary and Alternate NaK Cleaning Techniques

Utilizing the results of the two previous tasks, a primary and one alternate NaK cleanup scheme will be designated. The necessary techniques and equipment that will be required to carry out a safe and reliable NaK cleanup of homopolar machine current collectors on board ship will be defined.

Since quantitative data will now be available regarding the ability to cleanup NaK from machine internals, including associated small pipes and capillaries (with blocked and unblocked passageways), a primary cleanup method and one alternate method will be designed. Incorporated into this design will be the strict requirements defined by shipboard use and the fact that only moderately trained individuals* may be carrying out the entire operation. A review of each NaK cleanup method will be made by

---

*These individuals must be trained in the safe handling and use of alkali metals. Specific skills such as alkali metal transfer techniques, disposal, and fire fighting methods are considered essential.

engineers cognizant of shipboard facilities and safety requirements. As part of the overall design, consideration will also be given to the safe disposal of cleanup fluids and to the operations which will be required to permit the machine to be sealed and refilled with NaK. The proposed NaK cleaning methods will be incorporated into a plan that will eventually be tested on a full scale machine.

6.4 Task IV - Quantitative Evaluations of NaK Cleaning Techniques on a Prototype Current Collection System

A quantitative evaluation of the primary and alternate NaK cleanup methods will be carried out on a current collection system which utilizes NaK as the slipring fluid. This test stand will simulate materials, passageways, and current collector seals that are typical of an actual machine, thus verifying the feasibility of the proposed NaK cleanup methods. Tests will evaluate the effectiveness of the methods for both clean and highly contaminated (blocked passageways) NaK systems and provide an assessment of cleaning effects on machine materials and components. An analysis will be provided to aid in the eventual definition of machine requalification requirements for startup and operation of a homopolar machine for shipboard use.

6.5 Schedule

A schedule for each individual task outlined is given in Figure 1.

6.6 Reporting

The final report will contain the results of all the experiments and tests. Complete test apparatus description and operating parameters will be described. One preliminary and one alternate NaK cleanup method for use on board ship will be recommended.

ACKNOWLEDGMENTS

This effort was supported by the David W. Taylor Naval Ship Research and Development Center (DTNSRDC) under contract number N00167-76-M-8066, with funding provided by Naval Sea Systems Command Project SSL25. The authors wish to acknowledge Ms. Mary Ann Braithwaite of the Westinghouse Research Laboratories Library staff for her assistance in the literature review and acquisition part of this study.

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3. Mole, C. J., "Design and Development of a Segmented Magnet Homopolar Torque Converter", ARPA Contract DAHC-15-72-C-0229, Phase I Report, March, 1973.
4. "Enrico Fermi Atomic Power Plant Unit 1", Report for the First Quarter of 1974, Docket-50016-228, 1974.
5. Private Communication with Mr. A. R. Keeton, Westinghouse Research Laboratories, Pittsburgh, PA.
6. Private Communication with Mr. C. Lizanich, NASA Lewis Research Center, Cleveland, Ohio.
7. Cordfunke, E. H. P., and Ouweltjes, W., "The Reaction Between CO_2 and Liquid Sodium", J. of Nuclear materials, 33, pp. 328-332, 1969.
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10. The Alkali Metals, Special Publication No. 22, The Chemical Society, London, 1967.

11. Borisch, R. R., and Maffei, H. P., "Rate of Sodium Removal from Crevices in FFTF LMFBR Equipment", HEDL-TME-73-32, November, 1973.

12. C. F. Braun and Co., "Optimization Study D - Sodium Cleaning and Disposal", NTIS PB 208 967, November 17, 1967.

13. Borisch, R. R., and Maffei, H. P., "Sodium Removal Processes and Material Requalification for LMFBR Hardware", HEDL-SA-661, 1973.

14. Handling and Uses of the Alkali Metals, Advances in Chemistry Series #19. A collection of Papers from the Symposium on Handling and Uses of the Alkali Metals, 129th Meeting of the ACS, Dallas, Texas, Published May 1957 by the American Chemical Society.

15. Poucher, F. W., and Kohl, A. L., "Cleaning Techniques for Sodium System Components", ANS Transactions, 11, 1, June, 1968.

16. Bray, John A., and Donn, Ian M., "Foam Cleansing of Components Contaminated with Sodium", British Patent No. 1,251, 842, 1971.

17. Arkhipov, V. M., et al., "Problems of Sodium Removal and Deactivation of Fast Reactor Circuits and Components", AEC-tr-7431, 1973.

18. Burwell, R. L., "Clevage by Alkali Metals", Chem. Rev., 54, 672 (1954).

19. Scott, J. M., "Sodium-Heated Steam Generator Model Tests at SGTR, Volume 1", GEAP-10580-1, June, 1972.

20. Kilpatrick, M., et al., "Studies of Fast Reactions Which Evolve Gas. The Reaction of NaK alloy with Water in the Presence and Absence of Oxygen", J. Phys. Chem., 57, 4, pp. 385-390, April 16, 1953.

21. Pulham, R. J., and Berkey, E., "Laboratory Handling and Purification Practice for the Liquid Metal NaK", Westinghouse Research Report 72-8B6-LMSER-M3, January 4, 1973.

22. Private Communication with Mr. N. F. Craigue and J. N. Patterson, Von Karman Gas Dynamics Facility at the Arnold Engineering Development Center, Arnold Air Force Station, Tullahoma, Tennessee.

23. Berkman, S., and Egloff, G., Emulsions and Foams, Reinhold Publishing Corporation, New York, 1941.

24 Sherman, P., Emulsion Science, Academic Press, New York, 1968.

25. Arcella, F. G., and Witkowski, R. E., "Techniques for Surface Wetting of NaK to Copper for Liquid Metal Slipring Homopolar Machines", Research Report 76-6B6-NSRDC-R1, March 1976. (prepared under contract for the David W. Taylor Naval Ship Research and Development Center; Annapolis, Maryland).

TABLE I

Advantages and Disadvantages of Alkali Metal Cleanup Methods As Related To In-Situ Cleaning of NaK From Shipboard Homopolar Machines

Method	Advantages	Disadvantages
Distillation	no toxic or explosive gases generated no reactive fluids or gas required	cannot remove NaK compounds requires high vacuum requires high temperature and isothermal heating slow process
Moist Inert Gas	extensively used and studied quantitative data reported removes both NaK and compounds cleanup reactions can be controlled by water/gas ratio	hydrogen generated moderate temperatures involved condensation, water reaching "pockets" of NaK
Water-Mist Atomized Spray	Limited information available but appears to be similar to moist inert gas technique, ambient temperature involved removes both NaK and compounds	hydrogen generated only limited use reported - needs additional studies danger of liquid water reaching "pockets" of NaK
Foams and Emulsions	appears to be another reliable aqueous dilution method removes both NaK and compounds limited information indicates cleanup reaction rates and temperatures can be controlled cleaning may be carried out at low, ambient temperatures	not extensively studied difficult to maintain supported foams and homogeneous emulsions-needs additonal work hydrogen generated

Method	Advantages	Disadvantages
Alcohols and/or Other Organic Fluids	removes NaK easily	NaK compounds and reaction products insolubl
	reaction rate and temperature easily controlled by specifying alcohol chain length or substitution (steric hindrance)	potentially explosive and/or toxic vapors; chemical fires
		hydrogen generated
		potential of dangerous reactions with potassium super-oxide.
Ammonia	very controlled reaction with NaK, direct solution	low temperature and/or high pressure required
		not tested in large systems
		toxic vapors
		does not remove NaK compounds or reaction products
Liquid Metals	no toxic or explosive vapors	does not remove NaK compounds
	removes NaK by direct solution-low heat of reaction	introduces another liquid metal which must be removed
		only limited use reported
		difficult disposal problems
		high cost

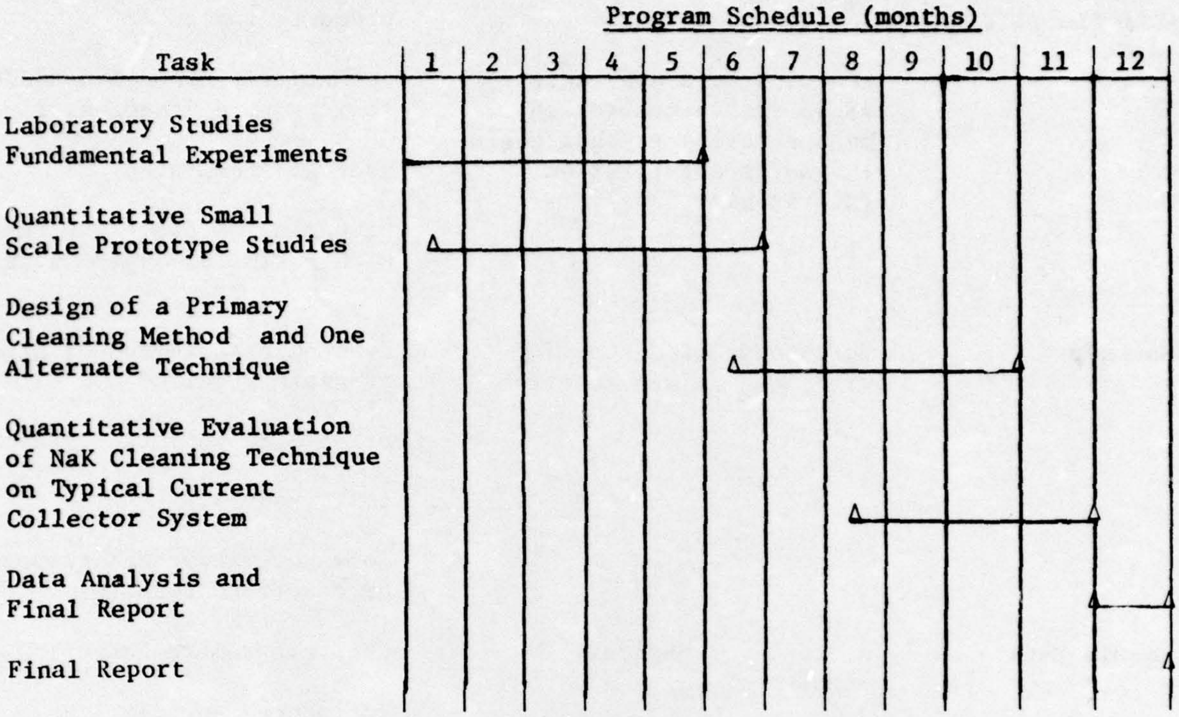

FIGURE 1. Proposed Program Schedule

APPENDIX A - LITERATURE REVIEW

APPENDIX A - LITERATURE REVIEW

As part of this program, a computer search literature review was carried out. A selected set of keywords was employed to search the various data banks, as outlined in attachment A-1. A total of nine separate data banks was searched. The highest yield, in terms of applicable references found, was through the N.T.I.S.* and Chem Abstracts search. The most pertinent and highest percentage were obtained through Engineering Index and Nuclear Science Abstracts.

An example of a typical search strategy for the first five data banks is given in attachment A-2. Note that high yield word descriptions were the combination of "alkali" with "metal(s)" and "liquid" with "metal(s)" or the singular words such as "sodium" and "potassium".

While conducting this review, it quickly became apparent that, depending upon which data bank was being searched, the words "cleaning", "removal", and "decontamination" had different meanings. For example, while searching Engineering Index the word "removal" referred to the physical removal of alkali metals from systems, whereas its use in Chem Abstracts referred to the removal of impurities in alkali metals. Similarly, the word "cleaning" is the proper and currently used term by the nuclear industry to indicate the actual cleaning of liquid metal residues from components, whereas references in Chem Abstracts use the term in conjunction with alkali metal purification. "Decontamination", in almost every instance, referred to the removal of radioactive species from liquid metals and, therfore, should not be used when referring to alkali metal cleaning.

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*National Technical Information Service.

The search strategy for the ARAC* literature survey is given in attachment A-3. A similar search pattern was employed, as noted above. As shown in attachment A-1, a rather low yield resulted, both in references found, and those which were applicable to this study.

An annotated bibliography of the most pertinent references found relating to this program is presented in attachment A-4.

---

*Aerospace Research Application Center.

A-1

Literature Search Summary - NaK Cleaning

	References Found	Applicable
1. N.T.I.S.* - 1964 to date	141	13
2. Chem Abstracts - 1972 to date	152	12
3. Engr Index - 1970 to date	46	5
4. Defense Documentation Center - 1955 to date)	0	0
5. Nuclear Science Abstracts - 1968 to date	31	6
6. Aerospace Research Applications Center (ARAC)	35	2
Scientific and Technical Aerospace Reports - 1962 to date		
International Aerospace Abstracts - 1963 to date		
NASA Tech Briefs - 1963 to date		
TOTAL	405	38

Applicable References = 8.9% yield

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*National Technical Information Service

A-2

Example Search Strategy

N.T.I.S., Chem. Abstracts, Engr. Index, DDC, Nuclear Science Abstracts

Set	Description	Items Found
1	NaK	59
2	Na with K	20
3	Sodium with Potassium	27
4	Alkali with Metal	594
5	Alkali with Metals	478
6	Liquid with Metal	1339
7	Liquid with Metals	983
8	Cleaning	958
9	Recleaning	0
10	Removal	1795
11	Decontamination	672
12	1-7/$^{+}$	3129
13	8-11/$^{+}$	3248
14	12*13	39
15	Sodium	4676
16	Potassium	2762
17	(15+16)*13	121
18	17-14	104

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January 21, 1976

Mr. Robert Witkowski
Westinghouse Research
1310 Beulah Road
Pittsburgh, Pennsylvania 15235

Dear Mr. Witkowski:

RE: NAK REMOVAL FROM EQUIPMENT, a search initiated January 12, 1976.

The enclosed booklet contains the abstracts from an unedited search of the following sources:

1. Scientific and Technical Aerospace Reports, 1962 to date.
2. International Aerospace Abstracts, 1963 to date.
3. NASA Tech Briefs, 1963 to date.

It was necessary to modify somewhat the strategy you gave me on the phone. The NASA dictionary does not include a NAK term. The final strategy used was as follows:

1. LIQUID METALS
2. LIQUID POTASSIUM
3. LIQUID SODIUM
4. SODIUM
5. SODIUM ALLOY
6. POTASSIUM
7. POTASSIUM ALLOY
8. ALKALI METALS
9. CLEANING
10. DECONTAMINATION
11. REMOVAL
12. CHEMICAL CLEANING

The strategy used was: (1+2+3+4+5+6+7+8) . (9+10+11+12)

Page 2
January 21, 1976
Mr. Witkowski

Due to the low number of abstracts retrieved, I did not add the third logic group, COMPONENT or EQUIPMENT.

I am very sorry for the delay in getting these results to you. Our NASA on-line system was down January 15 and 16.

The first seven titles were printed on-line and this is the reason for the different format. The remainder were reproduced from our card file.

Please do not hesitate to contact me if you have questions.

Sincerely,

Shirley Cordes, Ph.D.
Senior Staff Chemist

Enclosures

SC: tla

NaK Cleaning Bibliography

1. Arkhipov, V. M., et al., "Problems of Sodium Removal and Deactivation of Fast Reactor Circuits and Components", AEC-tr-7431, 1973.

   Discusses steam, steam/inert gases and alcohol cleaning methods. Also details methods of using liquid lead to clean and seal defective fuel elements.

2. Borisch, R. R., and Maffei, H. P., "Rate of Sodium Removal from Crevices in FFTF LMFBR Equipment", HEDL-TME-73-32, November, 1973.

   Excellent quantitative evaluation of three methods: distillation, steam-nitrogen and alcohol; steam-nitrogen method selected. Results show that sodium can be reliably and repeatedly removed from annular crevices with diametral widths from 2 to 58 mils, and with depths of six to eight inches. Moisture content ranged from 30 to 100%, 4 to 18 hours of exposure

3. Borisch, R. R., and Maffei, H. P., "Sodium Removal Processes and Material Requalification for LMFBR Hardware", HEDL-SA-661, 1973.

   Additional discussion of moist nitrogen process; some general empirical rates are presented for removing sodium from crevices.

4. Burwell, R. L., "Cleavage by Alkali Metals", Chem. Rev., 54, 672 (1954).

   Resistance of aliphatic ethers to NaK at temperatures below 200°C.

5. C. F. Braun and Co., "Optimization Study D - Sodium Cleaning and Disposal", NTIS PB 208 967, November 17, 1967.

   An abbreviated review of steam lance, steam bath, water, alcohol, anhydrous ammonia, molten metal, hot oil, ultrasonic, and vacuum distillation methods of sodium cleaning.

6. Cordfunke, E. H. P., and Ouweltjes, W., "The Reaction Between CO_2 and Liquid Sodium", J. of Nuclear Materials, 33, pp. 328-332, 1969.

   Liquid Na reacts rapidly with CO_2 at temperatures as low as 175°C; reaction rate dependent on temperature and contact surface. At > 350°C, sodium burns in CO_2.

7. "Enrico Fermi Atomic Power Plant, Unit 1", Report for the First Quarter of 1974, Docket-50016-228, 1974.

   Na and NaK passivation with carbon dioxide.

8. *Handling and Uses of the Alkali Metals*, Advances in Chemistry Series #19. A Collection of Papers from the Symposium on Handling and Uses of the Alkali Metals, 129th Meeting of the ACS, Dallas, Texas, Published May 1957 by the American Chemical Society.

   Good survey of alkali metal technology to late 1950's. Pertinent paper for this work: Bruggeman, W. H., "Recleaning Sodium Heat Transfer Systems". Details relative merits of various techniques for sodium cleaning, such as alcohol, steam and water. Also provides a detailed evaluation of the moist gas procedure; a comprehensive quantitative analysis.

9. James, L. A., and Borisch, R. R., "Exposure of Precracked and Preloaded Fracture Mechanics Specimens in Liquid Sodium with an Intermediate Moist Nitrogen Cleaning", HEDL-TME-75-51, May 1975.

   Analytical experimental study of crack extension in solution-annealed 304 stainless steel as a result of caustic environment. Moist nitrogen cleaning was employed; *no* crack extension resulted.

10. Kilpatrick, M., et al., "Studies of Fast Reactions Which Evolve Gas. The Reaction of NaK alloy with water in the Presence and Absence of Oxygen", J. Phys. Chem., *57*, 4, pp. 385-390, April 16, 1953.

    Delayed reaction of NaK with water (m sec) in presence of air, some work with ethanol.

11. Mole, C. J., "Design and Development of a Segmented Magnet Homopolar Torque Converter", ARPA Contract DAHC-15-72-C-0229, Phase I Report, March, 1973.

    Section 9.3 Homopolar Machine Clean-Up/Operation/Safety details equipment necessary during clean-up of NaK from homopolar machine current collectors. Also, details cleanup sequence, liquid metal loop cleaning and disposal of reaction products. Methods discussed include alcohol, alcohol/water, live steam, and liquid ammonia.

12. Poucher, F. W., and Kohl, A. L., "Cleaning Techniques for Sodium System Components", ANS Transactions, *11*, 1, June 1968.

    Detailed quantitative evaluation of Na removal with steam or steam/nitrogen (1/2 deep layer of liquid Na completely reacted in 60 hours). Also discusses use of 2-butoxyethanol for Na or NaK removal from fuel elements.

13. Pulham, R. J., and Berkey, E., "Laboratory Handling and Purification Practice for the Liquid Metal NaK", Westinghouse Research Report 72-8B6-LMSER-M3, January 4, 1973.

Discussion and numerous references on alkali metal handling; some details on NaK disposal and cleaning of apparatus.

14. Scott, J. M., "Sodium-Heated Steam Generator Model Tests at SGTR, Volume 1", GEAP-10580-1, June 1972.

Alcohol cleaning of superheater and evaporator units with sodium retention in crevices and zero flow areas; 300 gallons of 190-proof ethanol circulated at 2 gpm for 8 hours.

15. "Specialists Meeting of Decontamination of Plant Components from Sodium and Radioactivity", Held at Dounreay Experimental Reactor Establishment, Thurso, Caithness, Scotland, April 9-12, 1973, Published by the U.S. AEC, TID #26487.

Extremely comprehensive compilation of current methods used to remove sodium and NaK from large liquid metal loops and components; international in scope. Pertinent papers for this work include: Paper #10 - Taylor, G. R., et al., "Summary of Sodium Cleaning Experience in the LMFBR Program; Paper #13 - Davies, R. A., "Removal of NaK from Reactor Components"; Paper #18 - Halstead, W.D., "Reaction of Sodium and Water/Steam in Capillaries.

16. The Alkali Metals, Special Publication No. 22, The Chemical Society, London, 1967.

A collection of papers from an international symposium held at Nottingham, July 19-22, 1966. Pertinent papers include: Dransfield, P. B., "Preparation and Handling of Solutions of Sodium in Liquid Ammonia", page 222; Sinclair, V. M.,"Reaction of Carbon Monoxide With Sodium", page 260.

17. Bray, John A., and Donn, Ian M., "Foam Cleansing of Components Contaminated With Sodium", British Patent No. 1,251,842

5 wt % Na lauryl sulfate solution is foamed with nitrogen so that nitrogen-filled foam can be sprayed on alkali contaminated parts. Foam is collected, allowed to collapse into a drain.

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PAS-76-26, November 1976