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Sodium Purity Requirements (A Review and Evaluation)

✓ **Liquid Metal Engineering Center**

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Sodium Purity Requirements (A Review and Evaluation)

by

R.B. Hinze

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Liquid Metal Engineering Center

Operated for

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ABSTRACT

Impurities in sodium coolant used in a Fast Breeder Reactor are identified and their significance discussed. Sodium purity requirements are developed for a coolant system constructed of austenitic-stainless steel and 2-1/4 Cr - 1 Mo ferritic steel, operating at a 1200°F reactor outlet temperature for 30 years. The report concludes that a 2-ppm limit on oxygen concentration is necessary for acceptable corrosion (2 mil) during 3-year life of fuel-element cladding when cladding temperature is 1300°F. Use of a gettering device is indicated to achieve this low oxygen concentration. Recommendations are presented for additional work needed to strengthen the basis for these purity requirements.

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I. INTRODUCTION

The objective of this study is to establish the purity requirements for sodium used in the coolant systems of the Liquid Metal Faster Breeder Reactor (LMFBR). The various impurities of significance are to be identified, and acceptable concentration levels for these impurities defined.

Two requirements have been established for the prototype LMFBR: power level, 2400 Mwt; and reactor outlet temperature, 1200°F. This temperature requirement is tentative; the final outlet temperature may be less than 1200°F. In the preparation of these purity requirements the following assumptions have been made.

- 1) Structural material life, 30 years,
- 2) Fuel cladding life, 1 to 3 years,
- 3) Maximum fuel cladding temperature, 1300°F,
- 4) Candidate materials: 300-series stainless steel; ferritic steels, e.g., 2-1/4 Cr - 1 Mo.

It is the purpose of this study to select impurity concentration levels that will permit the coolant system to meet its operating criteria and service life.

Impurities potentially present in LMFBR sodium, which could contribute to metallurgical changes in structural members and fuel element cladding, are oxygen, carbon, and nitrogen. The first two impurities, along with hydrogen and calcium, can form insoluble compounds which precipitate in the sodium. These precipitates can collect, forming plugs which interfere with normal coolant flow, and can prevent the satisfactory operation of mechanical devices submerged in sodium. Other impurities which may require control are neutron-absorbing elements, miscellaneous impurities in manufactured sodium, fission products, and corrosion products.

These impurities are discussed in Sections III to VII of this report. Results of this study are collected in Section II, Summary and Conclusions. Recommendations for additional work appear in Section VIII.

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II. SUMMARY AND CONCLUSIONS

Any study of sodium purity requirements must be made with reference to a particular system. When this work was initiated, only power level and outlet temperature of the reactor had been fixed. Assumptions of key service conditions (listed in Section I) and the two most likely cladding and structural materials (300-series stainless steel and 2-1/4 Cr - 1 Mo) were made to complete the necessary framework of a reactor system. The system so defined is the basis for this study; results must be considered in the light of this particular system.

Requirements developed herein are applicable to sodium circulating in a coolant system. The complete set is not intended for use as a procurement requirement for sodium, although some of the individual requirements are applicable for procurement use. After developing these general requirements, the next step will be preparation of specific procurement requirements, followed by definition of purification processes, the purification control scheme, and equipment performance requirements.

Purity requirements for sodium used in LMFBR sodium coolant systems (see definition of system above) are summarized in this section. Requirements apply to both primary (core coolant, radioactive) and secondary (non-radioactive) coolant systems unless otherwise noted. For each impurity, or type of impurity, the purity criterion is listed. In some instances, notes on reasons for a criterion and its applicability to a particular system are included. In all cases, bases for the criteria appear in Sections III to VII. Concentration levels noted are the maximum permissible level. Units of concentration are parts per million (ppm) by weight. The requirements are enumerated below.

- 1) Plug forming impurities — saturation temperature 300°F or less.

Reason: Prevention of flow blockage and deposition of solids in submerged mechanisms. (Definitions and usage of the terms saturation and plugging temperature are given in Appendix A.)

- 2) Oxygen — 2 ppm in primary system; saturation temperature 300°F or less in secondary system. Reason for primary system criterion: Acceptable corrosion (weight loss) of 10 to 20 mil fuel-element cladding.

3) Carbon — Carburizing potential of the sodium shall be such that the surface-carbon content of a previously unexposed sample of Type 304 stainless steel shall be equal to or less than 0.5% by weight after at least 100 hr exposure to a sample of the sodium at 1200°F. Basis for criterion: Maximum surface-carbon limit for Type 304 stainless steel used at SRE.⁽¹⁾

4) Nitrogen — 5 ppm. Basis for criterion: Limit established in LMFBR fuel-cladding development program.⁽²⁾

5) Hydrogen — Saturation temperature 300°F or less. Reason: Prevention of plugging. Bases for criterion: Reactor operating experience cited in Section VI-B.

6) Neutron absorbers — Macroscopic cross section for the absorption of thermal neutrons having a mean velocity of 2200 m/sec shall not exceed 0.0146 cm²/g. Notes: A thermal-cross-section test is a convenient test to detect presence of elements in this class of impurities. In addition to the absorption requirement, a compositional limit of 10 ppm lithium has been established. These criteria are applicable to sodium procured for use in both primary and secondary systems. (The latter, because of possibility of leak from secondary to primary systems.) The primary system operating criterion would be based on available excess reactivity of a particular core loading.

7) Miscellaneous impurities (concentrations limits are met by typical manufactured sodium) are shown below.

Impurity	Concentration (ppm)	Impurity	Concentration (ppm)
Aluminum	10	Nickel	10
Barium	10	Phosphorus	20
Calcium	20	Lead	10
Chromium	10	Rubidium	50
Cesium	6	Sulfur	30
Iron	25	Silicon	10
Potassium	200	Tin	10
Magnesium	10	Strontium	10
Halogens	20		

8) Fission products — Limits on fission product concentration cannot be defined at this time because of lack of basic information.

9) Corrosion products — State of the art does not warrant specification of concentration levels.

Two ppm oxygen in core-coolant sodium is the most striking result of this study. Empirical equations developed in the Sodium Mass Transfer program⁽³⁾ were used to calculate corrosion of austenitic stainless steel as a function of oxygen content of sodium. The finding was a 2-ppm level to limit fuel-cladding corrosion to 2 mil in 3 years. This low oxygen level is beyond the capability of cold trapping; some form of gettering, such as zirconium hot-trapping, will be required. Thus, purification economics becomes of increasing importance in the material selection — fuel element life — sodium purity trade-off.

These requirements for sodium purity represent a best set of impurity levels to permit the coolant systems of the LMFBR (as defined above) to meet the requirements and assumptions listed in Section I. This set of impurity levels reflects the current state of the art. Recommendations are presented in Section VIII for those areas of sodium technology where additional information is required to strengthen the basis for these purity requirements. Since pertinent technology is developing, periodic review of these requirements is necessary to keep them abreast of advances in the state of the art.

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III. OXYGEN

A. EFFECTS AND SOURCES

From the time when sodium was first used as a nuclear reactor coolant, oxygen has received attention as a major impurity. Oxygen has three effects in a sodium system. First, the presence of oxygen can result in the formation of sodium oxide deposits which can impede coolant flow and the operation of mechanical devices submerged in sodium. Second, oxygen affects corrosion processes and mass transfer of metallic elements. Third, oxygen influences the solubility and transfer of carbon in sodium, and also reacts with carbon in sodium.

The chief source of the impurity oxygen is air, although water and other oxygen-bearing compounds that enter the system can act as sources. Oxygen can remain in a piping system as a residual gas after the inert-gas purge, and can enter the system by introduction as an impurity in the initial sodium charge. During operation it can enter by means of in-leakage of air through seals or during fuel handling operations. During maintenance or repair work, air is often admitted when portions of the system are open, in spite of the use of cover and purge gases.

B. PLUGGING

Experience with operating reactors and with non-nuclear test facilities containing normal sodium-piping design features has demonstrated that maintenance of a saturation temperature of 300°F or less is adequate to prevent plugging. (Definitions and usage of the terms "saturation" and "plugging temperature" are noted in Appendix A.) An example of such a sodium-piping design feature is the location of the first block valve in a branch line close to the main-stream tee so that the temperature at the block valve exceeds saturation temperature. Another example is the provision of controlled electric heaters on an instrument-connecting pipe to maintain temperature greater than saturation temperature.

C. CORROSION

The most recent, major work on the effect of oxygen on corrosion of structural materials is that of Lockhart and his co-workers.⁽³⁾ They have used statistical analyses of weight changes occurring on test samples exposed in flowing

sodium test loops to develop empirical equations relating weight changes to oxygen concentration and other major variables. They noted that corrosion rates were the same for all three steels tested (316 SS, 2-1/4 Cr, and 5 Cr). This is believed to be due to the fact that selective depletion of chromium and nickel from these alloys changes surface composition to similar iron-rich surfaces, and the steady state corrosion is then controlled by corrosion of the ferritic surface layer remaining. The fact that corrosion rates are the same for the different steels makes their equations particularly useful for a study of the effect of any one variable on corrosion in general.

As a check on the validity of Lockhart's equation for hot-leg corrosion, a corrosion test result reported by Nettley and co-workers in the United Kingdom⁽⁴⁾ was compared with a calculated value using Lockhart's equation. From Figure 3 of Reference 4 one reads a weight loss of 6.3 mg/cm^2 for 18-8-Ti stainless steel in flowing sodium at 600°C , 25 ppm oxygen, and 5.5 m/sec, after an exposure of 28 days. For these conditions, a weight loss of 8.1 mg/cm^2 was calculated using the Lockhart equation, as shown in Appendix B. This good agreement enhances confidence in the use of this equation for predicting corrosion.

Lockhart's corrosion-rate prediction equation⁽³⁾ for removal of material from a hot leg has been applied to a hypothetical fuel element in a LMFBR for the following conditions.

Sodium Velocity	20 ft/sec
Temperature	1300°F
Downstream Factor (for L/D = 0)	1
Exposure Time	3 years
Oxygen Concentration	as noted

The 1300°F temperature is taken as a typical maximum fuel cladding temperature for a reactor outlet temperature of 1200°F. Lockhart's "downstream" factor considers the variation of corrosion rate with concentration of transported element in the sodium stream. The equation was integrated to obtain the total weight loss in 36 months, the assumed life of the element. For convenience in evaluating results of the calculation, the total weight loss was converted to a decrease in cladding wall thickness using an assumption of uniform surface recession and taking the density of the material to be the same as Type 316 SS. A uniform surface recession assumption is acceptable in this case because the

corrosion mechanisms uniformly affected the entire surface of the isothermal-weight-change specimens. The calculation was similar to that shown in Appendix B.

The results, tabulated below, of this calculation of fuel cladding corrosion show the significant effect of oxygen concentration.

Oxygen Concentration (ppm)	Depth of Recession (mil)	
	Average	Maximum
2	0.4	2.0
5	1.1	5.5
10	2.5	12.5

As the authors⁽³⁾ point out, the significant variation of data points around the average must be considered when using their equations for average corrosion rates. The approximate maximum corrosion rates which fall within the 95% confidence levels are five times the average calculated rates. The results of this calculation show that control of oxygen concentration to 2 ppm or less is required for fuel cladding of 10 to 20 mil wall thickness.

These calculated results are supported by the data presented by Nettley (Figure 4 of Reference 4) which show metal loss of 3 mil in 3 years at 1300°F with approximately 10 ppm oxygen. Two data points at an oxygen concentration identified as "less than 5 ppm" indicate a three-fold reduction in corrosion rate between 10 and 5 ppm. The calculated values of fuel-cladding corrosion using Lockhart's equation, supported by Nettley's data, are the basis for the 2 ppm limit on oxygen concentration.

This 2 ppm limit on oxygen concentration in primary (core coolant) sodium is recognized as a distinct departure from previous sodium-purity requirements for austenitic-stainless steel systems. Cold traps permit control of oxygen concentration to or slightly less than 10 ppm; hot traps, or some other gettering method, will be required to reach 2 ppm. A zirconium hot trap purified the 50,000-lb SRE primary sodium system to a saturation temperature lower than the limit of detection of the plugging meter (225°F).⁽⁵⁾ Installation of hot traps to getter sodium will increase plant cost, so purification economics must be considered by the reactor designer in the material selection — fuel element life — sodium

purity requirement trade-off study. Also, on-line instrumentation and a chemical analysis method for the determination of oxygen concentration in the 1 to 10 ppm range must be developed to control purification processes in this low range.

The 2 ppm limit was developed for the core coolant system only. Although not considered in detail in this study, requirements for the secondary system would likely be less stringent. Principal considerations in the development of secondary system requirements will be intermediate heat-exchanger tube corrosion and mass-transfer product deposition. The latter problem is discussed for the primary system (where the problem is more severe because of higher temperature) in the following paragraphs. Pending detailed definition of the secondary system, and subsequent establishment of final oxygen-concentration requirements, the 300°F saturation-temperature limit should suffice as a preliminary criterion. The conservatism of this preliminary criterion has been established by successful reactor and loop experience. A saturation temperature of 300°F is equivalent to an oxygen concentration of 10 ppm, according to Claxton's generalized correlation.⁽⁶⁾

In addition to evaluating material loss from fuel elements in the hot leg, consideration was given to deposition of mass-transfer products in the cold leg. Here, Lockhart's deposition rate equation⁽³⁾ derived from cold leg samples was applied to the entrance of an intermediate heat exchanger. Assumptions for the calculation are tabulated below.

Oxygen Concentration in Sodium	10 ppm
System ΔT	275°F
Duration of Exposure	30 years
Uniform Thickness of Deposit.	

Deposit thickness was calculated for the following temperatures at the deposition site.

Site Temperature (°F)	Deposit Thickness (mil)	Maximum Deposit Thickness (95% confidence level) (mil)
1100	6.55	6.6
1125	7.75	-
1200	12.5	16.5

Deposit thicknesses derived from the approximate maximum corrosion rates which fall within the 95% confidence levels are also tabulated (derived from data of table, page 14, Reference 3). A sample calculation for the 1125°F case is given in Appendix C.

If an intermediate heat exchanger tube 1-in. OD by 0.020-in. wall is assumed, the 12.5-mil deposit at 1200°F would result in a 5% reduction inflow area. While this deposit may not seem too significant, one must be aware that this calculation does not give conservative results. One reason is that the deposition rate equation was developed from weight change data on metal specimens with measured cold-leg weight gains partially caused by semiadherent or loose deposition products. Removal of test samples from the loops, together with the sodium removal and handling required, might have easily resulted in loss of some of these deposits prior to weighing operations.⁽³⁾

Another reason to expect the calculation to give low results is due to specimen location in the test loops. The first specimen holder was located just downstream from the first cooler (see Figure 1, in Reference 3). In high temperature runs, this cooler reduced sodium temperature from 1200 to 1100°F. From results of the deposition calculation presented above, it is apparent that deposition decreases by a factor of two for the 100°F temperature difference between 1200 and 1100°F. Hence, it must be assumed that significant deposition occurred in the first cooler, depleting the concentration of mass-transfer products in the sodium stream prior to the first specimen holder. Results of calculation based on these test loop results do not, therefore, represent the maximum deposition that would occur at the entrance to the intermediate heat exchanger; results of these calculations underestimate the true situation.

Since there is insufficient information to adequately predict the deposition of mass-transfer products in the cold leg, it is not possible to specify an oxygen concentration limit that will assure prevention of excessive deposition. One is drawn to the conclusion of Lockhart and coworkers⁽³⁾ that further experimental data are needed to adequately understand the factors influencing the deposition of mass-transfer products.

D. INFLUENCE ON BEHAVIOR OF CARBON

The effect of oxygen on the solubility of carbon in sodium was documented ten years ago. At that time Gratton⁽⁷⁾ reported the solubility of carbon as a function of temperature at oxygen concentrations of 40 and 260 ppm. Carbon solubility is less at low oxygen levels. For example, at 500°F, the solubility of carbon is 43 ppm at 40 ppm oxygen; 132 ppm at 260 ppm oxygen. A low operating limit on oxygen concentration, such as has been established, should minimize the solution of carbon in sodium.

The literature contains a number of references to the effect of oxygen on the transport of carbon in sodium systems. Nettley⁽⁴⁾ has stated that present data indicate that rates of carburization and decarburization do not appear to be very dependent on oxygen concentration. Nettley's range of oxygen concentration was from less than 5 ppm to about 25 ppm. Anderson⁽⁸⁾ found that oxygen did not affect carbon content of steel at equilibrium conditions, and that carburization reaction rates of Type 304 stainless steel apparently are unaffected by oxygen impurity within the oxygen limits reported. Lockhart^(3,9) reported that no apparent effect of oxygen on the movement of carbon in their test loops was detected. He noted that his tests were possibly not sensitive enough to detect a small change in rate. His tests were conducted at 12 and 50 ppm oxygen.

In sodium with very high oxygen concentrations the decarburization rate of 2-1/4 Cr - 1 Mo steel in a Type 316 stainless steel loop at 1100°F appeared to be higher, according to Andrews and Barker.⁽¹⁰⁾ In 4000 hours 2-1/4 Cr samples lost 65% of their carbon in 300-ppm-oxygen sodium and 45% in 30-ppm-oxygen sodium. They concluded from their rupture test data that a very high (300 ppm) oxygen concentration in sodium might accelerate strength decrease in 2-1/4 Cr - 1 Mo steel. This finding supports their observation of a higher decarburization rate at 300 ppm oxygen. Lyashenko and Nevzorov⁽¹¹⁾ found that the rate of carbon transfer from carbon steels to austenitic steel is in obvious dependence on oxygen content of sodium in the range 50 to 500 ppm oxygen. They reported that this dependence was less obvious at lower oxygen concentrations.

From these reports it is seen that the dependence of carbon transport rate on oxygen concentration is measurable at oxygen levels greater than 30 to 50 ppm. At lower levels the dependence, if it exists, is not discernible. The

low limit on oxygen concentration (2 ppm) which was established on the basis of metallic mass transfer considerations is of the proper magnitude from the standpoint of minimizing carbon transfer.

Chemical reactions of carbon and oxygen in sodium have been reported. Lyashenko and Nevzorov⁽¹¹⁾ concluded that the carbon carrier in sodium was carbon monoxide formed at the surface of steels from carbon and oxygen ions. Carbon-oxygen coupled reactions in alkali metals are noted in Section IV-A.

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IV. CARBON

A. EFFECTS AND SOURCES

The presence of carbon in sodium can lead to a number of adverse effects. For most systems, carburization of materials is the most significant effect. Two types of carburization, grain boundary and volume, have been observed in Type 304 stainless steel exposed to sodium containing carbon.⁽⁸⁾ Grain boundary carburization appears to have little or no effect on mechanical properties. However, the brittle case formed by volume (bulk) carburization can produce two characteristic effects on mechanical properties, depending on the ratio of case depth to section thickness. Relatively thin carburized layers cause weakening of the section when plastically strained. This appears to result from cracking of the case and consequent reduction in cross-sectional area. Yet, under fatigue and stress-rupture testing of smooth and notched specimens, the cracks seem to have no tendency to propagate in the ductile base material, according to Anderson.⁽⁸⁾ Thus, for thin carburized layers, the strength of a member is reduced proportionally to the reduction in effective cross-sectional area resisting load. Carburized specimens with relatively high ratios of case depth to section thickness exhibit higher strength (under steady loads) and lower ductility than uncarburized material. When the case is thick, the carburized material is stronger than the uncarburized section; but, when failure occurs, it can occur at extremely low plastic deformation. When the case depth reaches approximately 10% of section thickness, the failure mode changes from ductile to brittle.⁽¹⁾ Carburization of refractory metals as well as steels can occur in sodium systems.

The opposite effect, decarburization of material, can also occur. This is a problem with ferritic steels, in particular. Decarburization is accompanied by a loss in strength.

Another problem is that of plugging. Deposits of elemental carbon and sodium carbonate, as well as agglomerates of sodium and thermal decomposition products of hydrocarbons,⁽¹²⁾ can cause flow blockages and interfere with the operation of mechanical components submerged in sodium. Related to the plugging problem is the fact that the presence of carbon in a sodium loop can cause plugging temperatures that are different from the oxygen-saturation

temperatures.⁽¹³⁾ Of course this masking of oxygen-saturation temperature can be caused by other impurities whose solubility is temperature-dependent when the saturation temperature for that impurity exceeds the saturation temperature of oxygen.

Several other possible effects have been reported. Experiments conducted at Atomics International have indicated the possibility that carbon in sodium may act as a carrier for nitrogen, with resulting nitriding of structural materials in contact with sodium.⁽¹²⁾ (See also Section V-B.) K. Goldman reported at the Sodium Working Group Information Meeting, Hollywood, California, in April, 1964, that carbon may be significant in oxygen-promoted corrosion. At the same meeting, L. F. Epstein presented evidence of carbon-oxygen coupled reactions in alkali metals, and stated that carbonyls, and mixed sodium-heavy-metal carbonyls may be present in some sodium systems. The significance of coupled reactions or the presence of carbonyls is not known.

Possible sources of carbon in a sodium coolant system are listed below.

- 1) Impurity in initial sodium charge, derived from the graphite electrodes used in the fused-salt-electrolytic manufacturing process, or from organic film on container vessel walls
- 2) Contamination on surface of as-installed piping or components
- 3) Cutting fluids swept in from improperly cleaned gas lines
- 4) Leakage of organic lubricants in pumps
- 5) Leakage of organic service coolant, if such a coolant is used
- 6) Decarburization of pipe or other structural materials, e. g. , ferritic steel
- 7) Graphite in or adjacent to the core

Carbon can be transported in a sodium system between various structural materials. The driving force for carbon transfer is the difference in thermodynamic activity of carbon in the various materials and the sodium in contact with these materials. The activity of carbon varies with the alloy in which it is located (e. g. , Type 304 stainless steel, 2-1/4 Cr - 1 Mo ferritic steel); the phase of the carbon in a particular alloy (e. g. , in solution or separate phase, as chrome carbide); and with temperature.

B. CARBURIZATION OF STAINLESS STEEL

A useful parameter in considerations of carburization in a sodium system is the surface-carbon content of the structural material exposed to the sodium. Surface-carbon content is defined as the concentration of carbon in the structural material at its surface in contact with sodium. The metal-surface-carbon content sometimes is referred to as "carburizing potential."⁽¹⁾ Surface-carbon content has been related to mechanical properties of stainless steel,^(13,1) and to the carbon concentration of sodium in equilibrium with the metal surface.⁽⁸⁾

In the Mechanical Properties Test Program⁽¹³⁾ conducted at MSA Research Corporation the mechanical properties of Type 316 stainless steel in a high-carbon sodium environment at 1200°F were determined. In these tests it was attempted to have sodium saturated with carbon at 1200°F. During the majority of exposure time carbon content of the sodium, by chemical analysis, was in the range of 25 to 40 ppm. The surface carbon content of Type 304 SS samples exposed in this test was 3.8%; Type 316 was 3.5%. A surface carbon content at this level was determined to be too high, due to adverse effects on mechanical properties. Type 316 SS piping developed a heavily carburized case which exhibited cracks; specimens had a severe ductility loss, reduced fatigue life, and significant notch sensitivity, compared to specimens exposed to low-oxygen sodium without carbon.

Sinizer and Pearson⁽¹⁾ used a maximum surface carbon content of 0.5% in Type 304 SS as the safe operating limiting criterion at the SRE in 1962. They stated that, if surface carbon content were kept below 0.5%, the service life, from the standpoint of carburization, was considered to be infinite. Under long-term exposure with a surface carbon content of 0.5%, the steel could approach a uniform carbon content of 0.5%. Even under these extreme conditions, no changes in mechanical properties were expected. The report considered the criterion to be conservative, although direct supporting data were not available.

In an earlier report Anderson⁽⁸⁾ stated that 0.3% surface carbon has negligible effects on mechanical properties of stainless steel.

Studies of the carburization of Type 304 SS in sodium⁽⁸⁾ have provided information necessary to calculate the surface-carbon content of a carburized specimen. With diffusion coefficients derived from experimental work, each of the

following combinations of data can be used for the calculation of surface-carbon content:

- 1) Mean carbon content of the full specimen thickness, time, and temperature of exposure.
- 2) Depth of volume carburization metallographically detectable, time, and temperature of exposure.
- 3) Hardness vs distance from surface, time, and temperature of exposure.

In practice at Atomics International, the surface-carbon content has been calculated from each of the three sets of data, with the result based on chemical analysis of a portion of the tab reported, if confirmed by the other two calculations. In future tests, it is recommended that results from chemical analyses be compared to results from one of the other two methods. If the comparison does not indicate carbon contamination in the chemical analysis procedure, results of chemical analysis should be used. A similar calculational method, based on bulk-carbon concentration in a metal tab, was reported recently by General Electric.⁽⁹⁾

It should be noted that the correctness of diffusion coefficients of carbon in stainless steel developed in Reference 8 has been challenged,^(14,15) since they were determined on the mathematical assumption that the diffusion of carbon through stainless steel was isotropic and followed Fick's second law in its most simple form. The same criticism applies to the coefficients developed in Reference 9. In the analysis presented in these reports, consideration was not given to the fact that carbon, as chromium carbide, for example, precipitates as a second phase. The presence of elements that form carbides has a perturbing effect on carbon gradients and on developed values of diffusion coefficients. Because of the presence of carbon sinks (e. g., chromium atoms) in the steel, carbon concentration at any position in a stainless steel specimen at a given time will be less than it would be in the absence of carbide-forming elements. Hence, the mathematical model used, while applicable for simple diffusion processes, does not accurately describe the complex process of the diffusion of carbon in stainless steel.

Even if the diffusion model is fundamentally incorrect, it appears that the diffusion equations used in the two reports^(8, 9) are suitable for calculating

surface-carbon content of stainless steel specimens exposed at a temperature equal to the temperature in the experiment from which the coefficients were derived. This assertion is supported by the close agreement of experimental and calculated carbon-content curves in Reference 8 and by the examples that demonstrate the validity of the calculational method presented later in this section. However, the use of these diffusion coefficients for predictions under new conditions (i. e., temperatures other than those used in the experiments from which the coefficients were derived) is highly questionable.

To avoid dependence on the use of these diffusion coefficients in the determination of surface-carbon content, thin specimens can be used. Specimen thickness should be such that the specimen will develop a nearly uniform carbon profile under the particular test conditions. Then the bulk-carbon content as determined by chemical analysis can be used directly as the surface-carbon content. Specimens in the thickness range of 5 to 15 mil have been practical as test specimens at Atomics International, although even a tab as thick as 10 mil, after exposure to carbon-bearing sodium for a long time (approximately 200 days) at 1200°F, crumbled during handling.

Anderson in his studies⁽⁸⁾ also developed a functional relationship between surface carbon content of Type 304 SS in equilibrium with a fixed amount of sodium in a sealed capsule and the carbon content of the sodium in the capsule. This relationship is shown on Figure 1, taken from Reference 1.

Discussion of surface-carbon content will be continued later in this section, following remarks on chemical analysis of carbon in sodium.

Sinizer and Pearson⁽¹⁾ analyzed SRE data obtained during the removal of carbon from the core coolant system by hot trapping. The analysis indicated that carbon in the SRE existed partially in a dissolved form, in which it is capable of carburizing stainless steel. Carbon also existed in a second-phase particulate form, in which it apparently is not available for carburizing stainless steel. This latter fact was confirmed after disassembly of primary piping when the effect of direct contact of carbonaceous material on Type 304 SS piping was determined.⁽¹⁶⁾ Metallographic examination of pipe wall directly under a carbonaceous deposit, and of pipe which was essentially free of deposits, did not show any significant difference caused by direct contact of the high-carbon deposit.

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Figure 1. Equilibrium Carbon Concentrations of Sodium and Type 304 SS at 1200°F

It was also shown by Sinizer and Pearson⁽¹⁾ that, even if no second phase carbon were present, with attendant sampling and phase-segregation problems, the accuracy and precision of the chemical technique are insufficient at the present time to predict carburizing potential from a reported total carbon in a sodium sample. From Figure 1 it can be seen that, at low values of carbon in sodium, surface carbon in the stainless steel rises sharply for a small change in carbon concentration in sodium. An increase in surface carbon content from 0.5%, the acceptable criterion used at SRE, and 1.0%, which may result in serious degradation of mechanical properties, is equivalent to an increase of only 3 ppm in carbon concentration in sodium, from 24 to 27 ppm. A review of data from the Second Round Robin of chemical analysis of carbon in sodium⁽¹⁷⁾ shows the mean of probable error of individual analyses to be greater than ± 10 ppm. When an accuracy of $\sim \pm 1$ ppm is required, the state-of-the-art of chemical analyses is clearly inadequate.

The inadequacy of results of chemical analysis for carbon in sodium in predicting carburizing capability of sodium is confirmed by an observation of Lockhart and co-workers.⁽⁹⁾ They reported in 1965 that, despite recent improvements

in chemical analyses methods for carbon in sodium, it continues to be apparent that carburization of Type 316 SS is not related to the carbon content of sodium as determined by chemical analysis. They found no observable difference in carbon impurity levels (by chemical analysis of sodium) for test runs with and without carbon sources (or carbon transfer) in their loops.

In a report published since the Second Round Robin activity, Bradley and Meacham⁽¹⁸⁾ note improved precision from a high-temperature, dry-oxidation procedure for carbon in sodium, ". . . . The proposed method is capable of giving reasonably precise results with the relative error estimated as being in the range ± 5 to 10%" Even if improved analytical techniques were available, no meaningful relation between chemical analysis of carbon in sodium and carburization of stainless steel has been reported to date. This is likely due to the fact that the particular chemical species of carbon causing carburization in sodium have not been identified; specific analyses must await such identification. It is concluded that the accuracy of chemical analyses methods, and current knowledge of the significance of the results of these methods in terms of carburization, are insufficient for prediction of carburization of stainless steel.

As an alternate to sodium chemical analysis methods (for use in the prediction of carburization), metal samples (tabs) can be exposed to sodium, and the surface-carbon content of the tabs determined. Two methods of this type are available. In the first, a sample of sodium can be placed in an inert capsule⁽⁸⁾ with a metal tab, and the sealed capsule held at 1200°F for 100 hours. After exposure, the tabs are examined metallographically, a hardness traverse made, and a portion chemically analyzed, from which the surface carbon content can be calculated. This method has been called, "metallurgical analysis for carbon in sodium." In the second method, tabs are directly exposed in a high-temperature flowing stream of the sodium being evaluated for carburizing capability. After removal of the tab, the same techniques outlined above are used to determine surface carbon content. The latter method is not necessarily indicative of the total carbon content of the entire system, but of the carbon currently available for carburization. In the capsule technique, second-phase carbon present in the sodium dissolves during the 100-hr exposure at 1200°F (until the high-temperature sodium becomes saturated) and is available for

carburizing the sample tab. Hence, the result of the capsule test includes carburization effects of, not only the carbon in solution at the time of sampling, i. e., the carbon available for carburization at the time of sampling, but also particulate carbon potentially available for carburizing structural materials in the coolant system.

Indications of the reproducibility of surface-carbon content determinations appear in Reference 1. The following data are from Table 3 of this reference for a hot-trap inlet pipe sample after 1600 hr exposure.

Surface-Carbon Content (wt %)

<u>From Hardness Traverse</u>	<u>From Metallographic Examination</u>
0.72	0.60
0.72	0.71
0.59	0.65

Other data give additional confidence in the reproducibility of the method. The surface-carbon content of a second pipe sample⁽¹⁾ was 0.72%, as determined from microhardness data, after 800-hr exposure in the hot-trap inlet line. The carburizing potential of this sodium stream was decreasing during the period, according to tab analysis. The surface-carbon content of the final tab exposed adjacent to this pipe sample was 0.65%. At the time the final tab was inserted, the pipe sample presumably had a surface-carbon content of 2.5%,⁽¹⁾ and could not follow the decreasing carburizing potential as closely as the final, raw-stock tab. It is therefore to be expected that the pipe sample would have a higher surface-carbon content than the tab. From these two examples of SRE data, the reproducibility of the method appears very good.

Lockhart also had an opportunity to check the validity of surface-carbon calculations.⁽⁹⁾ Terminal examination of a heater sheath by incremental machining and analysis of the turnings for carbon yielded a surface carbon content of 0.35%. Low-velocity sample tabs from the same loop showed an average surface-carbon content of 0.30%. This was considered very good agreement, since the heater was located upstream of the samples, and one would therefore expect a slightly higher surface content.

Since inadequacy of chemical analyses of sodium to determine the carburizing capability of sodium has been noted, and the determination of surface-carbon content of metal specimens has been demonstrated to be a practical and effective measure of the carburizing capability, it is concluded that surface-carbon content determination should be used as the primary measure of carburizing potential or capability of sodium in a LMFBR.

Additional experimental work is required relating mechanical properties to surface-carbon content of stainless steel and other candidate structural and cladding materials. This work is needed to verify the value of 0.5% established by Sinizer and Pearson⁽¹⁾ as the maximum surface-carbon limit for Type 304 SS and to establish limiting criteria for other materials. Pending this verification, 0.5% should be considered as the state-of-the-art criterion for an 18-8 stainless steel system.

C. DECARBURIZATION OF STAINLESS STEEL

Brief consideration has been given to the establishment of a minimum carburizing potential for sodium to prevent decarburization of structural materials. The minimum level could be set -- in terms of surface-carbon content of a metal specimen -- at or slightly below the as-manufactured or bulk carbon content of the major material, or of a critical structural material in a particular sodium system. However, while such a standard may be necessary at a future date, establishment of minimum carburizing potential at the present time is considered premature.

Typical reactor-grade sodium contains some carbon. Concentrations of 13, 30, and 34 ppm carbon in batches of commercially available sodium, and values in the range 4 to 14 ppm from samples of a shipment of ten tank cars received at Hallam have been noted.⁽¹⁹⁾ As Figure 1 illustrates, sodium containing less than 15 ppm carbon would decarburize most 18-8 stainless steel, and some of the samples noted above contained less than 15 ppm. While a potential problem is recognized, it is not proven that a serious problem exists, when the accuracy and precision of chemical analysis for carbon in sodium⁽¹⁷⁾ are considered. It is recommended that establishment of a minimum carburizing potential for sodium be deferred until a later date when more and better data are available.

D. CARBON TRANSFER IN BIMETALLIC SYSTEM

Maximum surface carbon concentrations measured in the Sodium Mass Transfer bimetallic loop⁽⁹⁾ are only slightly smaller than the value of 0.5%. From the test results in the bimetallic loop, it is therefore concluded that carbon transfer must be considered and minimized in the design of any sodium-austenitic stainless steel-ferritic steel reactor system. Lockhart and his coworkers suggested this minimization of carbon transfer might be accomplished by stabilizing the carbon in the ferritic steel or through trapping of the carbon from the sodium stream prior to its entry into the stainless steel portions of the system. Where such a choice must be made, it is strongly recommended that reactor designs utilize carbon-stabilized ferritic steels, rather than purification of sodium between the cold and hot legs. Trap throughput for such purification would have to be large to adequately protect the hot leg materials, and the hot trap location would be unattractive from the standpoint of energy conservation. This location for trapping equipment conceivably could prevent carburization of fuel elements, but would not prevent decarburization of the ferritic steel.

E. OTHER EFFECTS

Lockhart and coworkers have reported⁽²⁰⁾ that sodium carbonate appears to be reduced fairly rapidly at 1000°F, based on their observation of a sharp increase in plugging temperature in less than one hour after carbonate addition. The plugging temperature increase was assumed to be sodium oxide formed during reduction of the carbonate. They also noted that the products of this reduction reaction (e.g., carbon) do not act as a "ready carburizing agent." Cold trapping reduced the plugging temperature after carbonate addition, presumably by trapping the sodium oxide produced. (It was reported that the cold trap also removed some elemental carbon.) From this report one concludes that, in 1000°F sodium, a sodium-carbonate-impurity problem rapidly becomes separate sodium oxide and carbon problems. At lower temperatures the reduction reaction rate would likely be lower, but the end results similar. From the standpoint of system plugging, a plugging temperature limit (normal operations) of 300°F provides a suitable limit for sodium carbonate and its reduction product, sodium oxide.

In regard to the problem of plugging caused by carbonaceous material, reference is made to the discussion of a limit to prevent carburization. In the metallurgical-analysis-for-carbon-in-sodium method previously described (capsule technique) all carbon in the sodium sample is made available for carburization during the 1200°F capsule exposure. A Type 304 SS surface-carbon-content limit of 0.5% on results of the metallurgical-analysis method, applied to a representative sodium sample, should be a satisfactory limit on carbon concentration with respect to system plugging. For this purpose the metallurgical-analysis method is recommended rather than the method of exposure of metal tabs in a high-temperature branch stream of the system. This recommendation follows from the discussion of the two surface-carbon-content methods in Section IV-B.

V. NITROGEN

A. EFFECTS AND SOURCES

Nitrogen is of significance as an impurity because it can cause nitriding of structural materials in the system, and can induce a susceptibility to cracking when exposed to relatively weak corrosive environments, e. g., moist air.⁽¹²⁾ No effect from the latter would be expected while a component was in the coolant system. During or following steam cleaning of a stainless steel component, such as a fuel element, corrosion could occur providing, of course, that nitrogen had diffused into the material.

Nitrogen can gain entrance into a sodium system as an impurity in the cover gas, by means of air inleakage, by admission of air to the system during repair work, or possibly by diffusion through austenitic piping walls at 1100 to 1200°F.⁽²¹⁾

B. NITRIDING

In early work, Brush and Rodd⁽²²⁾ found that Type 347 SS is readily nitrified in a 900 to 1100°F sodium system having a nitrogen cover gas. However, Batutis et al.,⁽²³⁾ reported that 18-8 stainless steel was not subject to gross nitriding attack in an experiment with 1100°F sodium and a nitrogen cover gas. (Incidentally both of these investigators found that beryllium developed a nitrified surface layer which was brittle, not very adherent, and tended to flake off.) In more recent work, Gill and Bokros⁽²⁴⁾ found that Type 304 SS is nitrified in a 1000°F pumped sodium system with a nitrogen cover gas. The degree of nitriding is heaviest at the interface between the sodium and nitrogen. Nitride case thickness observed after 1400-hr exposure is noted below.

<u>On Sodium Side of Interface</u>	<u>Case Thickness (mil)</u>
Slightly below surface	2.0
3 in. below surface	0.3
18 in. below surface	very thin
<u>On Nitrogen Side of Interface</u>	
3 in. above surface	0.4

Bokros also nitrided mechanical-property specimens by commercial techniques using ammonia gas. One series of specimens was prepared with a 1 to 2 mil case; the other with a 4 to 6 mil case. With these samples they found that nitriding results in an increase in the yield strength and flexural fatigue life, and a marked decrease in the ultimate strength and ductility at temperatures up to 1000°F. At 1000°F, elongation (%) decreased from 32.8 (as-received steel) to 18.3 (with 1 to 2 mil case), and to 6.5 (with 4 to 6 mil case). Stress-rupture testing determined that the nitrided material has a much shorter life at a given stress than the mill-annealed Type 304 SS when measured at 1000°F. At a stress of 40,000 psi the life of the as-received specimens is more than fifty times greater than the specimens with a 1 to 2 mil case. These data establish the detrimental effects of nitriding on stainless steel.

Nitrogen transport modes in a sodium system are of interest. In Reference 24, it was noted that with specimens extending through the sodium free surface, the nitride case was thickest just below the sodium surface, and thickness decreased with increasing distance from the surface. This is similar to Thorley's observation⁽²⁵⁾ that nitriding of niobium occurred only when the niobium extended through the surface of a quiet bath into the nitrogen atmosphere, suggesting that nitrogen migration is due to surface, rather than to bulk liquid, diffusion. These observations of nitriding concentrated near the sodium-free surface likely were made in quite clean systems. From the previous history of the loop used by Bokros, one would expect the sodium to have very low concentrations of calcium and carbon. Introduction of either of these impurities would be expected to change the pattern of very localized nitriding and to cause nitriding throughout a sodium system with a nitrogen cover gas, as discussed in the following three paragraphs.

Results of an investigation of the influence of carbon or calcium impurities in sodium on nitriding of stainless steel, including undocumented results of an early test at Atomics International, noted in Reference 12, are summarized: So far as is known, the solubility of nitrogen in sodium is so small that it cannot be measured. Therefore, only negligible quantities of nitrogen could be retained in the sodium by the mechanism of solubility. However, nitrogen could be retained in the sodium by either of two other mechanisms: (1) reaction with any calcium impurity present to form calcium nitride and (2) formation of calcium or sodium cyanide (or cyanate) in carbon-bearing sodium. The calcium

nitride or sodium cyanide then could be carried throughout the primary cooling circuit by liquid sodium and could come into contact with various metal surfaces of stainless steel and zirconium. Nitriding could occur at a temperature-dependent rate and to a depth in the metal walls dependent on time. In the laboratory test, nitrogen was bubbled through low-temperature (~300 to 400°F) sodium containing carbon. After passing nitrogen through the sodium, the system was purged with helium to remove any remaining nitrogen cover gas. After reheating to 1200°F, it was found that nitriding then occurred to the same extent as when a nitrogen cover gas was present. This indicates that significant quantities of nitrogen were tied up by calcium impurity and/or carbon during the bubbling operation, and this source provided a means of nitriding during subsequent reheating to the higher temperature. This test demonstrated that one or both impurities combine with nitrogen in some manner and make the nitrogen available for nitriding structural materials throughout the system.

Support of the hypothesis that the presence of carbon in sodium enhances the transport of nitrogen in sodium is furnished by data from Hayes and Anderson.⁽²⁶⁾ They nitrified Type 304 SS in sodium containing carbon in a system using nitrogen as a cover gas. Exposure of 115 hr at 1200°F produced a 0.001 in. case; significant embrittlement was observed. Nitrogen content at the steel surface was reported to be 0.62%, but Anderson, because of the thickness of the sample used to determine surface-nitrogen content, considered the actual surface nitrogen concentration to be about twice this value.

The fact that calcium has a positive influence on the transport of nitrogen in a sodium system has been reported (see Section VII-A, Calcium). From the information described above it is concluded that calcium and carbon act as carriers for nitrogen, making the nitrogen available for nitriding stainless steel throughout a sodium system.

Nitriding of other materials in sodium systems has been reported. Zirconium and niobium nitriding was reported by Weeks,⁽²⁵⁾ and refractory metals nitriding by Goldmann at the Sodium Working Group Information Meeting, Hollywood, California, in April 1964.

The foregoing discussion has established that nitrogen is a significant impurity in sodium, and that the presence of nitrogen should be minimized in a sodium

system. However, there is insufficient information available to fix a maximum concentration level. A limit of 5 ppm nitrogen was recently established as typical of a fast reactor environment⁽²⁾ in an LMFBR development program. This is considered a conservative value from the standpoint of metallurgical change, and should be used until additional information is available.

VI. HYDROGEN

A. EFFECTS AND SOURCES

Hydrogen is an impurity of significance in some fast reactors because it can cause low temperature embrittlement of transition and refractory metals. However, in the absence of these metals in LMFBR, embrittlement is not considered a problem, since austenitic stainless steels are "notably insensitive to hydrogen embrittlement under ordinary conditions."⁽²⁷⁾ The presence of hydrogen can also cause sodium hydride plugs, which could impede coolant flow and the operation of mechanical devices in the same manner as sodium oxide plugs. The presence of hydrogen can cause plugging temperatures different from the oxygen saturation temperature.

Sources of hydrogen are, water vapor in air, liquid water, impure cover gas, and decomposition of organic compounds which may enter the sodium. It has also been postulated that hydrogen formed on the water side of steam generators diffuses through tube walls and enters the secondary sodium.⁽²⁸⁾

B. PLUGGING

Experience at SRE indicates that a saturation temperature of 300°F is sufficiently low to prevent plugging by sodium hydride. After the tetralin leak in 1959, it was estimated that from 0.7 to 7 lb of hydrogen was released (by decomposition of tetralin) to core-coolant sodium; it was believed conceivable that most of this hydrogen reacted with sodium to form sodium hydride.⁽¹²⁾ If all this hydrogen reacted with sodium, the concentration of sodium hydride would have been 0.03 to 0.3 wt %, assuming uniform mixing with the 50,000 lb of sodium. Even the lower concentration exceeds the reported⁽¹²⁾ saturation concentration of 0.01% hydrogen at 510°F. While some of the hydrogen was probably released to the cover gas before reactor outlet temperature was decreased below 750°F,⁽²⁹⁾ it is probable that hydrogen concentration was sufficient to cause saturation when sodium temperatures in the system were reduced to the 300 to 400°F range. While some plugging did occur in fuel-coolant channels, these plugs were characterized as being carbonaceous. No sodium hydride plugging was observed in the piping, even though plugging temperatures did occasionally exceed 300°F.

Operating experience at the NaK-cooled SNAP Experimental Reactor and SNAP 8 Experimental Reactor, where the fuel provided a source of hydrogen, supports SRE experience. Although plugging-temperature histories have not been published, it is known that, when plugging temperatures were maintained near 300°F, sodium-hydride plugging was not observed in the coolant systems.

This reactor operating experience at Atomics International is the basis for designation of a maximum saturation temperature of 300°F to prevent sodium-hydride plugging.

VII. OTHER IMPURITIES

A. CALCIUM

As described by L. F. Epstein at the Sodium Working Group Information Meeting, Hollywood, California, in April 1964, calcium will reduce sodium oxide, and the calcium-oxide reaction product can form precipitation plugs in the system. Calcium will also act as a carrier or transport agent for nitrogen, and will transfer nitrogen from the gas-sodium interface throughout the system, where the nitrogen is available to nitride structural materials. It also forms intermetallic compounds with certain elements, e.g., zirconium and niobium. The brittle compound formed with nickel is CaNi_5 .

Calcium is present in manufactured sodium as a result of calcium chloride additions to reduce the melting point of molten sodium chloride in the electrolytic cell. The removal of calcium to low levels (10 to 20 ppm) represents the principal difference between commercial and "reactor grade" sodium.

A maximum calcium concentration of 20 ppm in the delivered sodium should be acceptable. It is expected that this level will be further reduced by heating sodium in the charge tank to react any free calcium present with oxygen, followed by removal of the calcium oxide by cold trapping, settling, or filtering. Only 8 ppm oxygen is required to form a stoichiometric mixture with 20 ppm calcium.

B. NEUTRON ABSORBERS

The concentration of elements having a significant neutron-absorption probability should be limited for neutron-economy reasons. Sodium currently available commercially contains only a few impurities of this class, e.g., lithium, boron, that are of significance in a fast reactor. Nevertheless, limits on this class of impurity should be established to provide for possible changes in sodium raw material sources of the manufacturers or proprietary manufacturing processes which conceivably could introduce other nuclear poisons of this class into the delivered product. To meet this requirement, two methods have been used: (1) identifying limits on neutron-absorbing impurity content, and (2) specifying an appropriate value of a parameter which is a direct indication of the nuclear significance of the poisons present.

If the first method is followed, each high cross-section impurity must be identified. However, specification of a definite concentration limit for each impurity is not considered appropriate. A specific limit on each impurity would likely be overly restrictive, since the relative abundance of the poisons can vary from batch to batch. Total neutron-capture probability is the important parameter. Therefore, the specification must state a requirement on total capture. This can be done by limiting the increase in the cross section due to all impurities to that corresponding to a given concentration of one impurity, e. g., 20 ppm boron. In its application, this method involves chemical analysis of sodium for each neutron poison. From the results of chemical analyses, the increase in neutron absorption cross section of the sample must be calculated, and compared with the increase due to the specified limit on boron (or other "standard impurity"). Several disadvantages in this method are apparent:

- 1) Each impurity must be identified
- 2) Many of these chemical analyses are difficult and expensive
- 3) It is doubtful if some of the analyses have the required sensitivity
- 4) Calculations converting concentration to cross section are somewhat involved.

Because of its disadvantages, this method is not recommended.

In the second method, one can merely specify the maximum acceptable macroscopic cross section of the sodium in a neutron flux of appropriate energy. The measurement can be made in a reactor or critical facility. From reactivity measurements, the cross section can be calculated. This method is direct; a significant parameter, reactivity, is measured, from which the cross section is calculated. The second method has been selected because of its obvious advantages.

Consideration was given to both a test at thermal neutron energy and one with an energy spectrum typical of a fast breeder reactor. Though the latter test could be performed in a critical facility — and obviously would be the best test of poisons of concern in a fast reactor — the thermal energy test was chosen because of its relative convenience; there are a number of suitable facilities in which a test could be performed. Poisons significant in a fast reactor would be

detected in a thermal energy test; in fact the test would be more rigorous than actually required since most poisons have larger cross sections for lower energy neutrons. However, this rigorous test should not result in additional sodium cost, because commercially available sodium is sufficiently free of nuclear poisons to meet the requirements of thermal reactors.

The cross section criterion is based on successful SRE experience. The SRE sodium procured to the specification⁽³⁰⁾ that, "The increase in the cross section for the absorption of thermal neutrons due to impurities in the sodium shall not exceed that corresponding to the presence of 20 ppm boron," performed satisfactorily. A macroscopic cross section of 1.06 times the cross section for sodium alone ($0.0138 \text{ cm}^2/\text{g}$), equal to $0.0146 \text{ cm}^2/\text{g}$, represents the thermal neutron capture probability of sodium containing 20 ppm boron. This is the same value used in the HNPF sodium procurement specification;⁽³¹⁾ again, the sodium showed satisfactory nuclear characteristics.

One supplement to this thermal cross section criterion is necessary. In the absence of other poisons, the $0.0146 \text{ cm}^2/\text{g}$ criterion would permit lithium concentrations up to 135 ppm. At a neutron energy of 250 kev, Li^6 has a cross section (n, α reaction) of 3 barns. At the same energy, sodium has an absorption cross section of 0.7 millibarns. To prevent this resonance-absorption peak of lithium from controlling neutron absorption in the sodium, a lithium-absorption limit of 1% of the absorption in sodium at this energy is necessary. This additional criterion can be met by limiting the concentration of lithium to 10 ppm.

C. IMPURITY ACTIVATION

Provision for limiting concentration of elements whose neutron-activation products pose potential accessibility problems has been a feature of sodium procurement specifications. The need for such a provision is questioned. Predictions for HNPF⁽³²⁾ showed the following gamma activity, assuming that the sodium just met the activation requirement of the specification,⁽³¹⁾ based on 20 years operation followed by 25 days decay after reactor shutdown. The predictions are tabulated below in $\mu\text{c/g}$.

Activated Corrosion Products	15
Fission Products	10
Activated Impurities	10
Na^{22}	0.3

More than 95% of the activity in a HNPF sodium sample analyzed in February 1967 was attributed to Na^{22} activity,⁽³³⁾ which indicates that the sodium more than met the activation requirement, and was, in fact, free of these impurities. In reactors, such as SRE, that have experienced some fuel damage, activity due to fission products completely controls accessibility for maintenance in pipe galleries. From experience with commercially available sodium in reactors, and qualitative consideration of (1) anticipated ruptured fuel-cladding operating criterion, (2) possible use of open fuel elements, (3) expected fuel-cladding corrosion-product deposition in piping, and (4) Na^{22} production, activity contributions from neutron-activation products of original sodium impurities do not warrant a limitation on concentration of these impurities in a sodium purity specification.

D. CHEMICAL COMPOSITION

Significant impurities, the maximum concentration limits for impurities with concentration defined in terms of weight fraction in the coolant, and the basis for these limits are listed in Table 1. In those cases where a limiting concentration value has not been developed in this report, a reference is given for the existing specification which was the source of the limit. With the exception of oxygen, the concentration values listed are directly applicable to sodium procurement. Typical concentrations in an operating system generally would be lower than listed, even though engineered removal processes are not employed for these elements (oxygen excepted). Certain elements with concentration defined in terms other than weight fraction have also been included in Table 1.

Elements thermodynamically capable of reducing sodium oxide are of concern because any oxide precipitates formed might impede sodium flow and the operation of submerged mechanisms. The oxides of many elements other than those noted have negative free energies of formation of greater magnitude than sodium oxide. Only those elements which are expected to be present in commercial sodium in more than trace amounts have been listed.

E. FISSION PRODUCTS AND CORROSION PRODUCTS

Development of sodium technology for the LMFBR program includes consideration of open (vented) fuel elements. This leads into an essentially new area

of purity requirements and purification process for sodium-cooled reactors. While some consideration has been given to maintenance-accessibility requirements in the event of minor cladding defects, little, if any, consideration has been given to the effect of gross fission product contamination in an operating sodium-cooled reactor. Limits on fission product concentration cannot be defined at this time because of lack of basic information.

The low limit of 2 ppm on oxygen concentration was established to minimize mass transfer and therefore the concentration of corrosion products in the sodium stream. Again in this case, the state of the art does not warrant specification of concentration levels of corrosion products.

TABLE 1
BASIS FOR CHEMICAL COMPOSITION LIMITS

Element	Maximum Concentration (ppm)	Remarks					Reference for Concentration Limit
		High Concentration Observed(a)	Reducing Agent(b)	Alkali Metal(c)	Concentration Limited by Cross-Section Test(d)	Other	
Aluminum	10	x	x				34
Barium	10		x				34
Boron	—	x	x		x		
Cadmium	—				x		
Calcium	20	x	x			See text, Calcium	
Carbon	—					Concentration limited by saturation temperature and carburizing potential requirements.	
Cesium	6			x			30
Chromium	10	x	x				34
Cobalt	—				x		
Halogens	20					Possible corrosive agent	34
Hydrogen	—					Concentration limited by saturation temperature requirement.	
Iron	25	x					34
Lead	10	x					34
Lithium	10		x	x		See text, Neutron Absorbers	
Manganese	—	x	x		x		
Magnesium	10	x	x				2
Nitrogen	5					See text, Nitrogen	2
Nickel	10	x					34
Oxygen	2					See text, Oxygen	
Phosphorus	20	x	x				30
Potassium	200	x		x		Decreases thermal conductivity.	34
Rubidium	50			x			34
Silicon	10	x	x				34
Strontium	10		x				30
Sulphur	30	x					30
Tin	10						34

(a) High concentration has been reported in commercial sodium.

(b) Thermodynamically capable of reducing Na_2O at 1200°F.

(c) Other alkali metals which may be present due to chemical similarity.

(d) Composition limit not set. Concentration will be limited by thermal neutron absorption test.

1

2

3

4

VIII. RECOMMENDATIONS

On-line instrumentation and a chemical analysis method for the determination of oxygen concentration in the 1 to 10 ppm range should be developed.

Experimental mass transfer investigations should be extended to low oxygen concentrations (1 to 2 ppm) to verify the validity of extrapolating Lockhart's results⁽³⁾ to this range, and to determine if any unsuspected metallurgical changes occur. Additional experimental work is required to adequately define all the factors influencing the deposition of mass-transfer products in the cold leg of sodium systems. A goal of this work should be a method for predicting material balances for mass-transfer processes in a sodium system.

Additional experimental work is required relating surface-carbon content of stainless steel and other candidate structural and cladding materials to mechanical properties. This work is needed to verify the value of 0.5% established by Sinizer and Pearson⁽¹⁾ as the maximum surface-carbon limit for Type 304 SS and to establish limiting criteria for other materials. Limits for materials that may be decarburized, as well as those that may be carburized, should be determined. This effort should be conducted in parallel with the development of chemical methods for analyzing carbon in sodium. Development of chemical analyses methods will likely take a considerable period of time. The work recommended can be accomplished expeditiously. Results are needed to permit full utilization of the surface-carbon-content method for analysis of carbon in sodium in existing and planned LMFBR test facilities while chemical analyses methods are being developed.

When better methods for chemical analysis of carbon in sodium are available, the relation between surface-carbon content of metal and equilibrium concentration of carbon in sodium should be redetermined. Emphasis should be placed on (1) accurately defining the species and quantity of carbon causing carburization in sodium, and (2) evaluating the diffusion of carbon in stainless steel using a model which considers chemical reactions, i.e., carbide formation. This relationship, with results of work recommended above, should give significance to the results of chemical analysis for carbon in sodium in terms of material behavior.

If ferritic steels are to be used in sodium systems, metallurgical techniques for carbon stabilization should be evaluated carefully. These techniques have definite advantages compared to carbon-transfer control techniques involving removal of carbon from the sodium stream by hot trapping.

Experimental data should be developed to provide a basis for establishing an acceptable maximum nitrogen concentration in sodium.

The development of much basic information on fission products in sodium is needed prior to the use of open fuel elements. Methods for determining the concentration of fission products in radioactive sodium; metallurgical, nuclear, and other effects of long-term operation of a reactor with large concentrations of fission products; and fission product removal methods are examples of the information required.

Methods for determining the concentration of corrosion products in sodium, and relations between corrosion-product concentration and mass-transfer rates, should be developed, if possible, so that the extent of mass transfer can be predicted from analyses of sodium samples.

APPENDIX A

SATURATION TEMPERATURE - DEFINITION AND USAGE

A brief review of definitions concerning saturated solutions may be helpful. A solution can be defined as a homogeneous mixture of two or more substances. A saturated solution is a solution for which a state of equilibrium exists with undissolved solute. As an example, in a saturated solution of the solute oxygen in the solvent sodium, some oxygen must be present as a separate phase. In the sodium-oxygen system, the second, or separate, phase is sodium oxide, present as solid particles. Because the solubility of oxygen in sodium has a positive temperature dependence, the amount of oxygen that can be held in solution increases with increasing temperature. The solubilities of several other sodium impurities (e.g., hydrogen, carbon) have a similar temperature dependence.

For a given concentration of each of these impurities in solution there is a finite temperature above which the solution is less than saturated (all of the impurity present is in solution and the mixture of the impurity and sodium is homogeneous). At or below this temperature the solution is saturated (some of the impurity exists as a second phase and the mixture is nonhomogeneous). This temperature is defined as the saturation temperature, which is usually referred to as "plugging temperature."

Conversely, when the temperature dependence of solubility is known,⁽⁶⁾ a given saturation temperature serves to define impurity concentration. This is the basis for converting a system plugging temperature to a concentration level of a certain impurity. Of course, this conversion presupposes knowledge of the identity of a particular impurity in the sodium.

When a saturation temperature limit is used without reference to a particular impurity, as in Section II, a concentration limit on all impurities whose solubility is temperature-dependent and on insoluble impurities is implied. To meet this criterion, concentrations of solubility-temperature-dependent impurities must be so low, or particle size of these and insoluble impurities so small, that no flow impedance is detectable on a plugging meter at the specified saturation temperature.

APPENDIX B

HOT LEG CORROSION CALCULATION

The objective of the calculation is to compare Nettley's data⁽⁴⁾ with a calculated value from Lockhart's equation.⁽³⁾ From Nettley's Figure 3, a cumulative loss for 18/8/Ti of 6.3 mg/cm² is read after 28 weeks exposure at conditions tabulated below.

Temperature	600°C	1112°F
Oxygen Concentration	25 ppm O ₂	
Velocity	5.5 m/sec	18 fps
Time	28 weeks	6.54 months

It is assumed that Nettley had a negligible downstream factor, L/D_i .

The equation from Reference 3 for hot leg removal will be used.

$$R = V^{0.884} O_x^{1.156} \exp \left[12.845 - \frac{23,827}{T+460} - 0.00676 \left(\frac{L}{D_i} \right) + \frac{2.26}{t+1} \right]$$

where

R = steady state rate of removal of metal (mg/dm²/mo)

O_x = oxide level in sodium, ppm oxygen

V = sodium velocity (ft/sec)

T = temperature at corrosion site (°F)

L/D_i = downstream factor (site location in number of hydraulic diameters downstream from beginning of isothermal section)

t = exposure time in months

Let W be cumulative weight loss,

then

$$W = \int_0^t R(t) dt$$

Evaluating,

$$W = (18^{0.884})(25^{1.156}) \exp \left(12.845 - \frac{23827}{1112 + 460} - 0 \right) \int_0^{6.54} e^{\frac{2.26}{t+1}} dt$$

$$W = (12.9) (41.3) (0.101) \int_0^{6.54} e^{\frac{2.26}{t+1}} dt$$

On Figure 2, herein, the area under the curve $e^{2.26/t+1}$ vs t was integrated graphically. From the plot, the value of the integral was observed to be 15. So,

$$W = (12.9) (41.3) (0.101) (15) = 807 \frac{\text{mg}}{\text{dm}^2} \frac{\text{dm}^2}{100 \text{ cm}^2} = 8.07 \frac{\text{mg}}{\text{cm}^2}$$

Therefore, Lockhart's equation predicts a corrosion loss 29% greater than shown by Nettley's data.

Figure 2. Hot Leg Corrosion Response

APPENDIX C

COLD LEG DEPOSITION CALCULATION

The formula from Reference 3 for cold leg deposition will be used:

$$R = O_x^{0.94} \exp \left\{ \left[22.61 - 0.038 \Delta T \right] - \left[\frac{31,980 - 55.24 \Delta T}{T + 460} \right] - 0.0032 \left(\frac{L}{D_i} \right) + \frac{0.92}{t + 1} \right\}$$

where

R = steady state deposition rate (mg/dm²/mo)

ΔT = system ΔT (°F)

O_x = oxide level in sodium (ppm oxygen)

T = temperature at deposition site (°F)

L/D_i = downstream factor (site location in number of hydraulic diameters downstream from beginning of isothermal section)

t = exposure time in months

Assumptions for this calculation:

Oxygen concentration in sodium = 10 ppm

System ΔT = 1125 - 850 = 275°F (Reference 35)

Temperature at entrance of IHX = 1125°F

Time = 30 years = 360 months

Deposition site is entrance to IHX, so $L = 0$ in downstream factor

Let W be cumulative deposition, then

$$W = \int_0^t R(t) dt$$

Evaluating:

$$W = 10^{0.94} \exp \left\{ \left[22.61 - 0.038 (275) \right] - \left[\frac{31,980 - 55.24 (275)}{1125 + 460} \right] - 0 \right\}$$

$$W = 8.7 \exp \left\{ 12.16 - 10.592 \int_0^{360} e^{\frac{0.92}{t+1}} dt \right\}$$

$$W = 41.72 \int_0^{360} e^{\frac{0.92}{t+1}} dt$$

The area under the curve $e^{0.92/t+1}$ vs t on Figure 3 was integrated graphically. The integral curve was observed to be essentially linear for $t > 10$; the equation for the linear portion is

$$\int_0^t e^{\frac{0.92}{t+1}} dt = 1.047 t + 2.4 \quad ,$$

which has the value 379.3 at $t = 360$ month.

Substituting,

$$W = (41.72) (379.3) = \frac{1.58 \times 10^4 \text{ mg}}{\text{dm}^2}$$

Further assumptions:

- (1) deposit has a uniform depth, d
- (2) density of deposit, ρ , is same as density of Type 316 SS,
 $131.6 \times 10^3 \text{ mg/in.}^3$

Then

$$d = \frac{W}{\rho} = \left(\frac{1.58 \times 10^4 \text{ mg}}{\text{dm}^2} \right) \left(\frac{\text{in.}^3}{131.6 \times 10^3 \text{ mg}} \right) \left(\frac{6.45 \times 10^{-2} \text{ dm}^2}{\text{in.}^2} \right) = 7.75 \times 10^{-3} \text{ in.}$$

$$= 7.75 \text{ mils.}$$

Figure 3. Cold Leg Deposition Response

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