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EVALUATION OF TECHNIQUES
FOR THE CONTROLLED ADDITION
OF IMPURITIES TO FFTF CLOSED LOOPS

August 1969

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EVALUATION OF TECHNIQUES FOR THE CONTROLLED ADDITION
OF IMPURITIES TO FFTF CLOSED LOOPS

by

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FFTF Project

August 1969

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ABSTRACT

One of the most important variables in the FFTF closed loop testing program will be coolant purity, since variations in impurity levels could significantly alter long term test results. It will probably be necessary, therefore, not only to monitor the impurity levels but to adjust them by controlled concentration changes to meet the needs of various tests.

It is concluded that methods are available for adding known amounts of oxygen and hydrogen to sodium systems. In the case of carbon, it has been demonstrated that addition methods can increase the concentration for some carbon species such as cyanide and carbide. The total carbon content or carburization potential value for a system can be shifted by addition techniques but cannot necessarily be adjusted to a predetermined value.

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J. J. McCown

INTRODUCTION

The Fast Flux Test Facility (FFTF) will be used to study fuel and structural materials in prototypical environments. A majority of such testing will be done in closed loops under controlled experimental conditions.

One of the most important variables in the FFTF closed loop testing program will be coolant purity, since variations in impurity levels during a long term test could significantly alter test results. It will probably be necessary, therefore, to not only monitor the impurity levels but to adjust them by controlled concentration changes to meet the needs of various tests.

Many laboratories throughout the world have been engaged in sodium chemistry studies for a number of years. During the course of this work, several techniques have been developed for adjusting sodium purity levels. The function of this report is to review all of the impurity addition methods thus far developed and to evaluate them for use on FFTF closed loops.

The information for this report was obtained from three sources: published literature, visits to laboratories involved in sodium studies, and telephone communications with personnel at various LMFBR facilities. The emphasis of the survey was to determine the current state-of-the-art in

controlling the concentration in sodium of all chemical species containing carbon, hydrogen, and oxygen, both in pots and pumped loop systems. Any work involving the addition of nitrogen or fission products was also investigated.

SUMMARY AND CONCLUSIONS

Available information concerning methods of adding known amounts of impurities to a sodium system is reviewed in this report. The impurities included in this study were oxygen, carbon, hydrogen, nitrogen, and fission products. ~~Both pot and pumped loop sodium systems were reviewed.~~ The addition methods are discussed in relation to their potential for controlling impurity levels in the closed loops of the Fast Flux Test Facility.

~~It is concluded that~~ Methods are available for adding known amounts of oxygen and hydrogen to sodium systems. In the case of carbon, ~~it has been demonstrated that~~ addition methods can increase the concentration for some carbon species such as cyanide and carbide. The total carbon content or carburization potential value for a system can be shifted by addition techniques but cannot necessarily be adjusted to a predetermined value.

No work has been performed with addition methods on a sodium system the size of an FFTF closed loop (2400-2800 gal of sodium). The bypass sampler technique currently in use at several laboratories might be adaptable to the closed loop requirements but would require major redesign and loop testing using kilogram amounts of impurities. Methods involving additions to a cover gas in contact with the sodium, ~~to be adjusted,~~

although not as yet thoroughly evaluated, appear adaptable for use on closed loop sized systems. Some design studies and large system testing will be required before cover gas addition methods can be recommended.

A review of all the available data on impurity control techniques has clearly indicated the lack of knowledge about problems involved in adjusting more than one impurity species at a time. ~~Considerable evidence that~~ several chemical species interact in sodium and also react singly or together with the containment components, ~~was found.~~
~~It is felt, therefore, that~~ Methods providing any desired concentration of carbon, oxygen, and hydrogen ~~in sodium~~ in the FFTF closed loops will require further development work. Such work must be done in a large volume system ~~in order~~ to gain a better understanding of species and component interactions.

The state-of-the-art concerning nitrogen and fission products control appears to be nonexistent. ~~Tested techniques governing the addition of desired concentrations to sodium.~~ *There are no (or: we did not find)*
~~are completely lacking.~~

DISCUSSION

A primary objective of the FFTF will be the study of various fuel types and reactor component materials under in-pile conditions. Most of this work will involve the use of the FFTF closed loops. Since a closed loop facility will probably be used for many different tests, it may become necessary to vary the purity of the sodium in order to meet specific experimental objectives.

Most experimenters in sodium chemistry agree that the oxygen concentration level in the sodium has a large effect on corrosion behavior, fuel pin cladding attack, fission product plate-out characteristics, and other chemical phenomena. It follows, therefore, that oxygen is one sodium impurity most certainly requiring control and variation between tests. There is evidence that the oxygen concentration in a sodium system varies due to interactions with both carbon and hydrogen. Consequently, the control of carbon and hydrogen concentrations in closed loops may be necessary to control oxygen.

Over the past 15 years, a considerable amount of experience has been developed at various laboratories in operating sodium systems. During the course of this work, experimenters have attempted to vary the impurity levels of several elements in their sodium. In most cases, it was discovered that impurities in sodium can be removed and held at fairly low concentration levels (<10 ppm) by various cold and/or hot trapping techniques. Attempts to increase impurity levels and hold them at a steady state have not been as successful. This difficulty can be partially attributed to chemical interactions occurring in the sodium between carbon, hydrogen, and oxygen, and partially to pickup or release of chemical species by the large metal surface areas present in many of the systems. Therefore, a review of the literature has disclosed the types of chemical compounds previously used as additions to sodium, the methods of addition, and the success or failure in adjusting impurity levels in different types of sodium systems. This report tabulates the techniques tested at other laboratories for adding known amounts of carbon, hydrogen, and oxygen to sodium. It also evaluates these techniques for use on FFTF closed loops.

OXYGEN

Addition Techniques

The influence of oxygen on the chemistry of sodium systems has been studied in some detail at several laboratories. In addition, a number of laboratories have been evaluating United Nuclear Corporation oxygen meters for measuring the oxygen activity in sodium and have made oxygen additions for meter calibration purposes. Consequently, more work has been done on methods for adding known amounts of oxygen to sodium systems than with any other chemical species, and data available on oxygen addition techniques have been tabulated in this report. In all cases, the objective of the addition was to produce an increased concentration of sodium oxide (Na_2O) in the sodium system.

The addition methods summarized in this tabulation range from simply floating Na_2O powder on liquid sodium in a pot to the use of specially designed flowthrough baskets and/or drop valves. The most widely used procedures involve the use of either a "tea bag" or a bypass tube.

Tea bag devices consist of a steel chamber with flow slots which, in some cases are simply slotted steel tubes. This tube, basket, or chamber is introduced into the sodium pot or loop through a port and fastened in place with the tea bag in the liquid sodium. The sodium then flows through the slots and slowly dissolves the Na_2O , Na_2O_2 , or NaOH introduced to increase the oxide level.

A bypass tube arrangement consists of a removable piece of steel tubing which can be connected to a bypass leg on a sodium loop. The removable section is fitted with Swagelok connectors, and a piece of stainless mesh is used to prevent any undissolved material from washing into the flowing sodium stream. A weighed amount of material is placed in the tube on

the upstream side of the mesh and the tube is then connected to the bypass line and valved into the flowing system.

"Buffer" Effect

In those experiments involving small pots of sodium, the calculated and observed oxide concentrations were in good agreement after an addition. On loop experiments, however, the amount of oxide found after an addition was generally lower than that calculated.^(2,3) In some cases,⁽²⁾ four to six times the calculated amount was added before the desired level of concentration was obtained. Most experimenters agree that the pickup and release of oxygen by steel piping, tanks, and other components is dependent upon system temperatures and other variables. This result produces a so-called "buffer" effect whereby large amounts of oxygen must be added to, or removed from, a sodium system in order to produce a small change in the measurable oxide concentration.

Comparison of Methods

As shown in Table 1, several methods for adding oxygen to a sodium system have been successfully employed. The simplest from an operational standpoint (cold trap variations) is, one resulting in the addition of an unknown amount of oxide, unasmuch as the exact relationship between sodium temperature and oxide solubility are not in agreement. Also, cold trap adjustments may reintroduce other impurities such as sodium hydride. The addition of Na_2O pellets using either a bypass sampler or slotted steel tube (tea bag) technique appears to work well and has been used on many systems. The amounts added and the amounts found in some systems disagree due to the "buffer" effect of the steel present. It appears, therefore, that no method guaranteed to adjust a large system to an exact precalculated level of oxide concentration is presently available.

TABLE 1. Oxygen Addition Methods

Type of Material Used	Technique	Comment	Problems
Pot Experiments			
Na ₂ O Powder ⁽⁴⁾	Floated on Na	Unsuccessful	O ₂ went to Na vapor on gas plenum walls
Na ₂ O ₂ ⁽⁴⁾	Floated on Na	Unsuccessful	
Na ₂ O ⁽⁴⁾ pellets	Metal basket	Fair	Dissolution rate unreplicable
Na ₂ O pellets ^(2,5)	Tea Bag*	OK	Buffer effect due to wall interactions
Na ₂ O pellets ^(6,7)	Bypass sampler	OK	Buffer effect due to wall interactions
Na ₂ O pellets ⁽⁸⁾	Not stated	OK	
Na ₂ O ⁽⁹⁾	Cold trap ΔT change	OK	Unknown amount added
Na ₂ O ₂ ⁽³⁾	Bypass sampler	OK	Buffer effect
NaOH ⁽⁷⁾	Bypass sampler	OK	Get NaH in system
NaOH ⁽¹⁰⁾	Drop valve	Fair	Wall-vapor interactions
NaOH ⁽¹¹⁾	Tea bag	OK	Get NaH in system
H ₂ O (argon and ⁽⁴⁾ water vapor)	Gas line over Na	OK	Get some OH in system
O ₂ ⁽⁹⁾	Bulb	Poor	
O ₂ ⁽⁹⁾	Surge tank cover gas	OK	
Na ₂ CO ₃ ⁽¹²⁾	Tea bag, drop valve, bypass sampler	Unsuccessful	No change in oxide concentration

* Slotted steel tube

FFTF Closed Loop Consideration

In studying the application of the various oxygen addition methods for use on FFTF closed loops, certain problems stand out. The 2400-2800 gal of sodium involved is considerably larger than the volume used in any system tested for addition techniques. A change in the closed loop oxide level from 10 ppm up to 30 ppm would require about 600 g of Na_2O . Applying a 5/1 "buffer" effect, expected to be even greater in the closed loop situation, would appear to require as much as 3 kg of Na_2O . In order to use the bypass tube or "tea bag" methods for adding such quantities of material, some hardware design development will be required.

Alternate methods involving the addition of oxygen to the cover gas in a surge tank may prove to be more feasible for large systems such as a closed loop. Further research and development in a large sodium technology loop such as the 2000-gal system planned for BNWL will be required to demonstrate the applicability of cover gas addition methods.

CARBON

Addition Techniques

It is well known that the use of sodium as a heat transfer medium can create many problems because of carbon transport throughout the containment system. Carburization and decarburization can occur between ferritic and austenitic steels, between hot and cold legs, and from metal to metal, i.e., from steel to zirconium. Liquid metal researchers⁽¹³⁾ have observed each transport phenomena in their loops and have attempted to study the mechanism by carbon additions. Over the last several years, improvements in carbon analytical methods have indicated little correlation between the total carbon in a system and the carbon activity as evidenced by the carburization of metal specimens. This observation has led to further work

in attempting to identify the carbon species in sodium responsible for the carbon transport, and to increased attempts to add various carbon containing compounds to sodium in pots and loops. Table 2 summarizes the data available on carbon addition techniques. The apparatus used for carbon additions is generally quite similar to that used for oxygen control. One slightly different method involves flowing sodium through a chamber containing a bed of carbon rods. In this case, the rods do not dissolve completely, and only a small amount of carbon is introduced. The other techniques covered in Table 2 involve tea bags, bypass tubes, etc., as previously discussed under Addition Techniques for oxygen.

Comparison of Methods

The list of methods in Table 2, although not as extensive as the oxygen list, still represents a fairly broad variety of techniques. However, the majority of carbon addition work has been done in connection with solubility studies or carburizing species identification, and was not directed toward increasing concentrations to a known controllable level. A considerable amount of work apparently remains to be done in (1) identifying the carbon species responsible for the observed carburizing reactions, and (2) establishing means for adding the large amount of material required to cause a substantial concentration level change in a large volume system.

The simplest technique, that of contacting carbon rods with flowing sodium, appears to form several carbon species. More than one of these species are apparently involved in carburization reactions. To date, the analytical techniques in use are not sufficiently sophisticated to measure accurately all the species present when such an addition method has been used. Several laboratories^(13,14,15) have evidence that substantial amounts of carbide and cyanide are formed and are responsible for most of the carburization reactions.

TABLE 2. Carbon Addition Methods

	Type of Material Used	Addition Technique	Comment	Problems
Pot Experiments	$\text{Na}_2\text{CO}_3^{(12)}$	Tea bag	No carbon found	Insoluble in Na
	$\text{NaCN}^{(14)}$	Mix with Na	Remains as CN^- soluble to several thousand ppm	None
	$\text{CuC}_2, \text{Mg}_2\text{C}, \text{C}^{(15)}$	Powder in small capsule, Na on top	All had the same effect on C meter	None reported
	$\text{C}_2\text{N}_2^{(4)}$	Added to cover gas	Formed NaCN	None
Loop Experiments	$\text{Na}_2\text{CO}_3^{(15)}$	See above		
	$\text{CO}_2^{(12)}$	Added as gas to surge tank		
	C rods ⁽¹³⁾	Bypass stream through bed of rods	Species formed unknown, some Na_2C_2	None
	C powder ⁽¹⁶⁾	Stainless basket	Particulates introduced	Basket dis- integrated
	C pyrolytic plates ⁽⁴⁾	In bypass section	Used to form C_{64} Na compound	

FFTF Closed Loop Considerations

Sodium coolant purity requirements designated by future closed loop users are expected to include needs for controlling concentrations of carbon species. Varying the total carbon level from <20 ppm to as high as 120 ppm from one test to another may be necessary, according to one review of FFTF test requirements.⁽¹⁾ As additional data concerning the effects of various carbon species becomes available, it appears certain that some impurity requirements will be changed.

Should the prevention of steel decarburization be the only closed loop requirement, the system could possibly be controlled by using a ferritic steel trap placed on line to maintain the carbon activity at an optimum value. However, if certain carbon species must be maintained at known levels, then techniques for adding carbon compounds will be needed. The same considerations previously discussed for the closed loop would then apply, and a considerable amount of additional research and development would be required in a large sodium technology loop to test the applicability of various addition methods.

HYDROGEN

Several reasons for surveying the hydrogen content of a sodium system include (1) the effect of NaH on plugging meter readings, (2) the possibility of hydride formation with refractory metals present in a sodium cooled reactor, and (3) the use of a buildup of hydrogen from a Na-H₂O reaction to monitor leaks at Na-H₂O interfaces. Items 1 and 2 have not been of much interest to date, while Item 3 has been studied mainly on sodium systems containing a sodium-to-water heat exchanger.

Addition Techniques

The only work involving the addition of hydrogen to sodium loops has been performed in connection with in-line instrument

evaluation programs. Sodium hydroxide was used to provide increased Na_2O , and NaH was tested for its effect on a UNC oxygen meter.⁽⁷⁾ Work done at APDA while testing a diffusion type in-line hydrogen meter included bypass sampler additions of hydroxide and hydride, and the addition of H_2 to cover gases. The H_2 detector response was the same for all three methods.⁽¹⁷⁾

The Russians have used a controlled addition of H_2O to a sodium loop cover gas in studying cold traps and plugging meters.⁽¹⁸⁾

The techniques and apparatus for adding hydrogen would be the same as those described for adding oxygen and carbon compounds, as summarized in Tables 1 and 2.

Comparison of Methods

Although little work has been done in the area of hydrogen addition techniques, methods for adding hydrogen to sodium should be quite similar to the ones for oxygen control. Thus, compound additions using a bypass sampler and/or additions to cover gas systems are feasible. One difference, and a possible source of difficulty in hydrogen additions, is its tendency to disappear from a system by diffusion through the piping walls and by interaction with carbon to form methane in the cover gas spaces.

FFTF Closed Loop Considerations

The review of sodium coolant purity requirements for the FFTF closed loop tests includes only one request for hydrogen control.⁽¹⁾ It appears that hydrogen will either be of no interest or held at <2 ppm. Thus, there should be no need for adding hydrogen compounds to closed loop systems.

NITROGEN

Essentially no study of N_2 in Na or of methods for its addition has been performed. Nitrogen solubility in sodium

appears to be extremely low and, although the possibility of the nitride embrittlement of refractory metals in a system has been discussed, little concern has yet been shown. Calcium and barium present in sodium could form complexes, increase the N_2 solubility, and complicate this situation.⁽¹⁸⁾ Since the LAMPRE work demonstrated a N_2 buildup in the cover gas, further investigation of its influence may be required. Nitrogen control in the FFTF closed loop testing programs is not fully defined at the present time; LASL has indicated a preference for controlling nitrogen impurity levels as a condition for studying nonvented carbide fuels.⁽¹⁾

FISSION PRODUCTS

No work has been accomplished to date on the addition of mixed fission products to a sodium loop for the purpose of maintaining a controlled level of concentration. AI has studied the behavior and interaction of trace irradiated salts⁽¹⁹⁾ with loop components, while LASL contacted a piece of LAMPRE fuel with molten sodium⁽²⁰⁾ to study the distribution of the long-lived fission products in a loop. BNW is currently preparing fission products in sodium by a fission recoil technique in order to introduce them into a loop system and study their distribution on loop components.

Any method for adding fission products to a loop to maintain a steady concentration level would require a continuous addition technique since the natural decay and tendency of fission products to plate out in various loop sections would continuously decrease the amount present. Several potential FFTF closed loop users⁽¹⁾ have requested the capability for introducing prescribed amounts of fission products, but it is not known whether they desire to hold them at constant levels. Such a task could prove difficult and would require further investigation following clarification of closed loop test requirements.

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