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SODIUM TECHNOLOGY QUARTERLY REPORT

APRIL, MAY, JUNE 1970

August 1970

AEC RESEARCH &
DEVELOPMENT REPORT

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SODIUM TECHNOLOGY QUARTERLY REPORT
APRIL, MAY, JUNE 1970

By

The Staff of
Sodium Chemistry Subdepartment
W. R. Wykoff, Editor

August 1970

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ABSTRACT

This report, BNWL-1200-4, Sodium Technology Quarterly Report April, May, and June 1970, was prepared and published by the staff of the Sodium Chemistry Subdepartment of Battelle-Northwest. The objectives of the program are the development, prooftesting, and documentation of methods to characterize and control the coolant quality on FFTF according to established standards.

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1.0 SUMMARYSODIUM SYSTEMS DEVELOPMENT

Conceptual design work on the Sodium Purity In-Line Analytical Module (SPIAM) was completed and the design description transmitted to RDT. SPIAM will provide capability at PNL for application tests of in-line instruments developed at ANL and elsewhere. SPIAM is based on a design of the in-line instrument system to fit within the FFTF in-line instrument cell.

Detailed cleaning procedures based on RDT standards are being prepared to clean stainless steel during construction of sodium systems. Quality control tests have been developed to verify cleanliness of cleaned parts.

An analysis was completed of the uncertainties in the calculation of FFTF piping dose rates due to deposited radioactive corrosion products. This analysis indicated an uncertainty exists of about one order of magnitude; dose rates will be 0.007 R/hr to 3.6 R/hr.

Examination of six bellows sealed valves which had been cleaned and reinserted into 500 °F sodium for 400 hr indicated all had cracks in the bellows, probably due to high oxygen content of the sodium. A second set run under clean sodium conditions at 1000 °F had no cracks.

An analysis of the potential strength loss in the FFTF heat transport system due to carbon removal from the stainless steel during service concluded that decarburization will not be a problem in the HTS, and the ASME design properties for Type 304 SS be used without further reduction.

SODIUM SYSTEMS ENGINEERING

The Quality Assurance Plan for the Prototype Applications Loop has been submitted to RDT and tentative approval received. The loop will be designed to ANSI Code B 31.7 Class II, and the vessel to ASME Code Section III, Class C.

The existing Small Components Evaluation Loop is being modified to accommodate several developmental in-line instrument packages. This will permit preliminary development work on these items to proceed before the PAL loop is available.

Two scale model FFTF cold traps have been fabricated implementing the cleaning procedures of RDT Standard F5-1. The job was quite successful.

The Sodium Technology Program has responsibility for providing design and hardware for the cover gas, sodium purification, and sodium purity monitoring systems for the Fast Reactor Thermal Engineering Facility. The conceptual design for these systems has been completed and is shown in Figure 3.6.

A small task force has been implemented to initiate activities to make small valves conforming to RDT Standards available through the valve industry.

A thermal analysis of the diffusion cold traps being considered for use on the FFTF closed loop systems has been completed and a summary of the results is included in the body of this report.

SYSTEMS CHEMISTRY

The Systems Chemistry Section's primary objectives are to provide proven equipment and techniques for the analyses of sodium and cover gas from the FFTF, to provide analytical support for on-going PNL sodium technology and FFTF programs,

and to perform development work on a fuel failure locations system for the FFTF. Major task areas and their status at the completion of the report period are summarized below.

High Radiation Level Analytical Facility Design

The ability to obtain and maintain an adequate inert atmosphere (less than 1 ppm water and oxygen) in a remotely operated cell has been demonstrated through mockup operation. Oxygen determinations in sodium by the amalgamation procedure have been performed remotely to verify the atmosphere control. Results comparable to those obtained in the glove box facility have been obtained. Sufficient information has been obtained to predict with a high degree of certainty that FFTF sodium can be taken from the reactor and analyzed immediately by the laboratory techniques in common use today if the proper equipment is available.

Remote Sampler Development

Remote operation of the removable section bypass loop sodium sampler has continued. A method has been demonstrated for remote crimp-sealing the sample tubes while the sodium is molten. Samples sealed by this technique have been transported in air to the analytical facilities without contaminating the sodium. Samples taken with the sampler have been demonstrated to be uniform in oxygen concentration along their length at the 5, 24, and 28 ppm concentration levels.

Sodium Studies Facility Analytical Support

The installation of all major equipment and services for the laboratory to analyze alpha contaminated sodium has been completed. A program to standardize methods for the analyses of sodium has been initiated. A procedure for the determination of trace metals in sodium by vacuum distillation followed by atomic absorption analysis has been set up and evaluated.

Copper, magnesium, lithium, calcium and the primary stainless steel metals can be determined at the 0.1 ppm level. Evaluation of three modifications to the amalgamation method for oxygen in sodium is underway.

FFTF Analytical Support Facilities

Numerous samples of the aqueous insoluble particulates from CGEL have been examined metallographically. X-ray diffraction, metallographic, and electron microscope examinations have been made on in situ particulates collected on filters from the CGEL loop. The carbon species procedure development is nearing completion. Careful handling techniques have solved the filter contamination problem. Efforts are being concentrated on obtaining good carbonate recoveries.

Cover Gas Systems

Development of an in-plenum hydrogen meter based on the diffusion of hydrogen through a nickel membrane into an argon carrier stream followed by thermal conductivity measurement of the diffused hydrogen has been completed. The device has adequate stability and a section limit of 10 ppm in the cover gas. Modification of the device has begun to apply the approach developed at ANL based on diffusion into a vacuum.

Modifications to CGEL have been virtually completed to permit the engineering evaluation of the new controlled cooling profile sodium vapor trap design. Traps have been constructed and installed and a program plan completed. An analytical study of the FFTF cover gas system sampling flow rate has been completed and some flow requirements established.

Fuel Failure Monitoring System

A work plan outlining the FY 1970 and FY 1971 development effort for Task FS-B (Fuel Failure Location Subsystem) has been completed covering five location concepts.

Preliminary gas tag ratios were selected and an analysis was made of anticipated costs of tagging 80 subassemblies of 217 pins each. Four methods of selecting tags were considered. Tag gas costs ranged from \$4500 to \$26,000. An analytical study of entrainment in the FFTF reactor vessel was started. Hydraulic feasibility testing of the in-vessel gas separation concept and the conductivity probe concept was completed. Data obtained with the mockups show that both concepts hold promise as fuel failure location devices for FFTF. Preliminary static sodium tests of the fission product deposition concept have been completed. Cesium will deposit on the selected tab materials. Initial tests of the acoustical triangulation concept have been completed on the Hydraulic Core Mockup. The concept appears capable of locating gas releases as small as 10 vppm within a zone of about nine subassemblies. Further feasibility testing is scheduled during the next quarter.

OXYGEN-SENSING SOLID ELECTROLYTES

Improved tubes for the UNC oxygen meter have been produced by ZIRCOA and are now available for field testing in the oxygen meters.

Sodium compatibility testing of improved Zircoa tubes shows that, while some are compatible with clean sodium, none will withstand oxygen-saturated sodium. Apparent carbide formation limits firing in graphite furnaces to about 1900 °C, and the consequences of exceeding this temperature are seen. An etch test which may serve as a sensitive indicator of grain-boundary second phases is discussed.

The electrical conductivity of a 7.5 wt% Y_2O_3 -92.5 wt% ThO_2 solid electrolyte measured in and out of a ^{60}Co gamma field in air, argon, and helium indicates that gas conductance is a problem in the gamma field.

2.0 SODIUM SYSTEMS DEVELOPMENT

INTRODUCTION

D. W. Shannon

The objectives of the Sodium Systems Development Section is to develop methods to characterize the sodium purity of FFTF and FFTF test facilities and to provide design assistance to FFTF in the areas of sodium technology.

IN-LINE SODIUM PURITY MONITORS

The objectives of this task are threefold:

1. Definition and development of in-line instrument system concepts which are compatible with FFTF configurations.
2. Design, fabrication, and operation of test systems to be installed in the PNL Sodium Studies Facility (335 Building) to permit engineering application tests of in-line instruments (developed at ANL and elsewhere) in the proposed FFTF configuration.
3. Fabrication of a prototype FFTF in-line instrument system for reactor test at EBR-II.

Task objectives also include providing the sodium technology assistance for the selection, design, and installation of in-line monitors on FFTF facilities including the Small Components Evaluation Loop (SCEL), Prototype Applications Loop (PAL), Fast Reactor Thermal Engineering Facility (FRTEF), and the High Temperature Sodium Facility of the Composite Reactor Components Test Activity (HTSF/CRCTA).

Sodium Purity In-Line Analytical Module (SPIAM)

W. H. Caplinger and V. P. Kelly

SPIAM represents the test system conceived to achieve Objective 2 listed above. A simplified engineering flow diagram and conceptual arrangement was given in the previous quarterly report (BNWL-1200-3). Functional requirements for SPIAM include:

- Provide an experimental test loop for engineering application tests of in-line instruments. This loop will be connected to a source of sodium supply in the 335 Building and will provide test stations for the five basic in-line instruments: oxygen meters, hydrogen meters, carbon meters, plugging meters, and vacuum distillation samplers.
- Provide space for tests of techniques for on-line calibration of monitoring instruments.
- Provide additional loop space for addition of advanced or alternate concepts of in-line monitoring instruments and bypass samplers.
- Provide a basic system consisting of driver loop and instrument test sections arranged so that the geometry is compatible with the geometry of the FFTF sodium purity monitoring cell and will permit design verification testing and engineering development of the in-line instrument system within simulated constraints of the FFTF operational and maintenance system.

The block diagram for SPIAM is shown in Figure 2.1, for reference. Basically, SPIAM consists of a 1/2-in. pipe pumped-flow sodium distribution loop arranged in the conceptual FFTF configuration to permit operation of up to 10 test sections connected in parallel across the supply and return legs of the loop. SPIAM design is to be compatible with the configuration of the proposed Prototype Applications Loop, which will serve as the source of sodium supply.

The general arrangement of a typical test section is shown in Figure 2.2. Piping, flowmeter, throttling valve if required, heating or cooling components, and the sensors or samplers will be packaged in an 11-in. wide by 30-in. high by 33-in. long box to meet spatial requirements of the FFTF sodium purity in-line analytical cell. Special space requirements

H-3-29943

FIGURE 2.1 Sodium Purity Inline Analytical Module (Schematic)

SK-3-15599

FIGURE 2.2. Sodium Purity Inline Analytical Module Test Section Isometric

developed during detail design for individual test sections may change the shape of the test section box. Sodium enters the test section from the supply header below it, passes through a throttling valve (if required) and flowmeter, and exits to the return header above after passing through the test sensor or sampler and its related heat exchange components.

The preliminary design description of SPIAM has been issued and preparation of the quality assurance plan is underway. The SPIAM will be designed and fabricated as a Class II nuclear piping system in accordance with ANSI Code for Nuclear Piping B31.7-1969. RDT Standards will be applied to SPIAM, where possible, consistent with the requirement that SPIAM be operable during the third quarter, FY-71. It is planned to apply RDT Standards fully to the Prototype FFTF On-Line Instrument System. Test sections will be designed and fabricated as nonstandard sodium systems because of the experimental nature of key components.

FFTF In-Line Instrumentation Package

W. H. Caplinger and V. P. Kelly

Design concepts for the FFTF in-line sodium purity monitoring cell must be formulated to provide bases for SPIAM design and early identification of reactor application problems, as the first step in meeting Objective 3 of the In-Line Purity Monitoring task. One concept for the FFTF cell is represented in Figure 2.3, based on the following criteria and requirements:

- Instruments for up to six sampling points are to be installed in the cell.
- Instruments for each sampling point consist of the five basic instruments: carbon meter, oxygen meter, hydrogen meter, plugging meter, and vacuum distillation sampler, plus an in-line calibrator unit if required.

- Instruments must be contact maintained and operated and therefore must be designed for minimum radiation exposure to personnel.
- Individual instruments should be packaged to permit removal of individual packages from the cell with minimum overall disruption to in-line sodium purity monitoring. Maintenance or modifications to the instrument package would be done outside the cell.
- The instrument or instruments in each package should be connected to permit rapid removal of any one instrument without removing the entire package itself.
- The concept should provide sufficient flexibility to permit addition of radiation shielding around individual packages.
- The arrangement should provide for "horizontal" installation of in-line packages and "front face" access to the instruments and service functions, to permit removing individual packages without disturbing adjacent packages and to make possible the use of through-the-wall mechanical operators (e.g., throttling valve drive shafts).

The conceptual arrangement shown in Figure 2.3 provides for mounting each in-line instrument in an 11-in. wide, 30-in. high, and 33-in. long package, with a 10-in. deep pipeway between each row of packages for sodium piping and electrical and instrument services. Design will be detailed sufficiently to verify feasibility of the concept and compatibility with current FFTF design efforts.

Plugging Meter Selection

G. E. Meadows

A review of the in-line instrument needs for FFTF and related facilities has indicated the need for selection of a "standard" plugging meter and application of the meter to all

FFTF test facilities in order to ensure a uniform approach for using a plugging meter as an instrument for measurement of sodium purity. A design review of plugging meters used in other installations narrowed the selection of two basic designs:

(1) the plugging meter under recent development at LASL which incorporates an orifice plate as the plugging restriction and (2) the EBR-II type plugging meter which has a modified valve with a fluted stem as the plugging restriction.

The two plugging meter designs will be evaluated on the SCEL with an objective to select a "standard" meter and establish a uniform operating procedure.

Minor design changes have been completed. The economizer cooler sections which precede the plugging restrictions have been sized to allow for cooling 1200 °F inlet sodium to about 250 °F at the plugging restriction at a 1/4 gal/min sodium flowrate. Controls for the two meters have been selected for two modes of operation: (1) plugging temperatures obtained by cool down with a bare restriction and (2) plugging temperature obtained by oscillating on a partially plugged restriction.

Thermal Shock Test of Oxygen Meter Ceramic Tubes

W. H. Caplinger and V. P. Kelly

The objectives of this task are to determine the thermal shock rate which causes failure of the ceramic in the standard UNC oxygen meter geometry and to compare this shock rate with that for an improved geometry electrolyte.

The experimental apparatus for the thermal shock test was selected and designed, and was discussed in the last report.(1) The equipment was fabricated, and installed on the sodium heat transfer loop (SHTL) during this quarter. In the first test a ceramic tube was installed in the standard oxygen meter housing and the piping filled with sodium. However, a leak was discovered and traced to the Grayloc fittings on either side of

the test parts which were not properly assembled. The problem is being corrected and a new test run scheduled on the SHTL.

The experimental plan calls for first thermal shock testing ceramic tubes prepared by the original process used in the first generation oxygen meters. Tubes produced by an improved method developed at PNL and the tube supplier will then be tested. The technique to determine the failure point will involve ramping the temperature from 300 to 600 °F and increasing rates (i.e., shorter times) until the ceramic breaks.

CHEMICAL CHARACTERIZATION OF SODIUM SYSTEMS

The objective of this activity is to document the relationship between: (1) the condition (chemical contamination) of an assembled large sodium loop system, and (2) the identity and quantities of impurities which appear in the sodium during fill and startup of the system. These results are to guide the design of large sodium systems by describing the balance between expensive precleaning and assembly procedures on one hand and intolerably high sodium contamination on the other. These data are also required to determine the fraction of the installed cold trap capacity that will be consumed during startup of a sodium reactor or loop. Little information appears in the literature on this relationship. The problem is divided into three areas: (1) the specification of cleaning procedures to be used, (2) the examination of treated (cleaned) surfaces so as to specify their condition, and (3) the analysis of the sodium and cover gas as the system is filled and brought up to operating temperature. Studies this quarter have been in the general areas of testing of cleaning procedures, inspection techniques and functional basis for cleanliness criteria. They are directed toward the preparation of a Detailed Cleaning Procedure to be used for the construction of the Prototype Applications Loop (PAL).

Materials and Equipment Testing

A. P. Bohringer and W. H. Yunker

A 500 W, 20 Hz ultrasonic transducer in a 5 gal stainless steel tank has been put in service for cleaning small parts. Tests are in progress to identify satisfactory cleaning agents and conditions of time and temperature.

A 5000 psi, 10 gal/min water pump has been put in service for spray cleaning tubes and pipes (inside and out) and large vessels. The safety and operating procedures have been written and auxiliary equipment assembled. This includes the fabrication of an 8-ft steam heated chemical soak tank for pipes. Testing will proceed after the delivery of a special spray lance for cleaning the inside of 1/2 in. schedule 40 pipe.

Over a dozen chemical cleaning agents have been ordered (some already in use) for testing with various types of cleaning. They will be checked for chemical purity (to meet RDT Standards) and for satisfactory service. Local water supplies and other reagents are also being checked for compliance with RDT Standards. The chemical analyses for chloride in organic materials and cleaning materials suggested in the RDT Standards are found to be inadequate in some cases. Work is underway to establish methods for appropriate chloride analysis.

Cleanliness Verification Tests

W. H. Yunker

A number of qualitative tests are available for checking a cleaned surface to determine whether a surface is satisfactorily cleaned. Quantitative tests are lacking. Considering the economic incentives (or penalties) involved in comprehensive, clean construction programs, it is felt that some effort is necessary to provide quantitative tests so that procedures used can be better related to system requirements. Two techniques being tested at this time, while not perfected, illustrate what can be done.

Oil Residues. Four locally used machine cutting oils were examined by infrared spectral analysis and a calibration curve of concentration versus peak height was made for the C-H bond stretching frequency: four oils matched on one curve. A cleaned stainless steel sample was oiled and then wiped with clean white cloth until no further staining of the wipe was visible. Solvent rinse of the piece and IR analysis showed it to have 0.6 mg oil/in.² or one drop of oil on 9 in.² of surface. A second oiled sample was cleaned until its observation under 3600 Å ultraviolet light (black light) was inconclusive as to the presence of oil. The solvent rinse of this piece showed 0.1 mg oil/in.² of oil on the sample. The method was used on two shop test pieces. The results are given below in that section. The test, at present, has a sensitivity of 0.1 mg oil/cm³ of solvent. It is felt that this can be reduced by a factor of 5 by laboratory and instrumental refinements.

An analysis of system carburization effects is underway to establish the amount of oil which would be serious if left in the as-built system.

Particulate Residue Tests. It was reported by R. Borisch that the replicate technique, used with electron microscopy, had been observed to remove small particles from a metal surface. A few tests verified this and a technique was devised to do this and to view the particles under a low power metallograph (100-200X). Particulates of 1 micron are easily seen. Direct viewing of the metal surface fails to reveal such particles, because of the lack of contrast between particles and the metal background. Several examples of particles in replicas can be seen in Figures 2.4-2.7. Chemical analysis of the replicate tape showed it to contain less than 35 ppm chloride. Further work will be designed to obtain qualitative and quantitative analysis of the particulate residues collected.

Neg 470-2416

150X

FIGURE 2.4. From Pipe Picked at Random from Pipe Yard. Treatment: washed with acetone and wiped several times with acetone soaked cloth until no smear appeared on cloth.

Neg 470-2415

150X

FIGURE 2.5. From Well Cleaned Surface. Treatment: 5 min in warm detergent in ultrasonic cleaner.

Neg 470-2413

150X

FIGURE 2.6. From Metal Surface After Being Thoroughly Cleaned and Then Handled in Pipe Shop Under "Controlled Environment"

Neg 470-2414

150X

FIGURE 2.7. From Surface Sandblasted by Vendor, After Heated by 36 hr Acetone Soak, 15 min Mild HNO_3 -HF Etch and Followed by a 30 min Treatment in Warm Detergent in an Ultrasonic Cleaner

Cleaning Field Tests

R. V. Bowersock and W. H. Yunker

The components of five small stainless steel shop fabrication jobs were cleaned in the laboratory with trial cleaning methods. This was done to become more aware of actual field cleaning problems and to try out some of our ideas using "real" dirt. A construction procedure was written for the two fabrication shops involved, to control the shop environment and work practices and also to uncover some of the problems which arise under these working conditions. These procedures are considered to be intermediate toward meeting requirements specified in the RDT Standards. Spot tests for cleanliness are being made on selected pieces both after final cleaning and at selected times during construction. Tests are primarily for particulate residues by replica and oil residues by acetone rinse, as described above.

Results and conclusions to date are:

- The need was emphasized for testing (for ease of removal and for halide content) all compounds, markers, or other material to be used on the stainless steel both by the vendor and the fabrication shop.
- A sandblasted finish on stainless steel makes chemical cleaning much more difficult, if not impossible, and assures a high retention of contaminants picked up during final construction phases. Sandblasting should not be allowed. This can easily be seen from the replica shown in Figure 2.7.
- Neither of the two ultrasonic cleaning baths used performed in a completely satisfactory manner. One excelled in oil removal (by water break test), the other did better with particulate removal. More work is needed in this area.

- Oil picked up on two, 1-1/2 in. pipe sections during fabrication, was about 0.06 mg/in.² (probably below the threshold of the "black light" test).

SODIUM PROPERTIES

W. H. Yunker

The first complete draft has been finished for a document containing values of the physical and thermophysical properties of sodium recommended for use on the FFTF Project. Twenty properties, including PVT tables for sodium vapor, have been selected from current primary review sources. The information has been checked for accuracy, converted to engineering units, and presented in analytical and graphical form.

RADIOACTIVITY CONTROL

W. F. Brehm

The purpose of this task is to provide the means for estimating the radiation fields in FFTF from deposited corrosion and fission products. The corrosion products arise from the removal from the core of irradiated fuel cladding by the coolant; the fission products arise in the event of fuel failure in the core and transport of the fission products out of the core by the coolant. In the past three Quarterly Reports, (1,2,3) an extensive review of the theoretical, programmatic, and experimental effort of work on this topic was given. Some of the fundamental conclusions are these:

- Radioactivity sources from activated corrosion products (⁶⁰Co, ⁵⁸Co, ⁵⁴Mn) and fission products which are volatile or daughters of short-lived gas decay chains (¹³⁷Cs, ¹³¹I, ¹⁴⁰Ba, ¹⁴⁴Ce) will be present in primary sodium piping systems if fuel "leakers" occur. Radiation levels arising from these sources affect design decisions on plant arrangement, shielding, and maintenance philosophy.

- Experimental investigations of the transport of corrosion products in sodium-stainless steel systems showed that ^{54}Mn was very mobile, ^{51}Cr less so, and ^{60}Co and ^{58}Co even less mobile. The hot and cold legs of the test loop contained more deposited radioactivity than the heat exchanger. A strong tendency of ^{54}Mn to deposit in the cold trap was noted. These results indicate that the potential trouble spots in the heat transport system are the hot and cold legs (especially the hot leg FFTF pumps) and the cold trap.
- There is still almost no information on release and transport of any fission products except ^{137}Cs (4,5) with which to assess the maintenance implications.

Analysis of Calculational Uncertainties

In the last quarterly report (Reference 3), estimates of corrosion product activity in the FFTF were given. During the past quarter we have been examining the effect of the uncertainties in the accuracy of the various parameters in the calculations on the estimated FFTF dose rates.

The radiation level at a distance 2 ft from a pipe or component can be written for isotope i at time t :

$$L\left(\frac{R}{\text{hr}}\right) = \frac{S_i R}{\lambda_i} Z(t) f_i K_i, \quad (1)$$

where

S = average saturation activity of isotope i in corroding material, Ci/cm^3

R = total corrosion rate, cm^3/day

λ_i = decay constant, $(\text{days})^{-1}$

$Z(t) = (1 + \lambda_i t) \exp(-\lambda_i t)$; accounts for simultaneous in-core buildup and out-of-core decay

f_i = fraction of total inventory of isotope i deposited in component of interest

K_i = conversion factor from curies deposited in component of interest to radiation field in R/hr, for isotope i .

With this starting point, one can examine each term and try to assess the possible uncertainties in each.

This equation assumed (1) no decrease in S from burnout of target nuclides in the core, (2) no transfer of radioactivity from the core by isotope exchange over and above the amount transferred by gross material loss (corrosion). The first assumption is probably reasonable; the second one is probably not. The result of additional isotopic exchange from core to piping would be to increase R . The amount of increase is not known. This is essentially the method of calculation used in BNWL-808⁽⁶⁾ and outlined on page 37 of Reference 2.

The parameter S is a function of activation cross section and number of target nuclides in the core. Uncertainty in S may arise from uncertainty in activation cross sections and difficulty in obtaining an "average" flux for all regions of the core. In our calculation for the direct calculation of radiation levels that appeared in Reference 3, we used $S = 13 \text{ Ci/cm}^3$ of cladding for ^{58}Co , 4.1 Ci/cm^3 for ^{54}Mn , 0.6 Ci/cm^3 for ^{60}Co .⁽⁸⁾ These values are probably accurate to within a factor of 2.

The term R is a product of the corrosion rate (per unit area per unit time) with the total area. Uncertainty is possible here also. The corrosion rates are not really well known, especially in the 1-5 ppm oxygen-in-sodium ranges where FFTF will probably be operating. The "transient" in the corrosion rate has an unknown effect, as Mn is known to be preferentially leached, and Co to be retained. The total area of corroding material, and the method of obtaining an "average" corrosion rate, are certainly not well defined.

A value for R was obtained from the General Electric corrosion equation⁽⁸⁾ and corrected for preferential leaching by multiplying by 3 for manganese corrosion and by 0.5 for cobalt corrosion. At 1 ppm oxygen, average cladding temperature of 1000 °F, and 26 ft/sec, the GE equation predicts a corrosion rate of 4.2×10^{-3} mils/year. At 10 ppm oxygen the corrosion rate is 6×10^{-2} mils/year. The first value seems unnaturally low, and this may be due to a bias in the oxygen dependence coefficient in the GE equation which is based on plugging temperature data rather than oxygen activity. The GE equation is estimated to be correct within a factor of 2 to 5.

The term $Z(t)$ arises from solution of a differential equation, and is exact within the uncertainty in λ .

The fractional deposition f_i is probably the largest uncertainty in the analysis. Not only do we not know the deposition patterns very well, we don't even have a basis for determining how much in error the deposition estimates are. Values of f_i are listed in Reference 3; certainly other deposition schemes should be evaluated. The deposition prediction is probably good within a factor of 5.

The conversion factor K_i has been calculated by C. A. Mansius of FFTF Shielding Analysis Section.⁽⁹⁾ The numbers are accurate to $\pm 50\%$. Values of K_i obtained from computer code ISOSHL D are shown in Table 2.1 in (R/hr)/Ci deposited. A piping layout was taken from Bechtel drawing B4-03002-69.

TABLE 2.1. Values of K_i

	$K_i, (R/hr)/Ci$					
	30 in. Hot Leg	Pump	20 in. Hot Leg	IHX	Cold Leg	Cold Trap
^{60}Co	0.3	0.0065	0.44	0.0039	0.44	0.209
^{58}Co	0.1	0.0018	0.157	0.00091	0.157	0.069
^{54}Mn	0.091	0.0017	0.14	0.0009	0.14	0.062

The effect of uncertainties in the variables in Equation (1) can be calculated as follows:

If all the variables in Equation (1) are independent, then

$$\log L = \log S - \log R + \log \left(\frac{Z}{\lambda}\right) + \log f + \log K(10) \quad (2)$$

and the fractional change in L, differentiating Equation (2)

$$\frac{dL}{L} = \frac{dS}{S} + \frac{dR}{R} + \frac{df}{f} + \frac{dK}{K} \quad (3)$$

An uncertainty of $\pm 40\%$ means a fractional variation of 0.4, a variation of a factor of 2 means a fractional change of 1. Estimating the quantitative uncertainties, $\frac{dS}{S} = 1$, $\frac{dR}{R} = 2$ to 5, $\frac{df}{f} = 2$ to 5, and $\frac{dK}{K} = 0.5$, then $\frac{dL}{L} = 5.5$ to 11.5. Thus, the estimate of radiation level could be in error by more than an order of magnitude. This means that the radiation level estimates of 0.007 R/hr (IHX) to 0.15 R/hr (hot leg) and 0.36 R/hr (cold trap) may be increased to 0.07, 1.5 and 3.6 R/hr, respectively. Contact maintenance becomes economically difficult between 0.1 and 1 R/hr, and operationally difficult above 1 R/hr.

This analysis assumes that all the functional variables are completely independent, (i.e., that factors contributing to S have no bearing on R, f, or K). It is usually required for this type of analysis that the deviation or fractional error be small, but with the assumed independence of all variables, the reasoning should be valid for larger fractional errors.

Note that the weak link in this analysis is the uncertainty in corrosion and deposition behavior. There is really not much basis for estimating dR/R and df/f , especially when one considers all the variables such as oxygen level, velocity, mass flowrate, geometry, etc. that contribute to R and f.

The conclusion is this: For the initial operating conditions established for the FFTF, predicted radiation levels due to deposited corrosion products indicate that maintenance and repair operations planned for the facility can be carried out without undue economic penalty. However, indications are that after the ultimate FFTF operating conditions are achieved, deposited corrosion product activity may cause an economic or operational impact to maintenance and repair of the HIS components. This situation will most likely arise sooner in the closed loops.

While the reactor will not be faced with the activity problems for 5 or 6 years, plans to accommodate higher activity levels must be made very soon, if they are needed. The predicted activity levels have a large uncertainty factor, first, because the data used in the calculations are not reliable nor complete, and second, the corrosion rates are highly dependent on the oxygen content of the sodium coolant, and this depends on a variety of operating conditions. Before recommending expensive design modifications, efforts should be made to improve the calculational predictions. If the predicted activity levels prove to be real, it will be necessary to develop methods for activity control (preferential deposition), or decontamination methods, or review maintenance and repair procedures and perhaps provide more remote capability before system temperatures can be permitted to approach the ultimate operating condition.

SODIUM REMOVAL AND MATERIALS REQUALIFICATION

G. R. Bloom, R. R. Borisch, and W. F. Brehm

The purpose of this task is (1) to develop and evaluate methods for removal of sodium from equipment to be removed from the FFTF, (2) to develop methods for requalifying equipment for sodium service after it has been removed from the sodium, (3) to bring requalifying methods within the scope of materials coding procedures.

The activities of this task have to do with the problem: how do we know that sodium removal, repair, and reinsertion of a reactor component does not irreversibly damage it so that safe reactor operation is compromised? It is presently planned that parts requiring repair will be cut out of the reactor for repair. Thus, both the part and the adjacent pipework will have to be requalified for nuclear code service. Are the new material standards and tests sufficient or will new ones have to be developed?

The present state-of-the-art is that most laboratories have routinely repaired and replaced components, but the extent of the documentation of these procedures is uncertain. It is known that "pressure boundary" repairs were made on the EBR-II secondary systems.⁽¹¹⁾ One of the necessary functions of this task will be gathering and documenting all available information on repair and reinsertion of components.

Any acceptable requalification document must include procedures on how to remove, clean, store, and reinsert an item. It should also give guidelines on what items must be considered when determining economic feasibility of requalification versus replacement. Such a document for the FFTF is one of the end product goals of this task.

Effect of Sodium Exposure and Removal on Bellows-Sealed Valves

Twelve bellows-sealed valves which show extensive service on the Radioisotope Transport Loops were cleaned and reinstalled on the Fission Product Control Screening Test Loop in two groups of six valves each. Initial metallographic examination of the twelve 3/8-in. sodium valves described under "Sodium Removal Studies for Piping System" in Reference 3 indicates that all six valves in the first test run cracked on the inside convolutes of the bellows as shown in Figures 2.8a and 2.8b. Cracking was most severe on the sodium side. However, no

Neg 470-1448B

15X

FIGURE 2.8a. Cross Section of Valve Bellows.
Arrow points to typical crack location.

Neg 470-1440B

500X

FIGURE 2.8b. Inside Convolute of Valve Bellows on Sodium Side.
Valve had seen approximately 6000 hr of service prior to cleaning and reinsertion in the FPCTL for 400 cycles in 400 hr at 1000 °F and an oxide content of about 30 ppm. As-polished.

cracking has been found on the second set of six valve bellows in the second test run. Difficulties in loop operation resulted in the two sets of valves being run under considerably different conditions during their 400 hr in the Fission Product Control Screening Test Loop (FPCSTL). The first set ran at a nominal temperature of 500 °F with an oxygen content of about 30 ppm in the sodium. The loop was not operating properly and had to be drained several times during the test. The second set (uncracked) were at a nominal temperature of 1000 °F in the FPCSTL with an oxygen content of 10 to 12 ppm in the sodium. The absence of cracks in the second set of six valves indicates that cracking in the first set might well have been caused by a combination of the high oxygen content of the sodium (present much of the time) and the exposure of the valves to a storage environment where oxygen-contaminated sodium was left on the bellows during loop shutdowns. Since the solubility of oxygen in sodium at 500 °F is low, it is probable that considerable sodium oxide may have been on the bellows for the entire test at 500 °F.

Carbon Transport in FFTF

W. F. Brehm

An analysis of the potential for strength loss in FFTF heat transport piping due to carbon removal from the stainless steel construction material has been completed and a detailed report provided to FFTF design. The following summarizes this report.

The problem is possible removal of carbon from stainless steel by sodium with subsequent reduction in strength. It would then be necessary to use design stress limits for Type 304L SS rather than 304. The reason for concern is that carbon removal from the hot zone all-austenitic stainless steel sodium loops has recently been seen^(1,12,13) in unperturbed systems, (no introduction of carbon or carbon-bearing species into the sodium or cover gas).

The hot zone decarburization can be explained by this model: The preferential removal of chromium from stainless steel by sodium occurs possibly by oxidation. This removal of chromium causes the activity of carbon in the steel to increase,⁽¹⁴⁾ and hence carbon will migrate out of the steel into the sodium to equalize carbon activities. ~~As the sodium~~ then moves to a region of lower temperature, the equilibrium content of the Cr and C dissolved in the sodium decreases, and Cr_{23}C_6 will be precipitated. The decarburization can be repeated when the sodium again passes through the hot leg. This decarburization phenomenon is thus directly linked to the selective removal of chromium; if corrosion is not occurring then there should be no decarburization.

For the FFTF heat transport system, however, the components of concern such as the vessel outlet nozzles, hot leg, pump, and heat exchanger will be in the falling-temperature region. ~~This is true because the high-heat-flux in the core raises the~~ fuel cladding surface temperature above the bulk sodium temperature, and because the present design calls for bypass cooling of the outlet nozzle. Thus the heat transport system should be a deposition or possible carburization site rather than a decarburization site. It was therefore recommended that decarburization will not be a problem and that 304 stainless steel properties can be used for design.

A simple diffusion model for decarburization showed that 80% of the carbon would be retained in a 1/2-in. thick pipe wall even if it were decarburizing, for 20 years at 1050 °F. The same model predicts complete decarburization of fuel cladding after 10 months at 1200 °F. Thus far, even though some decarburization has been reported for the hot zone of sodium loops, the decarburization is not complete.⁽¹²⁾ It is not certain what the total effect of diverse phenomena such as ferrite layer formation, decarburization, radiation hardening, and sigma phase formation on the strength and ductility of the cladding will be.

The previous discussion has been concerned with the decarburization of a nonperturbed system. A potentially more serious problem is the effect of carburization of the stainless steel from introduction of carbon into the sodium. A key question not yet answered is: What is the amount of carbon or carbonaceous species injected into the sodium necessary to override the normal hot-to-cold-leg carbon transport and produce carburization of the fuel cladding?

CONTROLLED FAILURE METHODS FOR DEFECT FUEL PINS

W. F. Brehm and E. R. Wood

The purpose of this activity, which is being done for FFTF Fuels Evaluation Department, is to find a method for producing failure in a fuel pin in EBR-II at a known, controlled time. One concept for doing this is to drill a hole in the fuel cladding, inject a sodium-soluble alloy, and proceed with the irradiation. The alloy dissolves at a known fairly rapid rate, producing a hole in the fuel cladding. A thin protective layer of a slightly more soluble cladding may or may not be plated over the alloy. A schematic of the desired reaction is shown in Figure 2.9.

The test systems are thermal convection loops; the soluble alloy is "Silfos" (80 Cu-15 Ag-5 P), the protective platings are Ni and Cr.

Table 2.2 shows results obtained to date. Since it is now known that the unprotected Silfos dissolves in about 10 hr in 950 °F sodium, the program is now attempting to find the correct thickness of overlying nickel plate to last several hundred hours. We feel that additional tests should include

- (1) tests of present materials at higher sodium velocities,
- (2) use of a Ni- or Pt-base braze alloy plug that will dissolve more slowly and eliminate the need for the protective plating.

TABLE 2.2. Controlled Fuel Pin Failure Tests

Protective Plate ^(a)		Test Conditions		Results
Material	Thickness, mils	Temperature, °F	Time, hr	
None		950 ^(b)	1 ^(c)	Plug partially dissolved
			1 0	Plug mostly dissolved
			100	Plug completely dissolved, sodium entered hollow pin
Ni			1 ^(c)	Plating partially dissolved, plug nearly intact
			10 ^(c)	Plating dissolved, plug partially dissolved
			100	Plating dissolved, plug dissolved, sodium entered hollow pin
Cr		950	10	Plating badly cracked. (Large mismatch in thermal expansion between Cr and SS). Evidence of edge dissolution of plating and sodium underneath. Plug apparently intact.
			100	
Ni	1.5	950	100	Plating slightly dissolved but apparently intact;
	2.5		100	
Ni	0.6	950	100	Test in progress
	0.9		200	

a. Cu-Ag-P alloy plug in all cases.

b. All tests done in thermal convection loops with sodium velocity ~ 0.2 ft/sec.

c. Reported previously in Reference 3.

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3.0 SODIUM SYSTEMS ENGINEERING

INTRODUCTION

V. F. FitzPatrick

The mission of the Systems Engineering Section is three-fold:

- Provide engineering support to the Sodium Technology Department.
- Establish the basis for design and operation of FFTF sodium purification systems.
- Develop a technical base for improved design and operation of sodium purification systems in LMFBR's.

The specific functions of engineering support activity include responsibility for the design, fabrication, installation and check out of sodium facilities required for experimental programs in the Sodium Technology Department, and the developments and improvement of engineering devices to advance the state-of-the-art in sodium systems. Attendant with these major functions are the establishment of appropriate quality assurance procedures, RDT standards and commercial qualification programs.

The specific functions of the sodium purification activity include responsibility for direct support to the FFTF project for the design and design verification of the FFTF sodium purification systems, obtaining operational data from major FFTF support facilities to provide input to the FFTF operations manual. The longer range goals are to develop a technical base to improve the hydraulic, thermal, and crystallization characteristics of sodium purification systems.

ENGINEERING SUPPORT

Prototype Applications Loop (PAL)

V. F. FitzPatrick

Background

The Prototype Applications Loop (PAL) is an isothermal, forced convection controlled chemistry loop. The PAL is a versatile sodium system capable of operating to 1200 °F and 40 psig and is designed for experimental flexibility without modifying the basic loop system. During the previous quarter the design of the PAL was essentially completed, and a Quality Assurance Plan for the design, fabrication, and installation prepared. A design review was conducted to ensure the design included all of the features required for the experimental program.

Work This Quarter

The Quality Assurance Plan for PAL was approved by FFTF management and forwarded to RDT for approval. RDT approved the PAL Quality Assurance Plan and requested that PNL identify critical items within the PAL system. RDT also required that positive steps be taken to insure the quality of critical items by employing nuclear codes and RDT standards.

The activities on PAL have been directed toward resolving the issue of critical items and codes and standard so that the design phase may be completed and procurement initiated. The critical items in the PAL system and the components that could serve as pilot items for FFTF have been identified. A letter to RDT containing the pertinent information and a proposed plan of action has been drafted and will be forwarded early in the next reporting period.

The present position regarding codes and standards is that there are no critical items in the PAL system, based on the following definition of a critical item:

A critical component is any component which, if it fails, will seriously impair the safe, reliable operation of PAL or will seriously compromise the experimental results and/or programmatic objectives.

Therefore, the piping system will be designed to B 31.7, Class II, and the vessel to ASME Section III, Class C. The system will be fabricated in accordance with the rules in B 31.7 and Section III, Class C and stamped accordingly.

A stress analysis for the PAL piping system is being performed in accordance with the rules in B 31.7, Class II.

Small Components Evaluation Loop (SCEL)

D. W. Bennett and T. J. Owen

-----The existing Small Components Evaluation Loop (SCEL) is being modified to accommodate several developmental in-line instrument packages.* A header system is being installed that will accommodate a LASL-built vacuum distillation sampler, a LASL orifice plugging meter, an EBR-II valve-type plugging meter, a carbon meter, an oxygen meter, and a bypass sampler, with extra space for one more unidentified test unit. The header system will provide sodium from the main SCEL facility at 1200 °F, 50 psig and at approximately 5 gal/min maximum. (Figure 3.1). Trace heating on the header system and the experimental packages will be accomplished with a configuration that is similar to that of the system conceived for the Prototype Applications Loop (PAL). This involves the use of proportioning-type controllers with feedback control and

* For additional information on the SCEL modifications, see Section 2.

FIGURE 3.1.
Engineering Flow Diagram of the
S.C.E.L. Header/Sampling System

voltage limiters that are installed in a master-slave arrangement to allow sequential heating of the piping as described fully in Reference 1. The heater elements are of a radiant-type clam shell design that do not require intimate contact with the pipe surface (Figure 3.2). Use of this newly designed equipment on the SCEL sampling station will precede fabrication of the PAL facility by several months and will therefore provide beneficial experience for the operation of PAL.

A flow-through expansion tank will replace the existing SCEL tank and will be provided with access ports for materials addition and for cover gas sampling through experimental vapor traps. The new tank is somewhat larger than the existing unit, which reflects the increased loop volume due to the added header system.*

The materials addition ports in the new expansion tank will be used for placement of sample material coupons into the flowing sodium and for the addition of minute amounts of impurities to test the response of the experimental instruments to step-changes in the various coolant impurity levels.

The original SCEL design criteria are being adhered to for modifications to the main loop (ANSI B31.1.0, Code Case N-8 Reopened) which only includes the new expansion tank. The header piping is located in an adjacent experimental cubicle and is being documented as per BNWL-MA-64 as a nonstandard system, since it is not an integral part of the main loop and it contains components that are not specifically authorized by code. However, a stress analysis has been performed on the entire system with a computer code for B31.1.0⁽²⁾ and verified by a computer code B31.7.⁽³⁾ The computer code for B31.7 is being used for the PAL system.

* *For additional information on the SCEL modifications, see Section 2.*

GENERAL NOTES:
FOR NUMBER OF THERMOCOUPLES AND DESIGNATIONS ON EACH INDIVIDUAL HEATER OR HEATER SECTION SEE WG # H-3-29033

FIGURE 3.2. Typical Trace Heater Installation for S.C.E.L.

Modifications are to be completed early in the next report period, and beneficial use will be realized immediately thereafter.

A detailed status report of design, fabrication and procurement follows:

SCEL SAMPLE STATION HEADER SYSTEM

1. Mechanical design	100% complete
2. Mechanical drafting	100%
3. Electrical design	95%
4. Electrical drafting	85%
5. Procurement	95%
6. Receipt of procured items	~60%
7. Fabrication	35%

SCEL EXPANSION TANK

8. Mechanical design	~50% complete
9. Mechanical drafting	~50% complete
10. Electrical design	Not started
11. Electrical drafting	Not started
12. Procurement	65% complete
13. Receipt of procured items	~60% complete

Cover Gas Evaluation Loop (CGEL)

T. J. Owen

The System Chemistry Section requested that modifications be made to the Cover Gas Evaluation Loop (CGEL) to permit testing and evaluation of new vapor trap designs." The modifications include new heaters for the feed tank, installation of four vapor traps and associated gas lines and thermocouples, and the installation of a new liquid level measuring system.

* For further information see Section 2.

The modifications have been completed and are shown in Figures 3.3 and 3.4. The CGEL has been started up and check out of the new modifications is in progress. Experimental testing of the vapor traps will begin as soon as the check out is completed.

Spare Cold Traps for CGEL and FPSCTL

T. J. Owen

Spare cold traps were fabricated for the CGEL and FPSCTL loops. The design shown in Figure 3.5 is similar to those for PAL and FRTEF. They will be used to obtain preliminary design verification data for PAL and FRTEF and ultimately FFTF.

The fabrication of these cold traps provided an opportunity to evaluate cleaning procedures and the cost of maintaining a level of cleanliness comparable to Level B, specified in RDT F5-1. The details of the cleaning procedures and inspection requirements are reported in Section 2. In general the job went very smoothly and the cleanliness proves to be beneficial during welding. Nondestructive evaluation of the welds indicated that the welds were of a higher quality than welds made in normal shop practice. However, the cost of maintaining a high level of cleanliness is high. Normal shop scheduling is difficult if the fabricator is not set up to routinely maintain a high level of cleanliness. The cold traps cost about four times more than if they were fabricated according to normal shop practice.

A critique of the job is scheduled with the fabricator early in the next reporting period, and this information will be used to formulate the fabrication procedure for PAL. There are many improvements that can be made to reduce costs and improve scheduling. These comments will also provide input for

ITEM NO.	DESCRIPTION	MATERIAL OR SPEC.
1	ASSEMBLY	
1	VAPOR TRAP	
2	HEATER	304 SST
3	INSULATION	304 SST
4	INSULATION	304 SST

- GENERAL NOTES:
- 1. UNLESS OTHERWISE SPECIFIED
 - 2. TOLERANCES FRACTIONAL 1/16, ANGULAR 1/2°-30°
 - 3. ALL MACHINED SURFACES OF SURFACE TEXTURE SHALL BE IN ACCORDANCE WITH MIL-STD-199 (TEST REQ)
 - 4. ALL SURFACES SHALL BE FINISHED TO REMOVE ALL BURRS
 - 5. ALL QUALITY TO BE AS SPECIFIED OR APPROVED
 - 6. ALL HEATED SURFACES OF HEATERS (PT #3) MUST BE IN CONTACT WITH VAPOR TRAP (PT #2).

VAPOR TRAP ASSY

VAPOR TRAP
MATERIAL: 304 SST & NOTED

H-3-29904
Vapor Traps for the Cover Gas Evaluation Loop

FIGURE 3.4.

FIGURE 3.5. Typical Spare Cold Trap Unit

H-3-29914

formulating a training program for craftsmen to work to Level B requirements. The practice session was definitely worth the extra time and expense and will greatly benefit future work.

Assistance to Large FFTF Support Facilities

V. F. FitzPatrick and R. V. Bowersock

One of the important missions of the Sodium Technology Department is to provide the expertise and hardware for the cover gas, sodium purification, and sodium purity monitoring systems for large FFTF support facilities such as the Fast Reactor Thermal Engineering Facility (FRTEF) and the High Temperature Sodium Facility (HTSF). This is to ensure that FFTF quality sodium is used in important experiments and that design verification information is obtained for FFTF prototype equipment. The first major facility that the Sodium Technology Department will provide support to is the FRTEF.

Systems Engineering in cooperation with the Systems Development and System Chemistry Sections will provide a cold trap, plugging meter, oxygen meter, distillation sampler, bypass sampler, vapor traps and a gas chromatograph for FRTEF. Systems Development will be responsible for selection of the plugging and oxygen meters and the distillation sampler. Sodium Analysis will be responsible for the design of the vapor traps and procuring the gas chromatograph. Systems Engineering will be responsible for the NaK cooled cold trap, which is a prototype of the cold trap for the FFTF Closed Loops, and the preliminary design of the systems to be attached is FRTEF. The FRTEF designers will provide the detailed design of support systems to maintain continuity and the required level of quality. Systems Engineering will also be responsible for the fabrication and check out and will audit the installation.

The conceptual design of the FRTEF Support Systems has been completed and is shown in Figure 3.6. The flow path is from the suction side of the main FRTEF pump to the support systems pump, splitting off to the cold trap and the sampling header, and returning to the FRTEF dump tank. The details of the physical arrangement will be completed in the next reporting period and the information given to the FRTEF project.

The sizing of the cold trap (including the economizer), crystallizer tank, and NaK heat rejection system are completed.

Sodium Sampling Activities

K. C. Knoll

A small bypass sampler that may be operated manually is required for the Sodium Technology's program for large FFTF Support Facilities and ultimately for the nonradioactive systems in the FFTF.

Design work on this type of sampler was begun late in the quarter, and the preliminary design is completed. The cover over the "U" tube sampler acts as both an isolation barrier when the sampler is in use and a "trace heater." The first model of the sampler will be installed on the SCEL Sample Station Header for checkout. A perfected model will be installed on the FRTEF as part of the sample package.

Small Valves for Sodium Service

V. F. FitzPatrick

The FRTEF project prepared a draft of a RDT standard "Small Valves for High Temperature Sodium Service, RDT E1-19T" during the reporting period. The Sodium Technology Department will be using a considerable number of small valves in support of the FFTF and therefore is very interested in this standard. A small task force was formed to assist the FRTEF in obtaining

input from the small valve industry. The task force reviewed the standard with three well known manufacturers of small valves that were not covered by the FRTEF personnel. The comments from the manufacturers have been obtained and forwarded to the authors of the standard. In general, the manufacturers were very cooperative and provide beneficial input to the standard.

SODIUM PURIFICATION

G. R. Bloom and D. H. Lester

The sodium purification activity provides direct support to the FFTF project for the design and design verification of the FFTF sodium purification systems. Another function of this activity is to obtain operational data from purification systems on major FFTF support test facilities to provide input to the FFTF operations manual. The long range objective is to develop a more complete technical base for design and operation of purification systems including improvement of hydraulic, thermal, and crystallization characteristics of sodium cold trap systems.

Crystallizer Mockup Facility

D. H. Lester and G. R. Bloom

At present, program plans call for evaluation of methods to improve thermal, hydraulic and crystallization performance of cold traps. Due to restrictions in time available and limitations in sodium instrumentation, it is probable that this goal would be difficult to achieve if only tests in sodium were used. Water mockups in "see through" systems allow evaluation of internal crystallizer flow patterns and mixing behavior in full scale test systems. The theoretical heat transfer equations can also be partially checked in a water system. In the longer range, comparison of crystallization in a cold trap type of crystallizer with crystallization in a mixed chemical

process crystallizer would provide insight as to the validity of extrapolation of crystallizer theory from the chemical process crystallizer to sodium cold traps. A water mockup test system will be a valuable supplement to sodium cold trap test systems.

A more immediate need for water mockup testing is in the area of diffusion cold traps. Within a short time (early FY-71), design concepts must be fixed for the FFTF diffusion cold traps (notably for the closed loop secondary heat transport system). Some question as to hydraulic and thermal stability has been expressed by previous users of diffusion cold traps.

A small water mockup facility is being designed to run diffusion trap and forced convection trap mockup tests. Initially the system will consist of a pumped loop with 0 to 60 gal/min flowrate capability and a 1 in. glass test section containing a glass model diffusion cold trap. The 2 MW closed-loop secondaries in the FFTF will have a maximum design flowrate of 135 gal/min at a cold leg temperature of 800 °F in a 3-in. pipe system. The Reynolds number for these conditions is 4.6×10^5 . This same Reynolds number will be obtained in the 1-in. glass pipe test section with a 58 gal/min waterflow at 160 °F. Tests with dye tracers will be run to evaluate hydraulic properties of the diffusion cold trap (e.g., depth of penetration of turbulence into the dead leg and velocity sensitivity).

At a later date, the diffusion cold trap testing will be completed and modifications will be made to the basic pumped loop to prepare for forced convection cold trap testing. The forced convection cold trap mockup system will include a 70 gal clear plastic tank with internal hardware similar to sodium cold traps.

The design of the system is essentially complete. Many components are in hand and others are on order. It is expected that construction will begin immediately.

FFTF Purification Requirements Review

D. H. Lester

Earlier in the year this review was initiated at the request of the FFTF Sodium Technology Section. The intent of this review was to answer a request from RDT for a summary of the basis for purification system design in the FFTF reactor heat transport systems. The review included review of all published and unpublished cold trap and hot trap information available, calculations of cold trap crystallizer heat effects, and estimation of oxide source levels in the FFTF heat transport systems.

The original document is being revised into small **tradeoff** summaries and information packages for RDT and other FFTF contractors. (Tradeoff studies of diffusion versus forced traps in the closed-loop secondary and of one versus three cold traps in the reactor secondary are some examples.)

An information package detailing the heat transfer calculations for forced convection cold traps is being prepared. This package gives the details leading to the conclusion that forced convection crystallizers should not have a volume larger than 300 gal. Some of the details of this heat transfer analysis can be found in previous quarterly reports. (1)

FFTF Design Support Work

D. H. Lester

Forced Convection Cold Traps

The draft RDT standard for liquid metal cooled sodium cold traps was reviewed and comments transmitted to the authors. The data sheets accompanying the standard were revised.

Preliminary heat transfer calculations for the FRTEF cold trap (similar to the proposed FFTF closed loop primary cold trap) were completed. Liaison with FRTEF designers is being maintained by R. V. Bowersock and further design information will be transmitted through him.

Diffusion Cold Traps

At present, it is proposed that diffusion cold traps be used in the NaK heat rejection systems and the closed-loop secondary sodium system. In addition to the hydraulic testing described above, it is necessary to carry out a preliminary heat transfer analysis to estimate the size of the diffusion cold traps. A diffusion cold trap generally consists of a dead-end leg of pipe tied from the main system piping. The dead-leg is cooled (normally by free convection with fins on the pipe exterior) so that a region of frozen sodium exists near the dead end of the cold trap.

The following approximate method was used to estimate the required dead leg length:

The point at which the turbulence from the main stream subsides is called length position $Z = 0$ and the dead end of the cold trap is called $Z = L$. The total trap length will be L plus the depth of turbulence penetration (from hydraulic analysis). The radial direction is designated $r = 0$ at the center line and $r = R$ at the cold trap wall.

The following additional notation is used:

T = temperature at any point

T = main sodium system temperature

T_{∞}^o = ambient temperature surrounding cold trap

U_A = heat transfer coefficient from the sodium to the gas outside the cold trap

k_{Na} = thermal conductivity of the sodium

It is assumed that heat is transported in the region $0 \leq z \leq L$, $0 \leq r \leq R$ by conduction only. The value of U_A takes into account free convection and radiation from the cold trap surface to the air. U_A also accounts for the presence of fins. Define:

$$v = T - T_{\infty}$$

$$h_a = \frac{U_A}{k_{Na}}$$

A heat balance on a differential element in the cold trap gives:

$$\frac{\partial^2 v}{\partial r^2} + \frac{1}{r} \frac{\partial v}{\partial r} + \frac{\partial^2 v}{\partial z^2} = 0 \quad (1)$$

with boundary conditions:

$$\begin{aligned} \text{at } z = 0 \quad v &= f(r) \\ z = L \quad \frac{\partial v}{\partial z} &= -h_a v \end{aligned} \quad (2)$$

$$\begin{aligned} r = 0 \quad v &= \text{finite} \\ r = R \quad \frac{\partial v}{\partial r} &= -h_a v \end{aligned}$$

The solution is given in Carslaw and Jaeger⁽⁴⁾ as:

$$v = \sum_{n=1}^{\infty} A_n J_0(r\alpha_n) \frac{a \cosh a_{\eta}(L-z) + h_a \sinh a_{\eta}(L-z)}{a_{\eta} \cosh a_{\eta} L + h_a \sinh a_{\eta} L} \quad (3)$$

where

$$A_n = \frac{2\alpha_n^2}{R^2 (h_a^2 + \alpha_n^2) J_0^2(R\alpha_n)} \int_0^R r f(r) J_0(r\alpha_n) dr \quad (4)$$

and α_η are roots of:

$$-\alpha_\eta J_1(\alpha_\eta R) + h_a J_0(\alpha_\eta R) = 0 \quad (5)$$

The roots of Equation (5) are tabulated in Carslaw and Jaeger. (4)

The solution is further simplified by assuming that

$f(r) = T_o - T_\infty = \text{a constant}$. Furthermore the series described by Equation (3) converges rapidly for all practical cases so that only the first term is necessary. The solution is then

$$T = T_\infty + \left[\frac{2\alpha_1 v_o R J_1(\alpha_1 R)}{R^2 (h_a^2 + \alpha_1^2) J_0^2(\alpha_1 R)} \right] \left(\frac{\alpha_1 \cosh[\alpha_1(L-z)] + h_a \sinh[\alpha_1(L-z)]}{\alpha_1 \cosh[\alpha_1 L] + h_a \sinh[\alpha_1 L]} \right) J_0(\alpha_1^r)$$

where $\alpha_1 = \text{first root of Equation (5)}$.

For the cases calculated (3 in. diameter cold trap) the temperature gradient in the r direction was found to be negligible so that $J_0(\alpha_1 r)$ can be considered a constant. Temperature is then only a function of z . For large pipe diameters this would probably not be true.

Summary of Results

Case I: $T_\infty = 100^\circ\text{F}$ $k_{Na} \approx 40$ Btu/hr-ft- $^\circ\text{F}$
 (Closed Loop $T_o = 800^\circ\text{F}$ $U_A \approx 5$ Btu/hr-ft²- $^\circ\text{F}$
 Secondary)
 $R = 1\text{-}1/2$ in.
 $L = 2$ ft

Z, ft	0	0.5	1.0	1.5	2.0
T, $^\circ\text{F}$	800	414	243	169	152

Case II: $T_{\infty} = 100^{\circ}\text{F}$ $kN_a \approx 40$ Btu/hr-ft- $^{\circ}\text{F}$
 (Closed Loop
 Secondary) $T_o = 800^{\circ}\text{F}$ $U_a \approx 5$ Btu/hr-ft- $^{\circ}\text{F}$
 $R = 1\text{-}1/2$ in.
 $L = 1\text{-}1/2$ ft

z, ft	0	0.5	1.0	1.5
$T, ^{\circ}\text{F}$	800	417	256	214

The results indicate that an "L" of 1.5 to 2 ft would be desirable. The total trap length would be L plus the turbulence penetration depth. Since the diffusion trap is proposed for the cold leg of the closed-loop secondary a nominal temperature of 800°F was chosen.

CRCTA MIXING TESTS

D. H. Lester

As part of the sodium technology support for the FFTF test systems, a mixing mockup study of the large vessel in the Composite Reactor Components Testing Activity (CRCTA) has been initiated. The vessel in question is a 45,000 gal tank 16 ft in diameter. It will contain mockups of FFTF fuel handling machinery in a high temperature liquid sodium environment. Sodium will be circulated in and out of the vessel through a 500 gal/min driver loop. The driver loop flow will be used to mix the tank through the use of nozzles or jets.

The water test system will consist of a 3/4 in. piping system and 110 gal plastic tank. Capability for a "once-through" flow will be incorporated so that open-loop dynamic response of the mixing tank mockup can be tested. Dye and conductivity tracer tests will be used to determine internal circulation patterns and overall mixing response of the tank (i.e., comparison to an ideal mixer). Various types and sizes of inlet nozzles will be tried in the tank and sensitivity to

flowrate will be investigated. The test procedures and methods of data interpretation will be similar to those used for the SCTF Mixing Mockup Study.⁽⁵⁾ The water testing work will be necessarily short term as design concepts for CRCTA must be fixed by early FY-71.

At present, work is in progress on construction of the plastic tank and procurement of the required equipment is proceeding. It is expected that testing will begin in late July.

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4.0 SYSTEMS CHEMISTRY SECTION

INTRODUCTION

F. A. Scott

The mission of the Systems Chemistry Section is to provide support for the FFTF development effort through the measurement and interpretation of coolant systems chemistry. Unquestionably one of the primary causes of the wide disagreements which have occurred in the results of the various sodium technology development efforts has been a lack of control over the quality of the sodium employed in the work. A major activity of the group is therefore measurement of sodium and cover gas impurity concentration levels in the Hanford-located development systems. Continuous updating and standardization of procedures are required to assure adequate correlation of data both internally and with other LMFBR sites.

A second major function of the section is the generation of adequate technology for fuel failure location in the FFTF. During initial phases of operation, reactor availability will be markedly affected by the ability to rapidly locate and remove failed fuel assemblies. A good fuel failure location system is essential to maintain reactor availability at a maximum.

SAMPLING AND ANALYSES OF RADIOACTIVE SODIUM

High Radiation Level Analytical Facility Design

L. F. Lust and S. E. Nichols

The objectives of this activity are to provide proven equipment and techniques to analyze radioactive sodium from the FFTF and to develop design criteria and requirements for the FFTF chemical analysis laboratory.

Analytical procedures are well established for the determination of impurity levels in nonradioactive sodium. In order to adapt these procedures to the analysis of highly radioactive sodium coolant, a **mockup** facility was constructed.

The mockup facility consists of two adjacent portions, one with an air atmosphere and the other with a pure argon atmosphere. The operation of the equipment and several procedures to be used for analysis of radioactive sodium have been tested in the air atmosphere portion of the mockup.

The inert atmospheric portion is a sealed cell, approximately 7 ft by 8 ft by 9 ft, equipped with an evacuable air lock, a viewing window, an argon purification system, master-slave manipulators and instruments for monitoring the purity of the cell atmosphere. See Figure 4.1. The manipulators tested and used in the inert-atmosphere cell are the same pair of Central Research Model J sealed manipulators which were used in the air-atmosphere cell for mechanical testing. Ports are also provided in this cell for the installation and testing of the lighter duty, newly developed Model L manipulators. The argon purification system consists of two Vacuum/Atmospheres HE-293 Dri-Train units. Each has a capacity of 100 ft³/min and has dual full-flow purifiers. These two units are installed so that either unit can be on stream alone or both can be on stream simultaneously. Instruments provided for monitoring the purity of the cell atmosphere are Panametrics hygrometer, CEC moisture monitor, Westinghouse oxygen analyzer, Teledyne oxygen analyzer, and Honeywell potentiometric strip-chart recorder. Gages and automatic control circuits for regulation of the cell pressure and for control of the purifier regeneration cycle are located in the purifier consoles.

Purity of the argon atmosphere in the cell is of utmost importance for sodium analysis. Conventional inert glove boxes used for sodium analysis are normally maintained below 1 ppm oxygen and 0.5 ppm water. An atmosphere of this purity has not previously been maintained in a remotely operated facility and there has been doubt in the minds of some investigators that an atmosphere of adequate purity could be maintained in a master-slave-manipulator-equipped facility during operation.

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FIGURE 4.1. Remote Inert Atmosphere Analytical Mockup

To initially establish the inert atmosphere, the air was purged from the cell with bottled argon. Approximately 1500 ft³ of argon (three times the cell volume) was required to reduce the oxygen content to 1.2%. The purifiers were then used to reduce the oxygen level to less than 1 ppm. Typical curves of oxygen removal with one purifier on stream are shown in Figures 4.2 and 4.3. Figure 4.2 shows that one purifier has the capacity to reduce the oxygen in the 504 ft³ cell by approximately 4000 ppm or to remove approximately 2 ft³ of oxygen. Figure 4.3 illustrates the very rapid and complete removal of oxygen by a purifier bed which is not loaded to its capacity.

The purification units were even more effective in removing water. The first purification unit on stream brought the water concentration in the cell down to 12 ppm. Since the initial purification, water content, as measured with the Panametrics hygrometer, has never risen above 0.33 ppm, although the cell has been operated for one month with the same purifier continuously on stream.

The ultimate test of the inert-atmosphere analytical cell is its usefulness in performing analyses of sodium metal. The determination of oxygen by the amalgamation procedure was used for this evaluation because this analysis is sensitive to both oxygen and water in the cell atmosphere and is a well established laboratory procedure. The sodium samples were taken from the sodium purification loop and ranged from 10 to 20 ppm oxygen. As nearly as possible, all analytical operations and equipment were identical to those used in routine analyses by the Systems Chemistry Section. All steps normally performed in an inert atmosphere were done remotely with CRL Model J manipulators in the inert mockup cell.

FIGURE 4.2. Effect of One Purifier Bed with Initial Oxygen Level Higher Than Bed Capacity

FIGURE 4.3. Effect of One Purifier Bed with Initial Oxygen Level Lower Than Bed Capacity

The one difference in procedure was in-cell drying (at 150 °C) of the glassware instead of air drying at 150 °C and subsequent transfer through the airlock into the inert atmosphere glove box.

The samples analyzed in the inert mockup were also analyzed by the routine amalgamation procedure used in Building 325. The results are compared in Table 4.1. Precision of analyses in the remote facility was as good as in the sodium laboratory. Agreement between the laboratories was also good. Piggy-back blanks obtained in the remote facility were slightly lower than in the sodium laboratory.

From these analyses the following observations can be made:

- The blanks obtained in the remote facility are at least as low as those in the sodium laboratory.
- Precision of the remote facility analyses is as good as that of the sodium laboratory.
- There is excellent agreement between the oxygen values obtained in the two laboratories.

The following conclusions can be drawn from the operational and analytical experience with the inert mockup facility:

- Atmosphere purity at least equivalent to that of a conventional inert glove box can be readily obtained and maintained during normal operation.
- The determination of oxygen in sodium by the amalgamation procedure can be performed without difficulty and with results equivalent to those obtained in a conventional sodium laboratory.
- Mechanical operations required for the remote determination of hydrogen and carbon in sodium metal can be performed successfully.
- FFTF sodium can be taken from the reactor and analyzed immediately by laboratory techniques in common use today if the proper equipment is available.

TABLE 4.1. Comparison of Oxygen Determinations in Remote Facility and Sodium Laboratory

<u>Sample</u>	<u>Remote Facility</u>			<u>Sodium Laboratory</u>		
	<u>Blank, ppm Oxygen</u>	<u>Oxygen, ppm</u>	<u>Sample, avg</u>	<u>Blank, ppm Oxygen</u>	<u>Oxygen, ppm</u>	<u>Sample, avg</u>
1	1.6	16.3 15.3	15.8	1.7	15.9 16.3	16.1
2	1.5	11.5 13.3		1.8	19.0 ^(a) 11.3	
	1.4	10.2 10.2	11.3			11.3
3	1.4	14.8 14.2	14.5	1.8	16.3 17.8	17.0
4	1.5	18.3 19.6 20.0	19.3	1.6	16.6 16.6	16.6
5	1.5	12.6 11.1		1.8	10.5 11.1	
	1.1	11.5 11.3	11.6			10.8

a. Not included in average.

An objective of the program was to provide proven analysis equipment and techniques for highly radioactive sodium from the FFTF. A major portion of the work necessary for this has been completed. Before there is absolute assurance that adequate technology for remote analysis is available at FFTF start-up the following additional tasks remain to be completed:

1. Assembly and testing of each piece of equipment and each procedure for the determination of impurities other than oxygen in sodium, comparing the results with those obtained in the sodium laboratory.

2. Evaluation of standard analytical procedures, defined by ANL, as applied to remote operation.
3. Adaptation of the Model L manipulators to a remote sodium analytical cell, design and fabrication of equipment for installation and removal of manipulators.
4. Development of a method for leak-testing an inert-atmosphere cell after shielding is in place.

Because of program changes and reassignment of priorities, laboratory work on this phase of the program is being terminated. The feasibility of using a manipulator-equipped inert box for sodium analysis has been demonstrated, and a basic unshielded system is available. When firm requirements for the FFTF are established this basic system can be used, either directly or as a prototype, for the FFTF shielded analytical facility. In the meantime, the equipment will be deactivated and preserved intact until activities to provide the final FFTF shielded analytical facility are initiated.

A terminal report describing the facilities, the experimental work and the accomplishments of the program for the laboratory analysis of radioactive sodium is being prepared.

Remote Sampler Development

K. C. Knoll and E. L. Osborne

The objective of this activity is to develop and proof test remotely operable samplers for the highly radioactive sodium from the FFTF.

Sodium from the FFTF will be highly radioactive due to the presence of ^{24}Na . Little or no technology exists for sampling and immediate handling of sodium under this condition. Because of the radiation fields the operations will have to be performed remotely. As part of the development effort required,

a removable section bypass loop sampler has been constructed at PNL specifically for remote operation and is currently undergoing evaluation.

With the completion of the sampler modification and cooling profile measurements as described in the previous quarterly report (BNWL-1200-3, Section 4.1) ⁽¹⁾ a program of sampling the sodium from the Small Components Evaluation Loop was instituted. The purpose was two-fold: (1) to gain experience in the remote operation of the sampler and (2) to obtain a measure of any longitudinal segregation of impurities which might occur with the sampler as designed.

Remote operation did not prove to be difficult for a reasonably dexterous manipulator operator. A counter balance for the guillotine was installed on a trolley inside the sample enclosure so the crimper could be handled with manipulators. The sample tube with the conoseal gaskets in place is picked up with the manipulators and put in place on the sampler (Figure 4.4). While the tube is held in place with one manipulator, the nuts are tightened down with a wrench fitted with grips for the manipulator jaws (Figure 4.5). A vacuum is then pulled on the sample tube with a helium leak detector and the joints sprayed with helium from a hose held by the manipulator. If no leak is detected, the oven door is closed and latched, the oven is brought up to 300 °F, and sodium flow is started. At the end of the sampling run the outlet valve is shut off, and the oven door opened and the crimper brought into place at the outlet end of the removable section (Figure 4.6). The sample tube is crimped next to the fitting and then again several inches away (Figure 4.7). The guillotine is then moved to the inlet end of the removable section and it is crimped about 3 in. from the end and then a second time next to the inlet fitting. The inlet valve is then closed and the quench turned on. Less than 2 min are

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FIGURE 4.4. Placing Sample Tube

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FIGURE 4.5. Fastening Sample Tube

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FIGURE 4.6. Crimping Sample Tube

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FIGURE 4.7. Second Crimp of Sample Tube

consumed in the crimping so the temperature does not drop more than 100 °F. When the sample tube and inlet and outlet lines have cooled to below 200 °F, the conoseal nuts are removed with the manipulator operated wrench. The sample tube is removed with the manipulators by pushing down and pulling outward to break the solidified sodium column. Figure 4.8 shows a crimped sample tube after removal. The procedure has proved to be simple.

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FIGURE 4.8. Sample Tube Crimped

A total of 24 samples was taken after the cooling profile studies had been completed. The first 10 runs were made at a sodium temperature of 600 °F and a cold trap temperature of about 350 °F. Loop stability was poor during the sampling period and the oxygen content of the sodium varied from 4 to 14 ppm. However, measured values along the length of individual samples were consistent and showed no longitudinal segregation of oxygen. The sodium temperature was raised to 800 °F and the cold trap temperature to 400 °F. After eight samples were taken, the cold trap was raised to 440 °F and six samples were taken. The inlet, outlet, and center section of each sample were analyzed for oxygen and one section for carbon. At the time each sample was removed, a plugging run was made by the loop operator. The following table presents the results:

TABLE 4.2. Oxygen Measurements on Samples from Remote Sampler

Sample	Cold Trap Temperature, °F	Oxygen, ppm			Carbon, ppm	Plugging Run Oxygen, ppm
		Inlet	Center	Outlet		
11	400	19	17	17	5	11
12	400	28	26	25	5	17
13	400	24	24	22	6	19
14	400	24	25	26	4	16
15	400	25	23	23	7	18
16	400	24	25	25	4	17
17	400	32	27	28	4	17
18	400	24	21	21	4	19
19	440	30	32	25	2	25
20	440	27	27	26	4	24
21	440	29	27	29	4	24
22	440	29	28	28	4	24
23	440	32	30	35	4	22
24	440	28	-- (a)	-- (a)	5	25

a. Not analyzed.

These 14 samples were taken over a period of 16 working days (March 31-April 21) which demonstrates the ability for repeated sampling. The analytical results show no noticeable longitudinal segregation of oxygen under the sampling conditions used.

Because of the experimental nature of the program sodium leaks have occurred on five occasions since the start up of the sampler. These varied from an estimated 3 to 500 g of sodium. In all but one case the conoseal joints had been helium leak checked before sodium flow started. In three of the leaks, including the two largest, no fire occurred. About 2 to 3 g from Sample 24 leaked and burned, and when flow was started on the next sample, about 10 g leaked and burned. The use of the nitrogen quench controlled the fire in the oven and any fire outside of the oven was easily smothered in the vermiculite in the sampler catch tray or with a helium jet. All sodium oxide smoke was contained in the sampler enclosure and removed with the exhaust fan and its filter. The sampler was shut down after the last leak, and construction of a smaller sampler compatible with the latest FFTF size restrictions was begun.

Failure analyses investigation of the last two incidents indicated that the gaskets had apparently not sealed properly (Figures 4.9 and 4.10). This might have been caused by the galling of the threads of the conoseal joint which resulted in the nuts not being cinched down as tightly as they were thought to be. A more probable cause is the erosion of the gasket seating area by the hot sodium or sodium oxide. The female Conoseal ends have been used while some 30 samples have been taken and have been thermocycled violently a number of times. Additional studies are required before firm conclusions regarding connector life expectancy can be reached, but these results may foretell a life expectancy on the order of 50 samples per connector. This would permit 2500 samplings with the present sampler design before any welding was required.

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FIGURE 4.9. Outlet End of Leaky Sample Tube

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FIGURE 4.10. Leaky Sample Tube

SODIUM SERVICE ANALYSIS

R. F. Keough and M. B. Hall

The objective of this activity is to provide analytical support for existing PNL sodium technology programs.

Procedures and equipment for the determination of oxygen, total carbon, hydrogen species, total hydrogen, cyanide, ----- Kjeldahl nitrogen, and trace metals in sodium have been in place for some time and have provided service analysis during the report period. A laboratory manual describing these procedures has been compiled and issued. The more difficult procedures are continually under review and modification to improve their performance, and new procedures are being added to meet the expanding needs of the sodium technology programs. A program is underway to standardize laboratory methods for sodium analyses.

Plutonium Laboratory

The installation of all major equipment and services has been completed. Attempts to make the old U.S. Dynamics purification train usable have been abandoned, and purchase of a replacement train has been initiated. Temporarily, both inert atmosphere glove boxes are being served by one Vacuum/Atmospheres train and maintained at about 1 ppm of water and oxygen.

The oxygen removal agent in this purification train is a copper film on what appears to be an alumina or silica gel support. Since mercury vapor from the glove boxes could poison this agent, performance characteristics of the train were established (Figures 4.11 and 4.12) at start-up and will be re-examined periodically to detect any deterioration in performance. ----- The oxygen determination by amalgamation procedure has been established in this laboratory and a number of routine samples have been run in both this and the service laboratory for intercomparison. Results agree within the precision of the method.

FIGURE 4.11. Oxygen Content of Glove Box During a Constant Inward Leak of 8 ml/min of Oxygen

FIGURE 4.12. Recovery of Glove Box from the Addition of 40 ml of Oxygen

Standard Analytical Methods ProgramVacuum Distillation for Trace Metal Determination

A procedure for the determination of trace metals in sodium by vacuum distillation and atomic absorption analysis of the distillation residue has been set up and evaluated. The procedure and apparatus (Figure 4.13) are similar to the ones of Ramachandran and Hareland.⁽²⁾ A number of metals, dissolved in mercury, were added to sodium and determined in the residue after vacuum distillation. Recoveries (Table 4.3) were similar to what the previous workers found using a different standard preparation technique.

TABLE 4.3. Recovery of Trace Metals After Vacuum Distillation of Sodium

<u>Metal</u>	<u>Percent Recovery</u>
Calcium	79-115
Copper	98-105
Lithium	83-101
Magnesium	93- 99
Zinc	2- 4

In another recovery experiment, chloride solutions of a number of metals were evaporated in tantalum planchets, placed in a distillation cup, then covered with sodium which was subsequently removed by distillation. The residue on the planchet was analyzed separately from the cup contents and the results (Table 4.4) indicate that there was little or no dissolution of the standards by the sodium. The most reasonable explanation for failure of at least the more soluble metals to move into the sodium is that the assumed reduction of chlorides,

did not occur because of the presence of some residual water of hydration which would permit the formation of insoluble

FIGURE 4.13. Apparatus for Vacuum Distillation of Sodium

oxides rather than free metals:

This would not account for the failure of copper to move into the sodium since it does not form an oxide in sodium.

TABLE 4.4. Recovery of Traces of Metals Added as Chlorides to Sodium

Metal	Percent Recovery	
	Planchet	Cup
Calcium	96	<1
Chromium	98	0
Cobalt	102	0
Copper	98	4
Iron	Most	<10
Magnesium	99	0
Manganese	97	0

In a third type of recovery test, several milligrams of nickel and copper powders were added to sodium and carried thru the vacuum distillation procedure to determine if there was any physical loss by spattering from the cup. Analysis indicated that $97 \pm 2\%$ of both metals remained in the cup.

Conclusions which have been reached are:

- 1) Simple apparatus is required for distillation.
- 2) Up to 75 g of sodium can be distilled in about 3 hr.
- 3) Sodium in the residue may be kept as low as 0.5 mg without undue effort to protect the sample from the air.
- 4) Copper, magnesium, lithium, calcium, and the primary stainless steel metals can readily be determined on the residues at the 0.1 ppm level by atomic absorption or flame emission spectrometry.
- 5) Other metals such as aluminum, vanadium and titanium, although recovered by vacuum distillation, have poor detection limits and could be determined advantageously by colorimetric, emission, or mass spectrographic techniques on the distillation residue.

- 6) The results of Ramachandran and Hareland⁽²⁾ have been generally confirmed.

Oxygen Determination

The interim standard method for the determination of oxygen by amalgamation proposes three modifications to the procedure presently used by PNL. Two of the suggested modifications have been examined: cooling the mercury prior to amalgamation and special drying of the amalgam vessels (separatory funnels). Several approaches to each of these modifications have been made. In no case was a significant improvement made by cooling the mercury, however drying the funnels in the inert atmosphere glove box at high temperatures did reduce the blank by a factor of 2 to 3 (Table 4.5). It does not appear desirable or practical to incorporate this change into our routine procedure at this time because the small improvement in precision does not warrant the effort.

TABLE 4.5. Effect of Drying Amalgamation Vessels in Argon on the Oxide Blank

<u>Run</u>	<u>Drying Time</u>		<u>Temp., °C</u>	<u>Oxide Found, µg</u>	
				<u>Dried</u>	<u>Control</u>
1	10	min	500	2.2	10.0
2	30	min	400	5.2	7.0
3	16	hr	450	3.1	6.4
4	3.5	hr	450	2.0	6.5
5	16	hr	450	3.8	8.6
6	2	hr	450	1.7	7.0

A third modification, extrusion sampling, will be examined in the next report period.

Flame photometric and titration methods were compared for measuring the sodium oxide after amalgamation (Table 4.6). Each number in the table represents the average of triplicate amalgamations of a given sample. Titration values appear to

be slightly lower than flame photometric values on the low oxygen samples. No effort has been made to determine the cause of the discrepancy.

TABLE 4.6. Comparison of Methods for Measuring Sodium Oxide in Amalgamation Residues

<u>Sample</u>	<u>Oxide in Sodium, ppm</u>	
	<u>Flame</u>	<u>Titration</u>
1	20.0	17.7
2	26.7	25.0
3	22.8	23.8
4	24.9	24.8
5	26.5	29.0
6	4.5	3.7
7	3.6	3.4
8	5.9	4.4

Hydrogen Determination

The apparatus for hydrogen determination by the proposed interim standard method has been set up. The reflex vessel is inside the glove box for plutonium contamination control.

Laboratory Manual

Since the previous issue of the manual, a number of significant changes have been made and new procedures have been written. The manual is being revised to reflect these changes.

FFTF Analytical Support Facilities

W. O. Greenhalgh

The objective of this activity is to establish the extended analytical capability needed to support the fast flux test facilities and the sodium technology programs at PNL.

The FFTF test and sodium technology program will require a broad analytical support base. Use of the best available

technology will be required to ensure that the test facilities are operated with FFTF quality sodium and to aid in the interpretation of the effects of sodium quality on test results. Several published analytical procedures will be required in addition to those already in place. Two such procedures are those for particulate identification and carbon species measurement.

The Analysis of Sodium Particulates

The aqueous insoluble particulates of several CGEL samples have been examined with the Unitron Metallograph. The population densities of particles greater than 2 microns in diameter estimated from actual field counts are given below in Table 4.7.

TABLE 4.7. Population Densities of Particles in **CGEL** Sodium Samples

<u>Sample</u>	<u>Population Density, particles/g</u>	<u>Loop Temp., °C</u>
CGEL-fill	1000	~150
5-009	500	320
5-010	1600	350
5-011	400	370
5-012	1900	410
5-016	1800	410
5-017	1400	440
5-018	800	470
5-020	2700	530

There is considerable scatter in the population density from sample to sample, probably due to the non-ideal behavior of particles. There is some indication of a pattern with respect to loop operating time, with a slight decrease in the number of aqueous insoluble particles in the very early stages of loop operation followed by a gradual increase as operating time and temperature are increased.

There is also a shift in the nature of the particles as a function of loop operating time. Particles observed in the early runs were mostly (~75%) metallic bits and pieces. Particles observed included several pieces of what appears to be corroded iron or steel, metallic and nonmetallic threads, nonmetallic crystals, and even one piece that appeared to be copper or brass. Because essentially no fresh filings of stainless steel were observed, the particles investigated were authentic and not contamination from the sampling system. Particles observed in samples taken during the intermediate runs were smaller (10 to 15 microns compared with 10 to 25 microns initially), and only about 50% of them were metallic, 30% oxides and corrosion products, and 20% were miscellaneous crystals and amorphous shapes. Particles observed in the later runs were smaller still (≤ 10 microns) and approximately 30% were metallic, 40% oxides, and 30% miscellaneous material including several white or lightly colored clear crystals.

In other particulate studies a stainless steel filter which had been installed on the by-pass sampler of CGEL was examined by X-ray diffraction and metallography techniques. The 35 micron mean-pore-size filter had 120 gal of sodium pumped through it at 530 °C, but no particles could be detected by either analytical technique. It was hoped that the filter would collect particles of sufficient size that a diffraction pattern could be obtained. Particulate analyses carried out since the filter sample was obtained indicate that very few particles in CGEL would be of sufficient size to be retained by the filter.

Metallography of the sodium upstream from the filter was interesting. Approximately 50 dark particle inclusions were found on the 3/8 in. diameter surface. Many of the inclusions were large, as much as 40 to 50 microns in diameter, but the bulk of them were 5 to 10 microns in size (Figures 4.14 and 4.15).

FIGURE 4.14. Dark Sodium Particulate Impurities (large size) ^{176X}

FIGURE 4.15. Dark Sodium Particulate Impurities (small size) ^{176X}

Randomly distributed metallic crystallites of various sizes were also visible on the sodium surface (Figures 4.16 and 4.17). The population of metallic crystallites was estimated at approximately 1000 crystallites/cm². These crystallites were observable only under polarized light but they did shine through thin oxide or hydroxide layers indicating they were composed of metals such as iron, nickel, or chromium. Efforts to examine particles concentrated in and around filters will continue. A particulate sample collected on a 7 micron stainless steel filter has been prepared and is presently scheduled for examination with the electron microscope and X-ray probe instrument.

Next fiscal year the electron microscope and probe instrument, which has been successfully evaluated for sodium work, will be more readily available for routine analyses. Also, the services of a Particle Measurement Computer System is now available for sizing, counting, and grouping of particulates that can be clearly photographed.

The Analysis of Carbon Species

A Hewlett-Packard Model 700-2419F gas chromatograph with a WX thermal conductivity detector has been substituted as the instrument used for detection and analysis of the evolved gases in the carbon species analysis. Instrument problems with the other gas chromatograph were too frequent for practical operation. The Hewlett-Packard chromatograph is in routine operation and it is working well. The sensitivity is not as good as other instruments but it appears to be sufficient for present carbon levels. The carbon values obtained with the new instrument are comparable with earlier runs on the old instrument.

The carbon species procedure development is nearing completion. Careful handling techniques have solved filter contamination problems which previously interfered with the analysis of carbon solids. Carbonate recoveries remain to be

176X
FIGURE 4.16. Metallic Crystallites (Polarized light source)

176X
FIGURE 4.17. Metallic Crystallites (Polarized light source)

checked out before the analytical procedure is complete. Efforts to check out the carbonate recoveries by using the aqueous hydrolysis method have been complicated because of container corrosion during sodium dissolution. Carbon dioxide is formed by oxidation of container carbon by the hot aqueous hydroxide media. Solution of this problem should be possible very shortly and the development of the carbon species procedure should be finalized in the near future.

COVER GAS SYSTEMS

G. B. Barton and H. P. Maffei

Engineering Systems Support

The objective of this activity is to provide support to FFTF development systems in the area of cover gas analysis and the interpretation of these analyses.

There are a number of planned or existing sodium systems operated in support of the FFTF development program. All must operate with FFTF quality sodium and cover gas to ensure meaningful development information. Cover gas quality, particularly during the initial temperature ramp of a system, must be monitored as part of this assurance.

In-Plenum Hydrogen Meter for Development Program Application

The hydrogen concentrations in the cover gases of many sodium systems have been constantly monitored and reported. These cover gases have been monitored either by grab samples or by pumping a gas sample from the cover gas plenum through small sample lines to an analytical instrument. These methods have never resulted in a quantitative hydrogen analysis. This is due to the scavenging effect sodium has on hydrogen. The hydrogen present in the cover gas is in equilibrium with the sodium hydride in the sodium of the sodium pool and cover gas plenum. During sampling of the cover gas, the cooling of the gas sample changes the equilibrium conditions and results in erroneous hydrogen values.

A detector which could measure the hydrogen concentration within the gas plenum would eliminate the sodium scavenging problem and give a quantitative hydrogen measurement. It was previously reported that such a detector is being evaluated at BNW⁽³⁾ and that the detector employs the principle of hydrogen diffusion through a nickel membrane. The detector employs a nickel tube with an area of 27 cm² and a 10 mil wall thickness. The membrane portion of the tube is heated internally with an electrical resistance heater, while the outside of the tube is exposed to an argon atmosphere. The inside of the nickel tube is purged with argon gas to sweep the diffused hydrogen to a thermal conductivity detector where the hydrogen concentration can be measured.

During the past quarter hydrogen diffusion rates through the nickel membrane were measured. The nickel membrane was maintained at 1400 °F and hydrogen concentrations of 50 to 1000 ppm were used for the diffusion studies. The effect of varying the nickel membrane temperature was also investigated. The measured diffusion rates are shown in Table 4.8.

It can be seen that the hydrogen flux through the nickel membrane increases by approximately a factor of 10 with a 20 fold increase in hydrogen concentration in the argon cover gas. The table also shows that changes in the nickel membrane temperature affect the hydrogen flux by 0.0008 ml/hr/°F or less than 1% for every degree change in temperature.

An expression which describes the diffusion of hydrogen through metal⁽⁴⁾ is

$$\phi = \frac{AKP^{1/2}}{X} \exp \frac{-Q}{R\pi}$$

where ϕ = flux of hydrogen through metal
 K = permeation constant
 P = hydrogen pressure

X = membrane thickness

Q = activation energy

R = gas constant

π = absolute temperature

A = surface area of membrane

TABLE 4.8. Measured Diffusion Rates of Hydrogen Through Nickel Membrane

Hydrogen Concentration in Cover Gas, ppm	Diffused H_2 Concentration in TC Detector, ppm	Purge Gas Flow Rate, ml/min	H_2 Flux, ml/hr	Membrane Temp., $^{\circ}F$
50	22	27	0.035	1400
50	10.5	31	0.020	1400
200	43	33	0.084	1400
200	47	33	0.092	1400
500	100	27	0.162	1400
1000	190	27	0.308	1400
1000	238	17	0.243	1400
1000	130	39	0.304	1400
1000	180	27	0.286	1400
1000	235	19	0.286	1400
1000	158	29	0.274	1400
1000	285	18	0.307	1400
1000	375	12	0.270	1400
1000	390	12	0.280	1400
1000	232	26	0.379	1400
1000	218	28	0.366	1400
1000	195	28	0.321	1400
1000	158	33	0.311	1400
1000	190	27	0.308	1400
1000	148	27	0.239	1200
1000	95	27	0.154	1000

The equation predicts a straight line for a plot of $\log \phi$ versus $\log p^{1/2}$. A plot of the data in the above table is

shown in Figure 4.18. The pressure terms have been corrected for the particular geometry of the nickel membrane. The experimental values follow the straight line closely.

The sensitivity of this type of hydrogen detection is limited by the type of sensing device used for measuring the diffused hydrogen. Figure 4.19 shows the response of the thermal conductivity detector to various hydrogen levels in the argon cover gas. With the ability to read two chart division accurately, the lower detection limit appears to be between 10 and 20 ppm of hydrogen in the cover gas.

ANL is developing an in-plenum hydrogen monitor based on the diffusion of hydrogen through a nickel membrane into a vacuum.⁽⁵⁾ The quantity of hydrogen diffusing is related to the system ion pump current. A pump has been purchased and plans are underway to compare the two measurement techniques. An instrument based on the most promising technique will be installed on the SCEL.

Selection and Testing of Cover Gas Instruments and Sampling Systems

The objective of this activity is to provide proven sampling and analysis equipment and technology for the cyclic on-line monitoring of impurities, including fission gases, in the FFTF sodium cover gas.

Evaluation of mass spectrometric and gas chromatographic equipment suitable for monitoring impurities in FFTF sodium cover gas has been completed and the gas chromatographic approach selected as best suited. This decision has shifted emphasis in the development effort to the sampling area and the solution of the sodium vapor removal problem.

FIGURE 4.18. Plot of Hydrogen Permeation Through Nickel

FIGURE 4.19. TC Detector Response to Hydrogen in Cover Gas

Sodium Vapor Trap Design Study

The report detailing the bases of the current refluxing vapor trap design has been rewritten and is in the final editorial review. Several vapor traps have been constructed based on the design and are undergoing laboratory evaluation. Traps have been constructed and a long term performance testing program on an engineering system is about to begin. The primary objective is to determine whether or not sodium compounds will plug the vapor trap or will degrade its performance. These tests are to be conducted on the CGEL system.

Tests are to be run with air and moisture added to the argon cover gas. The first tests are planned with 500 ppm of each impurity in binary mixtures with argon. In addition to measuring trap efficiency, these tests will determine if the sodium compounds such as the oxide and hydride remain in the trap or reflux back to the sodium pool. The exit gas will also be analyzed to determine the effect of the vapor trap on the impurity concentration and to obtain a measure of the validity of the analytical sample taken through a vapor trap of the new design.

Past studies have indicated that hydrogen concentration is perturbed by the vapor trap. Equipment has been set up to study this problem on the other gaseous impurities of major interest (methane, nitrogen, oxygen, and carbon oxides). Sodium trapping performance has been checked to verify the adequacy of the trap prior to beginning the trap gettering tests.

Selection of FFTF Cover Gas System Sampling Flow Rate

The current FFTF cover gas system design has a primary gas space in the reactor of about 320 ft^3 , a system to recirculate this gas at about $30 \text{ ft}^3/\text{min}$ (standard) and three primary sodium circulating pump gas buffer spaces of about 275 ft^3 .

The gas spaces are interconnected by relatively large diameter pipes and have a purge gas flow into each system of about $0.5 \text{ ft}^3/\text{min}$ (standard). The total purge flow (about 2 ft^3 per min) exists to the radioactive gas handling system.

The analytical system used to monitor the impurity levels in the cover gas system should respond rapidly enough to follow changes in the system. A mathematical analysis has been made to provide an estimate of the response time required. The study assumes the need to detect and follow the impurity concentration levels resulting from the introduction of a sudden constant rate leak. The equation for the approach to equilibrium concentration can be expressed as:

$$C = C_o e^{-Ft} + C_i (1 - e^{-Ft}) \quad (1)$$

where C is the concentration at time t

C_o is the initial concentration

C_i is the inlet impurity concentration in the gas-leak tanks expressed as concentration per unit purge inflow

F is the dilution factor, purge flow rate/volume of gas space, normalized for temperature and pressure effects.

The fractional approach to equilibrium at various times has been calculated for the above conditions and is presented in Table 4.9. Column 1 assumes the impurity introduction and measurement in the heat transfer system pump space and Column 2 assumes impurity introduction directly into the reactor gas circuit. Column 3 assumes introduction of the impurity into the pump space and measurement in the reactor space. Under these conditions Equation (2) is applicable. A basic assumption that must be made is that each gas space is well mixed.

$$C_{R+M} = \frac{C_i F_H}{F_r} + \frac{F_H(C_o - C_i)}{(F_R - F_H)} e^{-F_H^+} + e^{-F_R^+}$$

$$\left[C_o - \frac{C_i F_H}{F_R} - \frac{F_H(C_o - C_i)}{(F_R - F_H)} \right] \quad (2)$$

It can be seen that the approach to equilibrium in any case is very slow. This is because of the large dilution factors involved. The calculations indicate that it may be possible to obtain considerable information about the location of the leak by measuring the rate of approach to equilibrium in cases where only a single leak is involved. Sampling and analysis must occur at a rate and frequency which will permit the detection of a leak and the monitoring of significant impurity concentration changes. Current system design calls for a chromatographic analysis every 10 min. The data in Table 4.9 indicate that this will be more than adequate in view of the inertia of the system.

TABLE 4.9. Fractional Approach to Equilibrium Concentration

<u>Time, min</u>	<u>C_H</u>	<u>C_R</u>	<u>C_{R+H}</u>
1	0.005	0.035	0.000044
10	0.049	0.3003	0.004
25	0.1175	0.5905	0.0232
50	0.2212	0.8323	0.0776
100	0.3935	0.9719	0.2228
200	0.6321	0.9992	0.500
300	0.7769		0.6919
500	0.9179		0.8860
1000	0.9933		0.9906

An additional mathematical analysis of the system is required for the case of fuel element rupture detection. A single burst release of impurity in the reactor plenum must be assumed. Under these conditions the rate of mixing in the gas space becomes the limiting variable. An exact calculation of this mixing rate is not possible because of the large number of variables involved. However, one turn-over time may be used as a first approximation of the mixing time. Current FFTF design calls for a turnover time of approximately 4 min (approximately 320 ft³ of gas at 858 °F and a 30 ft³/min pump rate). The sample system hold up to the fission gas detector should therefore be on the order of one minute to eliminate this as a possible bottle neck in leak detection.

FUEL FAILURE MONITORING SYSTEM (TASK FS-B)

R. J. Cash

The objectives of this task are to evaluate, develop, and test several Fuel Failure Location (FFL) subsystems for application to the FFTF Reactor System. Five concepts are presently under study. They are: (1) xenon and krypton gas tagging of individual fuel pins, (2) in-vessel gas separation from sodium sampled above each subassembly, (3) conductivity probe monitoring of released gas volume in each subassembly instrument package, (4) fission product deposition on removable tabs located above each subassembly, and (5) acoustical triangulation of the three reactor outlet pipes for released fission gas.

Present development work places the major emphasis on the gas tagging concept, but further FFTF cores may be vented for release of gaseous fission products during normal operation or may be operated with "gas leaker" fuel failures. Under these circumstances the gas tagging concept could not be functional and an alternate system would be necessary for fuel failure location. It is therefore essential that

development of promising alternates also be pursued. The present development schedule⁽⁶⁾ calls for evaluation of the five concepts in FY 1970 and early FY 1971 with an evaluation board selection of the location reference system and alternate backups in September 1970.

Work was officially started on evaluating the five concepts at the end of March. A work plan has been completed outlining development and feasibility testing to be conducted with water and sodium mockups on each of the various concepts for FY 1970 and FY 1971.

Xenon and Krypton Gas Tagging Concept

R. J. Cash and T. J. Kabele

The purpose of this phase of the development effort is to evaluate gas tagging as a Failed Fuel Location subsystem for the FFTF. In this concept approximately 1 cm³ of mixed isotopes of xenon, krypton or both are sealed in each fuel pin as part of the helium fill gas. The same isotopic ratio is used in each pin for a given subassembly with different ratios in different subassemblies. When a fuel pin fails, part or all of the tag gas is released to the sodium coolant and in turn disengages into the argon cover gas. A sample of cover gas is then taken and passed through a charcoal bed for adsorption of the xenon and krypton tag gas. Later, the tag gas is directed into a mass spectrometer to determine the unique tag ratio and hence identify the faulty subassembly.

C. A. Mansius specified 80 unique preliminary tags using $^{129}\text{Xe}/^{124}\text{Xe}$ isotopic ratios. These ratios are listed in Table 4.10. They vary from 6/1 through 220/1 and were spaced at a 3% differential. The ratios were rounded up to the next whole number for convenience since they are only preliminary. R. E. Schenter of Theoretical and Neutron Physics recalculated cross section values for xenon isotopes 124, 126, 128, and 129

and krypton isotopes 78 and 80. Based on the new calculations, the differential burnout between ^{124}Xe and ^{129}Xe using the "most likely" value cross sections is about 1% for the average burnout in the gas plenum region at goal exposure. When ^{124}Xe burnout using the "most likely" value is compared with ^{129}Xe burnout using the "probable maximum" value, the differential burnout is about 3%. These calculations do not include the effect of neutron-streaming, but since 60% of the burnout occurs within the lower 1/3 of the plenum region where the effect of streaming is small, the overall streaming effect would not be more than 10% of the values stated (i.e., 1.1% or 3.3% burnout, respectively). ^{126}Xe burnout is much less than either ^{124}Xe or ^{129}Xe (highest of all). Using $^{129}\text{Xe}/^{126}\text{Xe}$ ratio; creates a 2.5% and a 4% burnout at goal exposure for the "most likely" and "probable maximum" cross section values, respectively.

TABLE 4.10. $^{129}\text{Xe}/^{124}\text{Xe}$ Isotopic Ratios for 80 Distinct Tags

Tag No.	$^{129}/^{124}$ Ratio	Tag No.	$^{129}/^{124}$ Ratio	Tag No.	$^{129}/^{124}$ Ratio	Tag No.	$^{129}/^{124}$ Ratio
1	220	21	120	41	61	61	25
2	214	22	116	42	59	62	24
3	208	23	112	43	57	63	23
4	202	24	108	44	55	64	22
5	196	25	105	45	53	65	21
6	190	26	102	46	51	66	20
7	184	27	99	47	49	67	19
8	179	28	96	48	47	68	18
9	174	29	93	49	45	69	17
10	169	30	90	50	43	70	16
11	164	31	87	51	41	71	15
12	159	32	84	52	39	72	14
13	154	33	81	53	37	73	13
14	149	34	78	54	35	74	12
15	144	35	75	55	33	75	11
16	140	36	72	56	31	76	10
17	136	37	69	57	29	77	9
18	132	38	67	58	28	78	8
19	128	39	65	59	27	79	7
20	124	40	63	60	26	80	6

The ^{78}Kr and ^{80}Kr isotopes have low cross section values and also values quite close together. Therefore, burnout is not expected to be a problem if $^{80}\text{Kr}/^{78}\text{Kr}$ ratios are used. Their use in combination with xenon is contemplated to greatly increase the number of useable tags and thereby keep the cost of the tagging materials to a minimum. Cross sections for the xenon and krypton isotopes are given in Table 4.11. Table 4.12 lists burnout fractions for ^{124}Xe and ^{129}Xe after 90, 180, and 270 days of operation. (7)

TABLE 4.11. Calculated Tag Isotope Cross Sections⁽⁷⁾

Isotope		Bottom Plenum Region σ , barns	Middle Plenum Region σ , barns	Top Plenum Region σ , barns
^{78}Kr	Most Likely	0.36	0.398	0.44
	Probable Maximum	1.29	1.61	1.89
^{80}Kr		0.40	0.468	0.542
		1.21	1.53	1.82
^{124}Xe (a)		1.62	2.20	2.82
		2.34	3.16	3.99
^{126}Xe		0.397	0.43	0.46
		0.952	1.15	1.32
^{128}Xe		0.281	0.30	0.324
		0.640	0.77	0.88
^{129}Xe (a)		2.26	2.79	3.27
		3.69	4.91	6.08

a. The cross sections for ^{124}Xe and ^{129}Xe from Table 4.11 were used to calculate burnout fractions for the three plenum regions over full power (400 MW) operating periods of 90, 180, and 270 days. This data is presented in Table 4.12 with the top value representative of the most likely burnout fraction and the bottom value the probable maximum burnout fraction.

TABLE 4.12. Burnout Fractions of ^{124}Xe and ^{129}Xe

Plenum Position	Operating Day					
	^{124}Xe			^{129}Xe		
	90	180	270	90	180	270
Bottom	0.019	0.039	0.057	0.027	0.054	0.08
	0.028	0.056	0.083	0.044	0.088	0.013
Middle	0.006	0.012	0.018	0.008	0.016	0.023
	0.009	0.018	0.026	0.014	0.027	0.041
Top	0.001	0.002	0.003	0.001	0.002	0.003
	0.0015	0.0028	0.0042	0.002	0.004	0.006

Information from EBR-II and knowledge of mass spectrometry indicate that presently, a single channel mass spectrometer can only identify the higher tag ratios with an accuracy of ± 1 in the third significant figure (i.e., ratio is 220 ± 1). A dual channel mass spectrometer may be capable of a greater accuracy.

With the idea in mind that 3% or slightly greater change in the ratio could occur from burnout with goal exposure, four cases of selecting 80 unique tags were examined for quantities of gases required and their overall cost. The assumptions made in each case are outlined below:

Case 1: Using 80 Unique Xenon Tags (No Krypton)

The xenon tags used are those specified in Table 4.10. As the ratio decreases from 220/1 to 6/1, less of the natural xenon is used and more of the enriched 5% and 10% ^{124}Xe is required. The cost of the tag increases approximately in proportion to the actual enrichment. Required volumes and costs are shown in Table 4.13.

Case 2: Using Xenon and Natural Krypton

Case 2 assumes that the 3% xenon ratio spacing in Case 1 is sufficient for burnout effects and attempts to use only the first 40 tags, the less enriched ones, listed in Table 4.10.

To make the 80 unique tags, the 40 tags are used first without krypton and then again with natural krypton as an additive. The added amount of stable krypton found in the reactor cover gas after a failure in one of the latter 40 tagged subassemblies should be easily distinguished from background krypton.

TABLE 4.13. Gas Requirements Per Core

Enrichments	Cost/ml	Volume, liters			
		Case 1	Case 2	Case 3	Case 4
Natural Xenon	\$ 0.06	30.0	30.0	31.0	32.0
1% ^{124}Xe Enriched	1.05	2.3	4.6	3.4	2.6
5% ^{124}Xe Enriched	7.70	0.077	0.15	0.11	0.075
10% ^{124}Xe Enriched	17.40	2.5	0.025	0.53	0.95
Natural Krypton	0.05	0	18.0	14.0	10.5
1% ^{78}Kr Enriched	1.50	0	0	7.9	9.6
8% ^{78}Kr Enriched	3.50	0	0	0	4.4
Total Cost Per Core(a)		~\$24,000	~\$4,500	~\$14,000	~\$26,000

a. At arriving at the costs, it was assumed that 1 cm³ (standard) of xenon gas mixture and 1 cm³ (standard) of a krypton gas, when used, would be placed in each fuel pin without any gas wastage (80 subassemblies of 217 pins each).

Case 3: Increasing Xenon Tag Spacing and Using Enriched Krypton

This case considers the possibility that the spacing between the 80 unique tags is not sufficient for reliable identification over the lifetime of the fuel. Therefore, every other xenon tag in Table 4.10 is a candidate. Selection of the xenon and krypton tags involves a cost optimization procedure. After using the first 29 odd numbered xenon tags, first without then with natural krypton (giving 58 unique tags), it becomes more economical to use most of these same xenon tags

(first 22 odd) over again with enriched krypton. The gas volumes and costs are shown in Table 4.13.

Case 4: Every Third Xenon Tag and Enriched Krypton

Similar to Case 3, the tag spacing is increased further by using every third tag and again optimizing at the point where it becomes more economical to use the commercially prepared enriched krypton gases. No attempt was made to mix the commercially prepared 1% and 8% krypton enrichments to intermediate enrichments. The volumes and costs are shown in Table 4.13.

All costs and tag selections should be considered preliminary. However, even in the extreme cases of 1 and 4, the tag material cost per core is a small fraction of the total estimated fuel pin fabrication cost. Final tag material costs will be dependent on the contract gas prices, the number of unique tags, the mixing and loading processes (gas wastage), and results of the tagging development programs (final loading volume).

In the United States the only producer of enriched xenon and krypton is the AEC's Mound Laboratory. The amount of gas required (including an estimated 100% wastage) for the first two cores of the FFTF would take about 6 months for Mound Laboratory to produce. The natural gases are readily available. Mixing of the unique tag ratios, on the other hand, may require an additional 6 months. If FFTF fuel fabrication is to commence early in CY 1972 contract negotiations must be started for tag gas procurement in the Fall of 1970.

A major uncertainty for the gas tagging concept is the problem of possible entrainment and carry-out of the gas released by a failed pin. If sufficient quantities of the tag gas are entrained in the sodium or transported out of the reactor vessel without disengagement, identification of the

tag ratio may be impossible. The present cover gas design has a gas recirculation rate of $30 \text{ ft}^3/\text{min}$ with a $2 \text{ ft}^3/\text{min}$ side-stream of fresh argon for seal integrity. This $2 \text{ ft}^3/\text{min}$ purge rate results in an effective half life of the cover gas of approximately 2.8 hr (i.e., every 2.8 hr half of the cover gas is replaced by fresh argon). If disengagement occurs over a long period of time (a few hours to 1 or 2 days), tag ratio identification would be impossible. The French have evidence which suggests that gas release may be very slow, sometimes occurring over a period of days. (8)

A tag release experiment recently conducted under sodium in EBR-II revealed no problem with entrainment. The EBR-II reactor concept, however, is a pool-type and entrained gas can easily disengage at the sodium free surface. During a recent visit to EBR-II, we discussed the possibility of conducting a similar experiment which would measure the gas concentration in the cover gas as a function of time. Preliminary experiments would be conducted to determine the delay time in the cover gas system. Then, a given quantity of tracer gas would be released in the sodium and timed for delay in reaching a gamma detector. In this manner an indication of entrainment should be obtained.

An analytical study of gas transport out of the reactor vessel is being conducted on the FFTF geometry and should be completed by August 1. Carry-out is a function of three variables: (1) bubble diameter, (2) position of release, and (3) sodium flowrate. Any gas transported out of the reactor vessel will probably become entrained in the sodium and be recirculated back through the core. Little disengagement is expected at the three pumps because of the turbulent fluid action and the gas baffle seals built into each pump cover gas space. Hence, entrainment is a significant problem to be considered in the gas tagging concept.

In-Vessel Gas Separation Concept

R. J. Cash and D. R. Dickinson

The objective of this phase of the development effort is to test the feasibility of the in-vessel gas separation concept by hydraulic testing of a Plexiglas mockup. In this concept, the sodium flow from each subassembly is passed through a mixer (the same mixer used to achieve sodium mixing for thermocouple sensing) and past a small diameter sample inlet. Part of the dispersed sodium flow enters the sample line and is carried to the upper guide tube near the sodium to cover gas interface where gas disengagement can occur. Bleed holes below the reactor vessel sodium level permit continuous sodium flow within the sample line. Small fractions (10^{-3} or better) of fission gases released upon failure are collected by natural disengagement in the guide tube. After indication of a fuel failure, each guide tube chamber is sampled for abnormal levels of fission gas. This sampling will probably be done with the reactor shutdown.

Feasibility hydraulic testing of the in-vessel gas separation concept has been initiated. A Plexiglas mockup of a gas separator was designed, constructed, and installed on the loop facilities in the 189D Building. Initially difficulties were encountered in measuring the gas disengaged because the capillary tube for gas measurement was too small to permit water separation from rising gas bubbles. With modifications complete, efficiencies of 1 part per 1000 or better were accomplished using a 1/2 in. diameter inlet in the center of the 3-1/2 in. ID flow channel. The air bubbles were dispersed with the mixer previously developed to achieve uniform temperature distributions for the instrument package.

An isokinetic sample line pickup will also be used to determine the effects of velocity at the pickup on the separation efficiency. The isokinetic pickup will increase the

inlet sample line velocity to that of the main loop flow and should increase the efficiency to that expected from the ratio of the cross sectional areas of the sample line inlet and the main loop (approximately 1 part in 100).

The overall efficiency of the in-vessel gas separation apparatus is also dependent upon the sample line flow rate, since at larger flow rates the smaller bubbles tend to be carried downward in the guide tubes. These bubbles are lost if swept out the overflow outlet which is below the sample line inlet to the guide tube mockup (see Figure 4.20). The test plan calls for testing at main loop flow rates of 225 and 450 gal/min and varying the overflow and air injection rates. Tests will also be conducted using small bursts of helium and analyzing for efficiencies and delay times with a helium leak detector. Graphs of efficiency versus air injection rate and sample flow-rate will be prepared from data obtained from the tests.

Conductivity Probe Monitoring Concept

R. J. Cash and D. R. Dickinson

The purpose of the studies conducted on this concept is to determine the feasibility of locating a failed fuel element by measuring the released gas with a conductivity probe. The conductivity probe operates on the same principle as a liquid level indicator, i.e., by measuring a change of resistance of the probe. (9,10)

Neg 702933-3

FIGURE 4.20. In-Vessel Gas Separation Hydraulic Test Mockup

FIGURE 4.21 was deleted at final proofing

FIGURE 4.22 was deleted at final proofing

FIGURE 4.23 was deleted in final proofing

FIGURE 4.24 was deleted in final proofing

Hydraulic testing of the mockup apparatus for this concept was completed June 4. Several design modifications were necessary to achieve satisfactory operation. The signal from the probe was measured on a high speed visicorder using the circuit shown in Figure 4.25.

Test conditions included runs at main loop flows of 225 and 450 gal/min. The sample line flow rate was varied from 0.3 to 2.6 gal/min and 40 to 50 psig air injection rates from 0.1 to 11.0 cm^3/sec were employed. The efficiency of each run was determined by calculating the ratio of the gas collected at the probe to that initially injected below the vortex generator. Graphs of the efficiencies obtained under these test conditions are shown in Figures 4.26 and 4.27. Bubble sizes at the injection port were 1/4 in. or smaller (Figure 4.28). After passing the vortex generator, the bubbles ranged between 0.004 in. (100 microns) and 0.012 in. (300 microns) in diameter (Figure 4.29).

Figure 4.30 shows the results of Run 34 which consisted of a series of 0.45 ml bursts in air spaced several seconds apart. Event markers indicate the release of air bursts at the point of injection. Each minor chart division represents 1 sec, and a horizontal deflect of 1 in. (10 minor divisions) corresponds to a change in water level of about 0.16 in. in the range of normal operation (i.e., near the background level). The efficiency for this run was about 50% at 225 gal/min loop flow and a 1.0 gal/min sample flow. This run defines the probe sensitivity for a slow leaker-type fuel failure and demonstrates that bursts of gas of less than 0.5 cm^3 can be detected. A large volume gas release is also easily detectable

FIGURE 4.25. Conductivity Probe Circuitry Schematic

FIGURE 4.26. Conductivity Probe Mockup Efficiency as a Function of Sample Flowrate

FIGURE 4.27. Conductivity Probe Mockup Efficiency as a Function of the Air Injection Rate

Neg 702755-3

FIGURE 4.28. Air Bubble Shapes and Sizes Injected into Thermal Hydraulics Loop Upstream of Vortex Generator at 450 gal/min (10 squares per 1/2 in.)

Neg 702755-7

FIGURE 4.29. Air Bubble Distribution Downstream of Vortex Generator Before Reaching Pickup Slots at 450 gal/min (10 squares per 1/2 in.)

Neg 702868-1

FIGURE 4.30. Visicorder Trace of Run 34 for Individual Bursts of ~ 0.5 cm³ (Event markers show time of air release upstream)

since the gas causes flooding of the probe by a blanket of air which registers on the recorder as a continuous deflection of several inches above the normal background reading until the burst passes.

The preliminary feasibility tests on this concept were ~~successful and show the conductivity probe to be a very promising~~ FFL device. Additional data and a detailed report on the concept are being readied for distribution.

Fuel Failure Location by Fission Product Deposition

W. F. Brehm and R. J. Cash

The purpose of this task is to investigate the feasibility of a method of fuel failure location to complement gas-tagging. The basic concept is simple: fission products released from failed fuel elements deposit on tabs located above the fuel pins in the flow channel. The tabs are removed and gamma scanned; those tabs with adhering fission products indicate the leaking subassemblies.

Candidate Nuclide

A fission product suitable for leak location must be a gamma-emitting, fairly long-lived, nonvolatile nuclide which is a decay product of a fission gas nuclide. Any intermediate nuclide in the decay chain must have a short half life. The most likely candidates are: ^{137}Cs , ^{140}Ba , ^{144}Ce . pertinent information about each isotope is shown below:

Nuclide	Half Life ⁽¹¹⁾	Parents ⁽¹¹⁾	Inventory of Parent Xenon Isotope, curies per pin at shutdown ⁽¹²⁾
^{137}Cs	30 years	^{137}Xe	1200 ^(a)
^{140}Ba	128 days	$^{140}\text{Xe}, \text{Cs}$	285 ^(a)
^{144}Ce	285 days	$^{144}\text{Xe}, \text{Cs}, \text{Ba}, \text{La}$	10 ^(a)

a. Based on 300 days at 4×10^{15} nv.

Candidate Tab Materials

The depositing isotope must adhere to the deposition tab and the tab itself must be stable in flowing sodium. Nonactivating* materials (there will be about a 10^{14} neutron flux at the probable tab location) would be helpful but may not be absolutely necessary.

Thus far we have chosen ZrO_2 on Zr, TiO_2 on Ti, electropolished stainless steel, and "passivated" stainless steel as candidate tab materials. The ZrO_2 is reported to be relatively inert to sodium and has been considered as a "trapping" material for ^{137}Cs ; ^(13,14) the TiO_2 should be chemically very similar to ZrO_2 , with the added advantage that the Ti will not be activated. Vanadium oxide on vanadium could be another attractive tab material as vanadium does not activate. Passivated stainless steel has been reported to preferentially trap cesium. ⁽¹⁵⁾ (No claim is made that the chosen tab materials and nuclides are the only ones that can be used.)

Counting Considerations

The amount of ^{24}Na contained in the sodium that will adhere to the tabs after their removal will overwhelm the signal from any deposited fission products. Therefore, sodium will have to be removed from the tabs before counting. It may be necessary to dissolve both the sodium and surface of the tab and do a chemical separation to isolate the nuclide of interest. This procedure is possible for ^{137}Cs . ⁽¹⁶⁾ If the tab material itself is radioactive, these operations must be done remotely. (See "Operational Considerations" below.) Cobalt, chromium, and manganese corrosion product isotopes will also be found on the tabs but present no real problem

* *Activate = become radioactive in the neutron flux.*

(a) because of their low concentration (b) a modern multi-channel analyzer can easily resolve the spectrum from the group of isotopes expected. ⁽¹⁷⁾

Operational Considerations

The ideal situation would be a fission product nuclide that would adhere to a nonactivating tab after sodium removal. However, a wide spectrum of operating conditions is possible in FFTF so that less-than-optimum situations may still be useful. This fission product deposition concept is to be used to positively identify leading fuel subassemblies pointed as suspect by gas-tagging or other means. Therefore, in all probability the suspect subassembly and tabs can be removed, and some time will be available for tab retrieval, chemical separation if necessary, and counting. This could possibly be done in a "hot" laboratory in the 300 Area if no remote chemistry facilities are available at FFTF. In this case, activated tab materials and residual ²⁴Na could both be accommodated. It will be advantageous, however, to try to approach as close as possible to the optimum situation of adherent nuclides on nonactivated tabs.

Multiple fuel failures, or continued operation of the reactor with a failed fuel element, will decrease the resolution of the gas-tagging concept and make the deposition concept more valuable as a backup. The deposition concept is also valid for locating "advanced" failures such as the propagation of a "gas leaker" failure into a "sodium logging" failure where fission products are leached from fuel by sodium. Another candidate fission product, not part of a gas-decay chain, would be used.

Additional Problems

The following aspects have not been considered as yet but must be before the fission product deposition concept can

become reality: a simple means to remove the tabs from the flow channel must be provided, probably based on the use of the fuel handling machine. Some provision must be made to ensure that the tabs cannot break loose and hinder coolant flow. It may not be possible to locate a small fission gas "burst" followed by immediate reactor shutdown, by this technique because of the small amount of fission products released.

Experimental Results

Static capsule screening tests of cesium deposition on candidate tab materials were conducted. Candidate tab materials were placed in stainless steel capsules containing a ^{134}Cs -Na mixture. The radioactive cesium was obtained by irradiating a ^{133}Cs -Na mixture in the Hanford K-East reactor, then diluting the mixture in a glove box to the desired source strength. Each capsule contained about 3 mCi of ^{134}Cs , and the total fraction of cesium present in the sodium was about 4 ppm by weight. The radiation level of each capsule was about 170 mR/hr at contact. Figure 4.31 and Table 4.14 give details of capsule construction and specimen preparation.

TABLE 4.14. Tab Specimens

<u>Specimen</u>	<u>Size</u>	<u>Preparation</u>
Electropolished SS	Sheet $3/4 \times 1/2$ in. $\times 60$ mils (a)	Electropolished
Passivated SS	Sheet $3/4 \times 1/2$ in. $\times 60$ mils	Electropolished then passivated in 15% HNO_3 for 30 min at room temperature
TiO_2/Ti	Ti sheet $3/4$ $\times 1/2$ in. $\times 60$ mils	Oxidized 72 hr in 750 °F, 1500 psi steam
ZrO_2/Zr	Zr sheet $3/4$ $\times 1/2$ in. $\times 30$ mils	Oxidized 36 hr in 750 °F, 1500 psi steam

a. Approximate dimensions

CAPSULES - 3/4 in. SCH 40 STAINLESS STEEL PIPE

FIGURE 4.31. Capsule Arrangement for Static Sodium Tests

Capsules were heated for 9.5 and 96 hr, respectively, at nominally 900 and 1050 °F (total of four capsules). Following the test, the capsules were inverted to allow sodium to drain off the specimen tabs. This procedure reduced the possibility that cesium would segregate to the sodium tab specimen interface upon cooling.

Only preliminary counting (gamma spectrometry) could be completed this quarter. The counting showed (1) that measurable (300 counts/min) amounts of ^{134}Cs were deposited on the tab specimens, (2) there was less than a factor of two difference in counting rate among the various tab materials, and (3) the material heated at 900 °F had a slightly higher count rate than that heated at 1050 °F. This latter result would be in agreement with the physical adsorption mechanism for cesium depositing on surfaces in a sodium environment proposed by Cooper and Taylor. (16)

Future Work

It is felt that the work should be extended to the barium and cerium nuclides and into dynamic systems. Both barium and cerium will probably be less mobile than cesium in a dynamic sodium system and may deposit more readily on surfaces near the release site. It is planned to (1) finish detailed examination of the present specimens, (2) expose further specimens to ^{134}Cs -Na mixture in thermal convection and pumped loops, and (3) produce ^{140}Ba and hopefully ^{144}Ce in sodium by irradiation of enriched uranium foil in sodium and rejection of fission products by recoil, to test deposition behavior of the other isotopes.

Acoustical Triangulation Concept for Zonal Fuel Failure Location

R. J. Cash

Development work on the feasibility of the acoustical triangulation concept has just recently been assigned to Task FS-B. This concept while not capable of locating single faulty subassemblies should be studied as a complementary location system. Preliminary tests conducted by J. Spalik and J. J. Regimbal indicate the concept is capable of failure location to within a zone of 5 to 9 subassemblies with a high degree of confidence. The concept also provides a redundant detection function and could possibly be applicable for use with a vented core to locate severe fuel failures.

The concept works on the principle that flow from a given subassembly is strongly biased toward the closest outlet nozzle of the reactor vessel. This will be true for FFTF as long as there is no contrivance above the subassemblies for mixing or deflecting the flow exiting the vessel. Thus, flow from each subassembly should be effectively split with the largest flow moving to the nearest outlet in the shortest time. If the effect is reproducible and capable of being mapped and measured, then it is practical to determine a zonal location based upon synchronous monitoring of released gas bubbles (or delayed neutrons) in the three outlet pipes.

A typical ultrasonic detector for the Hydraulic Core Mockup (HCM) operates at a frequency of 2.25 MHz with the apparatus (transducer) attached to the pipe wall acting as both the transmitter and receiver. The signal is reflected off the inner pipe surface opposite the transducer, amplified, and displayed on an oscilloscope and/or recorded. Bubbles passing the detector attenuate the signal to an extent easily detectable.

Preliminary tests on the Hydraulic Core Mockup (HCM), a 1/3 scale model of the reactor vessel, were described briefly in BNWL-1200-2.⁽¹⁷⁾ An ultrasonic transducer was used to measure controlled releases of bubbles from various positions in the core mockup. A sensitivity of at least 10 ppm bubble concentration was demonstrated. This amounts to about 8.8 cm^3 per sec of gas exiting past an ultrasonic transducer as part of the normal 14,000 gal/min ($8.8 \times 10^5 \text{ cm}^3/\text{sec}$) flowrate in each outlet pipe.

Additional proof-of-principle tests are planned early in FY 1971 on the HCM using three synchronous transducers (one on each outlet pipe) and a three pen, high speed recorder. Various rates of air will be released from given positions in the core mockup to determine the practicality of this concept for the FFTF. By measuring delay times and the amplitude of the signal from each outlet, it will be possible to evaluate the usefulness of this concept. The sensitivity of the method will also be re-evaluated.

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5.0 OXYGEN-SENSING SOLID ELECTROLYTES

FABRICATION

W. A. Ross and P. E. Hart

The interaction between PNL and Zircoa on solid electrolyte tube fabrication continued during the quarter. Two batches of Zircoa powder were calcined in our high purity system, and--- Zircoa fabricated tubes from both batches. Tubes from the second batch (Z-4) will be used as interim tubes for testing in flowing sodium. They are presently being shipped to PNL from Zircoa. Zircoa also fabricated two additional sets of tubes with their own equipment and furnaces. One of these sets (Z-3) was made by the slip cast process illustrated in Figure 5.1 and the other (Z-5) was isostatically pressed (Figure 5.2) using the process suggested by PNL for the final tube fabrication. Tubes from each of the three batches which have been received were characterized in terms of chemical analysis, microstructure, and sodium compatibility. Results of the chemical analysis are summarized in Table 5.1. The values listed are in parts per million by weight and have an estimated accuracy of plus or minus a factor of three. The values listed for isostatically pressed tubes are averages of three analyses. Much of the difference in the analyses stem from processing variations. Starting powders generally contain less than 200 ppm cation impurities. As shown in Figure 5.1, it is necessary to calcine the mixed powders at about 1800 °C to form a solid solution. Before Y_2O_3 is in solution it is quite reactive with SiO_2 , Al_2O_3 , and FeO. Zircoa's refractory oxide furnace contains these impurities to the degree that they are added during calcining or during sintering in the case of isostatically pressed tubes. The PNL furnace uses a graphite resistance element; thus solid solution is achieved without adding large amounts of impurities. In the slip cast process, it is necessary to ball mill the calcined powders for long

FIGURE 5.1. Process Flow Diagram for Solid Electrolyte Slip Cast Tubes

FIGURE 5.2. Process Flow Diagram for Solid Electrolyte Isostatically Pressed Tubes

periods to reduce particle size. The ball milling is performed with MgO stabilized ZrO_2 balls which accounts for the high Mg + Zr content of the slip cast tubes.

TABLE 5.1. Chemical Analysis of 7.5 wt% Y_2O_3 - ThO_2 Tubes Fabricated by Zircoa

Element	Concentration, wppm		
	Slip Cast PNL Calcine	Slip Cast Zircoa Calcine	Isostatically Pressed
Al	7	10	300
Ca	10	100	40
Fe	1	40	300
Mg	20	200	26
Si	5	70	300
Zr	1000	5000	120

It appears that for Zircoa to fabricate higher purity materials it will be necessary for them to obtain a graphite resistance or refractory metal-furnace.

Work has proceeded at PNL to develop a firing method for the full size tubes. The Centorr furnace from Argonne arrived during June. The furnace had been used as a reaction vessel and requires some modifications, which have begun, before it can be used to sinter the full size tubes. A power controller-programmer is being procured for the furnace. Equipment installation and modifications should be complete by the end of July. An isostatic pressing unit with a long chamber was received from Autoclave Engineering. Its intended use will be for PuO_2 - UO_2 fabrication, but it will be used to fabricate ThO_2 - Y_2O_3 tubes before it is committed to PuO_2 .

The tube design was modified to provide for tubes 4 in. long and approximately 0.250 in. diameter instead of the current 8 in. long, 0.500 in. diameter design. The shorter tube should make fabrication simpler. Some development will be necessary to fabricate the new dimensions.

Work will continue to establish the maximum impurity content which can be tolerated without compromising compatibility with liquid sodium. Pellets fabricated from powder which contained impurities listed in Table 5.2 were attacked by sodium in the usual 800 °C test. This powder had been prepared in a stoneware ball mill by milling 4 hr with MgO-stabilized ZrO₂ balls.

TABLE 5.2. Chemical Analysis of 7.5 wt% Y₂O₃-ThO₂ Powder Prepared in Large Ball Mill

<u>Element</u>	<u>Concentration, ppm</u>
Al	300
Ca	40
Fe	20
Mg	40
Si	60
Zr	100

ELECTROLYTE - SODIUM COMPATIBILITY

L. R. Bunnell

Sodium compatibility testing of solid electrolyte tubes fabricated under subcontract by Zircoa has continued as they become available. The tubes were designated Z-2, Z-3 and Z-5 at PNL (Z-1 was tested during the previous quarter). A brief description of the fabrication variables represented is elsewhere in this report. All testing was done at 800 °C, 100 hr, as in most earlier work; both oxygen-saturated sodium and that containing less than 10 ppm oxygen were used on each tube. Results are below with references to pertinent micrographs.

Z-2: In oxygen-free sodium, there was little cracking, see Figure 5.3. In oxygen-saturated sodium, however, gross cracking occurred, as shown in Figure 5.4. The cracking was along grain boundaries and was confined to the outer surface of the tube.

Neg 4E 179

80X

FIGURE 5.3. Minor Cracking in Tube Z-2, Exposed 100 hr to Clean Sodium, 800 °C

Neg 4E 180

80X

FIGURE 5.4. Extensive Cracking Near Outside Surface of Tube Z-2, Exposed to Oxygen-Saturated Sodium at 800 °C, 100 hr

Z-3: In oxygen-free sodium, Z-3 performed well, even though there seemed to be small amounts of second phase present, confined mostly to a band about 50 microns deep at the outer surface, see Figure 5.5. In addition, small islands of second phase were observed at apparent "triple points," the intersection of three grains. The Z-3 specimen tested in oxygen-containing-sodium, however, performed very badly; grain-boundary cracking occurred through the tube wall. This is shown in Figure 5.6.

Z-5: The three tubes represented minor processing variables (binders added for isostatic pressing). They all behaved badly in both clean and oxygen-saturated sodium. Figures 5.7 and 5.8 are typical of these two cases, respectively.

All of the tubes which behaved poorly contained visible second phase which was apparently responsible for the sodium attack. Its exact origin is unclear, but the firing step in processing is most-suspect.

A PNL-fabricated pellet from a batch which had previously performed well in sodium tests, 18-3,* was subjected to a very stringent test by exposing a polished surface to sodium. The sodium was pure (<10 ppm oxygen), and the test was conducted at 800 °C, 100 hr with encouraging results. Figure 5.9 shows the surface exposed to the sodium. The grain boundaries in a portion of the photo were revealed by cathodic etching prior to the test, and the only visible change was the delineation of polishing scratches. The above should be a very sensitive test and might be applied further, especially if pre- and post-exposure photos of the same area can be obtained for detailed study.

* Batch 18-3 contained 7.5 wt% Y_2O_3 and was fired at 1800 °C, W crucible, ThO_2 setting sand.

Neg 4E 262A

80X

FIGURE 5.5. Second Phase at Edge of Z-3 Tube,
But No Cracking After 100 hr,
800 °C Exposure to Clean Sodium

Neg 4E 263A

80X

FIGURE 5.6. Extensive Cracking in Z-3 Tube
After 800 °C, 100 hr Exposure
in Oxygen-Saturated Sodium

Neg 4E 359

160X

FIGURE 5.7. Extensive Cracking in Z-5 Tube
Exposed to Clean Sodium at
800 °C, 100 hr

Neg 4E 360

160X

FIGURE 5.8. Catastrophic Cracking in 2-5 Tube
After 800 °C, 100 hr Exposure to
Oxygen-Saturated Sodium

Neg 4E 246

400X

FIGURE 5.9. Polished Face of PNL Pellet Exposed to Clean Sodium, 800 °C, 100 hr. The only visible damage is early polishing scratches.

Thoria-yttria samples prepared at PNL are usually fired under highly reducing conditions (graphite furnace, tungsten crucible) in the temperature range 1700 to 2100 °C. The upper end of this range apparently produces a deleterious reaction. Figure 5.10 shows an as-fired pellet (7.5 wt% Y_2O_3 , 2000 °C). Note the bright metallic particles which are not identified as tungsten from the crucible by microprobe. Note also the lamellar growth (arrow) in Figure 5.10; they have the appearance of carbides, but this cannot be verified by microprobe because of the difficulty in detecting light atoms in a heavy matrix. Figure 5.11 shows a pellet from the same batch as Figure 5.10 after heating to 1000 °C in air. Most of the apparent porosity can be attributed to pullout, but the grains which pulled out were probably loosened by a reaction with air, possibly the carbide to oxide conversion. Because of this reaction, 1900 °C is about the maximum firing temperature when graphite furnaces are used.

Neg 4E 275A

400X

FIGURE 5.10. PNL Pellet Fired Under Highly Reducing Conditions, Showing Possible Carbide Formation (Lamellar), Possible Metallic Second Phase (Bright Areas)

Neg 4E 276B

160X

FIGURE-5.11. Sample in Figure 4.10, After Heating to 1000 °C in Air. Note large amount of pullout.

The $\text{SiO}_2\text{-Y}_2\text{O}_3$ second phase in the solid electrolyte is attacked vigorously by phosphoric acid at about 90 °C. In the solid electrolyte tubes manufactured a year ago, the large islands of second phase are completely removed in a few minutes in H_3PO_4 at 70 to 90 °C. This behavior led to the idea that an etch test may prove useful for detecting second phase in solid electrolyte samples, when the second phase is present in small amounts as in a grain boundary phase. Some PNL samples will not etch in phosphoric acid. A recent etch test comparing a tube from Zircoa Batch Z-5 with a PNL sample revealed no etching at 90 °C in 30 min and etching of both at 150 °C in 30 min. While etching is probably a useful concept, some more work needs to be done to define which impurities etch and the sensitivity of the technique. Etch temperature needs particular attention.

ELECTRICAL CONDUCTIVITY OF A $\text{Y}_2\text{O}_3\text{-ThO}_2$ SOLID ELECTROLYTE

J. J. Rasmussen

The electrical conductivity of a 7.5 wt% $\text{Y}_2\text{O}_3\text{-92.5 wt% ThO}_2$ solid electrolyte was measured in and out of a ^{60}Co gamma field in air, Ar and He. The results are shown in Figure 5.12. The plot of conductivity versus reciprocal temperature shows a uniform slope for all atmospheres at temperatures greater than 550 °K. The change in slope below this temperature is apparently caused by gas conductance. When the sample was removed and runs made with an electrode spacing, the resistances measured in the gamma pit were similar to those measured with the sample present. The resistances measured without a sample increased as the gamma field intensity decreased. To verify that gas conductance is responsible for the change in slope, the conductivity furnace design must be changed to restrict the passage of gaseous ions from one electrode to the other around the sample.

FIGURE 5.12. The Electrical Conductivity of 0.85 ThO₂-0.15 YO_{1.5} as a Function of Reciprocal Temperature In and Out of a ⁶⁰Co Gamma Field

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