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FAST FLUX TEST FACILITY
CHEMICAL ANALYSIS LABORATORY
PRELIMINARY DESIGN STUDY

F. A. Scott
J. F. Jarosch
G. B. Barton

August 1968

AEC RESEARCH &
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FAST FLUX TEST FACILITY CHEMICAL ANALYSIS
LABORATORY PRELIMINARY DESIGN STUDY

F. A. Scott, J. F. Jarosh, and G. B. Barton

INTRODUCTION

The following document presents the results of a preliminary design study scoped to identify the analytical chemistry needs anticipated for support of FFTF operations, survey the current technology which pertains to those needs, select promising analytical methods, specify the facilities needed for application of these methods, and delineate areas of analytical technology which need further research and development to assure that the FFTF analytical requirements will be fully satisfied. The equipment and procedures described do not represent final selections for use in the FFTF analytical facility. They are selected as representative of the present state-of-the-art and as illustrative of the unit operations which will be encountered. Their examination will reveal the complexities which will be encountered in remote operation. It is probable that some of these methods will prove too complex for remote application and that they will be replaced or supplemented with procedures which are less widely accepted but which are more amiable to remote operation. One of the FFTF research and development tasks (SG-3) has as one of its objectives to select, test and evaluate such approaches. Typical of the techniques under consideration are uranium tab immersion for oxygen determination and carbonization tab measurements for carbon.

1.0 FUNCTIONS AND DESIGN REQUIREMENTS

1.1 FUNCTIONS

The Chemical Analysis Laboratory (CAL) is to be a facility in which the analytical work on sodium coolant, aqueous solutions, and reactor atmospheres needed for operational support of the FFTF can be carried out. It is to be designed to service an operating reactor with four primary and four secondary coolant loops and up to six closed test loops. The CAL is to have the capability to:

- Determine trace levels of oxygen, hydrogen, carbon, nitrogen, corrosion products, fission products, particulate matter, plutonium and uranium, on a routine basis and in highly radioactive sodium metal. Heavy shielding will be required and many of the procedures will require operation in inert-atmosphere enclosures.
- Carry out similar determinations on non-radioactive or lightly contaminated sodium (from secondary loop and aged primary loop samples) on a routine basis. The complexity and high cost of hot cell operations makes it highly desirable to minimize the number of samples to be run in the shielded facilities, even at the expense of installing duplicate unshielded facilities.
- Carry out special determinations such as other alkali and alkaline earth metals, halogens, refractory metals, and particulate matter on a non-routine basis.

To permit the CAL to fulfill its basic analytical chemistry function, facilities for these two supporting tasks are also to be provided:

- Receipt and storage of samples.
- Disposal of waste.

1.2 DESIGN REQUIREMENTS

The following are the Design Requirements of a facility performing the functions outlined in Section 1.1.

1.2.1 Achievement of Required Analytical Sensitivity

Corrosion and mass transport phenomena in molten sodium systems are significantly affected by concentrations of impurities in the low parts per million range. The present state-of-the-art does not permit the definition of exact concentration ranges which will be important and under which tests will be carried out in FFTF loops. In many instances, such as in the case of certain carbon species, interest will occur at concentration levels below those measurable by existing techniques. In almost all cases some interest will occur at concentrations down to the detection limit of the best available method.

Presently methods exist which will measure impurity concentrations in non-radioactive sodium down to the following levels:

Total carbon	5 ppm
Total oxygen	1 ppm
Total hydrogen	1 ppm
Hydroxide hydrogen	1 ppm
Nitrogen	1 ppm
Corrosion products by survey techniques	1-20 ppm
Corrosion products by individual techniques	0.1-1 ppm
Fission products and fuel materials	10^{-3} -1 ppm

Every effort should be made to attain comparable detection limits in the FFTF analytical facility both in the presence and absence of sodium with high levels of ^{24}Na .

1.2.2 Prevention of Sample Contamination

Sodium is a very reactive metal and chemically reacts with or "getters" oxygen, carbon dioxide, and water from the atmosphere. The "gettered" impurity is indistinguishable from that originally present in the sodium. Therefore in the trace-level determination of these elements it is absolutely essential that the sodium be rigorously protected from the atmosphere during sampling and during certain crucial steps of the analyses. The inert atmosphere used for this protection cannot contain more than a few tenths of a part per million of oxygen, carbon dioxide or water vapor and the sodium cannot be exposed to even this atmosphere for prolonged periods (hours or more).

Sodium can also react with materials on any surfaces with which it may come in contact. The samplers and tools used to handle sodium prior to and during analysis must therefore be scrupulously clean.

1.2.3 Shielding and Containment of Radioactivity

Many of the samples will be highly radioactive due to neutron activation of the sodium (with the formation of γ -emitting ^{22}Na and ^{24}Na isotopes). The samples may also contain fission products and activation products from other components of the reactor coolant system and from trace impurities in the sodium. Hence, provision must be made for shielding of operating personnel and for containment of any radioactive particulate matter released during the analytical operations.

1.2.4 Manipulative Capability and Visibility

The sampling and analytical operations are complex and require an unusual amount of manipulative ability. Versatile

manipulators and larger-than-normal viewing ports must be provided to meet these unusual requirements.

1.2.5 Replacement of Equipment

An essential design feature of the CAL will be the capability to replace the equipment in any station with new equipment as new or additional procedures become available in future years.

1.2.6 Number of Samples to be Analyzed

It is assumed that at least some of the analytical results for the test loops will be required for process control purposes and therefore will be needed relatively soon after sampling (4-8 hr): It is assumed that short series of samples can be taken on any given loop with a sampling frequency of one per hour but that on the average not more than one sample will be analyzed per loop per day. Further definition of sample load will be required in order to fully determine the size of the high level analytical facility.

2.0 PHYSICAL DESCRIPTION OF THE FACILITY

2.1 SUMMARY DESCRIPTION

The CAL will require these major structural facilities:

- A hot cell containing inert-atmosphere and air-atmosphere enclosures equipped with evacuable air-locks for receipt and transfer of samples.
- Unshielded inert-atmosphere boxes and fume hoods.

2.1.1 Summary of Hot-Cell Functions

The general arrangement of the hot cell components is shown in Figure 1, along with a tabulation of the major activities to be undertaken in those components. These activities are summarized in more detail in the following paragraphs. Somewhat arbitrary selection of the sodium analysis methods to be used was made for purposes of this design study. The selected methods are all typical of those available and will adequately reveal the complexities which may be expected in remote operations. Every effort has been made to select methods which the current state-of-the-art indicates are most desirable, but the final selection of optimum methods will have to await the results of considerable development effort directed toward remotization. The following discusses each of the tasks listed in Figure 1.

A. Sample Receiving and Storage

This activity requires an evacuable air-lock through which samples enter the shielded inert-atmosphere analytical facility. The air lock is to interface with the large sample transfer casks used to transport the samples from the loop sampling stations. A sample storage rack capable of cataloging and storing samples for the duration of each of the loop tests is to be included.

Task	Function	Function	Function
A. Sample Receiving and Storing	Storage and Cataloging		
B. Sample Preparation	Aliquotting and Weighing		
C. Oxygen Determination by "Amalgamation"	Sodium Amalgamation	Acid-Base Titration	Hg Clean-up and Recovery
D. Oxygen Determination by "Distillation"	Sodium Distillation	Acid-Base Titration	
E. Total-Carbon Determination	Sodium Oxidation by Flux or High-Temperature		Product CO ₂ Measurement by gas chromatography apparatus (G.C.A.)
F. Carbon Species Determination	Not Yet Specified	Not Yet Specified	Not Yet Specified
G. Total-Hydrogen Determination	Capsule Pre-Cleaning and Welding	Hydrogen Diffusion	H ₂ Measurement by G.C.A.
H. Hydrogen Species Determination	Sodium Amalgamation	Thermal Decomposition of Amalgam	H ₂ Measurement by G.C.A.
I. Nitrogen Determination		Kjehldahl Method	
J. Sample Dissolution	Aliquotting and Weighing	Dissolution in Alcohol, Conversion to Aqueous System	
K. Chemical Separations		Miscellaneous Chemical Operations	
L. Absorption Spectrophotometry		Color Development and Sample Examination by In-Cell Optics	Light-Beam Reading by Spectrophotometry.
M. Emission Spectrography		Sample Examination By In-Cell Source and Optics	Light-Beam Reading by Spectrograph
N. Radiochemical Analysis			Radioisotope Counting
O. Particulate and Total Solids Determination		Not Yet Specified	Not Yet Specified

FIGURE 1. General Arrangement of Components

B. Sample Preparation

Capability is to be provided for receiving, sectioning, extruding and weighing sodium samples while rigidly controlling contamination from the atmosphere and sampling tools. It consists of a tubing cutter sectioner, an extruder, a recrimping vise and tool and an automatic balance. Viewing and manipulative ability are also to be provided.

C. Oxygen Determination by Amalgamation

The primary method of oxygen determination is to be by the Amalgamation method, which requires amalgamation of the free sodium with mercury in an inert atmosphere followed by acid-base titration in an air atmosphere of the oxide containing residue. Amalgamation vessels, mercury source, and amalgam storage facilities as well as visual and manipulative ability are to be provided in the inert-atmosphere facility, and an automatic titrator is to be provided in the air-atmosphere cell. A vessel cleaning facility and a vacuum drying air lock are to be provided to return amalgamation vessels to the inert atmosphere facility. A separate amalgam clean-up and recovery system is to be provided outside of the shielded area.

D. Oxygen Determination by Distillation

Vacuum distillation will be used as a second method of sodium separation from residual impurities, primarily for oxygen determination and will be in the inert atmosphere shielded facility. Vacuum distillation requires the following major components: glass distillation cell, vacuum system, induction heater, temperature measuring and control devices, manipulative ability, and visual ability. The subsequent titration will be accomplished by the same equipment used in

the oxygen determination by amalgamation. Vacuum distillation could alternately be located at the sampler station.

E. Total-Carbon Determination

Total carbon will be determined in sodium by either high temperature combustion or flux oxidation of the sample, followed by gas chromatographic measurement of the carbon dioxide formed. Sample preparation and loading will require rigid atmosphere control. Carbon dioxide measurement can probably be made outside the shielded facility. An ultra pure combustion gas supply is provided. A combustion train, furnace, temperature measuring device, and special manipulative tools are provided in the inert gas facility. In addition a temperature controller, a gas collecting system and gas chromatograph are provided outside of the shielding.

F. Carbon Species Determination

Methods for the determination of carbon species are not yet available. An exact description of this equipment is therefore not possible at this time. It may involve hydrolysis, acidification and aqueous oxidation with each step coupled to a gas chromatographic analysis of the liberated gases.

G. Total Hydrogen Determination

Total hydrogen in sodium will be determined by thermal decomposition of hydrogen-containing compounds, diffusion of the liberated hydrogen through a steel capsule, and measurement of the diffused gas by gas chromatographic techniques. Equipment is to be contained in the inert atmosphere facility for the precleaning and welding of the capsules. A vacuum train, furnace, temperature controller, and palladium valve are also to be provided in the air atmosphere facility. A gas

chromatograph is to be provided for the quantitative measurement of the liberated hydrogen.

H. Hydrogen Species Determination

Information concerning the hydrogen species present in the sodium is to be obtained by controlled thermal decomposition of an amalgam. The mercury supply and disposal system are to be integrated with those used in the oxygen determination. In addition a vacuum train, heater, and temperature controller are provided in the air atmosphere facility. Actual hydrogen measurements will be made with the same gas chromatographic equipment used for total-hydrogen determination.

I. Nitrogen Determination

Total nitrogen determinations and some nitrogen species identification work are to be accomplished by the Kjeldahl method, with its facilities located in the air atmosphere shielded facility. A conventional Kjeldahl distillation still and its associated equipment are to be provided. In addition a spectrophotometric ammonia measuring apparatus will be required.

J. Sample Dissolution

Many determinations require separation or measurement in an aqueous solution. Because of sodium's unusual reactivity special apparatus and precautions are required for its dissolution and conversion to the aqueous solution. Facilities to be provided for this purpose include manipulators, alcohol supply, acid and distilled water supplies, volumetric glassware, and an aqueous waste disposal system.

K. Chemical Separations

Because the impurities of interest will be present in such low concentrations a preliminary separation and/or concentration will generally be required before impurities can be quantitatively measured. Because of the need to measure such a wide range of trace elements, both ion exchange and solvent extraction capability are to be provided. This will require the inclusion of volumetric equipment and an organic waste disposal facility.

L. Absorption Spectrophotometry

Specific sensitive colorimetric procedures are available for many of the corrosion products and refractory metals which will be of interest. Individual reagents and sample preparation will be required for each trace element to be determined. In-cell optics coupled to a spectrophotometer are to be provided. This optical system is to be versatile enough to permit ready conversion to atomic absorption spectroscopy if such conversion is proven desirable during laboratory testing.

M. Emission Spectrography

A general survey of metallic impurity levels is a necessity and can best be accomplished by emission spectrographic techniques. Sample preparation facilities are to be provided for these operations within the high level cell. An in-cell spectrographic arc stand and optical system are also to be provided. Outside the cell a spectrograph, photographic equipment and densitometer are to be provided.

N. Radiochemical Analysis

Fission products, activation products, and fuel materials are to be determined radiochemically after pretreatment at

the separations station. A multichannel gamma energy analyzer, an alpha energy analyzer and a total beta counter are to be installed in the laboratory space outside the shielded facility.

0. Particulate and Total Solids Determination

The technology for the measurement of total solids and particulate matter is not yet available and therefore it is impossible to adequately describe the requirements at this time.

2.1.2 Unshielded Sodium Analytical Facilities

Many sodium samples such as those from the reactor and loop secondary system will not be radioactive and time schedules will permit radioactive decay of others. These samples are to be handled in unshielded facilities.

Unshielded duplicates of the equipment described in Section 2.1.1 are to be provided. Inert atmosphere glove boxes are to be used wherever an inert atmosphere is required. Other operations are to be performed either on the bench top or in fume hoods. Additional facilities are to be provided to perform unusual determinations and to provide standards for the calibration and check-out of procedures.

2.1.3 Other Analytical Facilities

Laboratory capability for the analysis of cover gas, aqueous solutions, and other gases is necessary as a backup to the planned in-line instrumentation. This capability will be located in laboratory space outside the shielded facility and will consist of sample handling facilities and a versatile gas chromatograph. Consideration should also be given to the analytical support required by the Radiometallurgy facility. The CAL described in this study does not cover this need.

2.1.4 Inert Gas Supply and Purification System

An inert gas blanket is to be provided in a portion of the shielded facility. Components necessary to supply and maintain this blanket are an inert gas supply, a large gas circulating pump, a particulate filter, a gas purification train, gas purity monitoring devices and proper devices to ensure against air leakage such as air locks and manipulator seals.

2.1.5 Other Cell Services

In order to perform all the operations necessary for the various analyses several common laboratory services are to be provided. These include: electric power, cooling water, deionized water, adequate lighting, vacuum, adequate heat-removal capability, decontamination facilities, aqueous waste disposal facilities and radioactive sodium waste disposal activities.

2.1.6 Alternative Approaches Considered

Among the alternative approaches which have been considered are the following: the modification and use of shielded analytical facilities which already exist at PNL; the use of shielded analytical facilities at other AEC installations; the performance of all required analyses by the individual customers of the irradiation facility; and the analysis of sodium samples only after the decay of ^{24}Na . None of these approaches is considered satisfactory, however, since they seriously reduce the value or amount of the technical information which can be obtained.

2.2 DETAILED DESCRIPTION

2.2.1 Components for Sample Receiving and Storage

A. Entrance Air Lock

The entrance air lock has three primary functions:

- To permit the transfer of a sample from a transfer vessel to the shielded analytical facility
- To provide adequate radiological shielding during this transfer
- To prevent the contamination of the inert atmosphere of the facility by air during the transfer.

Rather unusual sample shapes are expected and the air lock must be big enough to accomodate them. The air lock will be designed to couple to the relatively massive sample transfer casks required, and will be evacuable and flushable with inert gas. A high capacity vacuum pump and a supply of high purity inert gas are therefore supplied at the air lock. Oil vapors from the vacuum pump will be trapped to prevent carbon contamination. Consideration should be given to the use of the transfer cask itself as the air lock if the problems of an adequate, reliable, and practical seal can be overcome.

B. Sample Storage Racks

Portions of selected samples will have to be stored for periods up to six months. For the first two or three weeks of this storage these samples will be highly radioactive and will require considerable radiological shielding. An orderly storage system is therefore to be provided within the shielded facility. The system is to be capable of indexing approximately 100 separate samples.

The half life of ^{24}Na is short (15 hours) and the radiation level of the stored samples which are not contaminated with

other radioisotopes can be expected to drop rapidly enough to permit their storage outside the shielded facility after about a three week period. It may be possible to save valuable in-cell space by providing additional storage racks for "cooled" samples outside the shielded facility.

C. Sample Logging System

The simultaneous and continuous operation of five separate test loops will generate a large number of samples. Each sample may have a different history and require different determinations. A complex, orderly and efficient labeling and logging system will be necessary in order to avoid confusion. This system will have to be completely integrated with the sample storage facility, with the analytical operations and with the data reporting system.

2.2.2 Components for Sample Preparation

A. Sample Cutter

Representative segments of measured, reproducible size must be taken from the as-received sodium sample for each of the analytical determinations. Most samples will be received as capped or crimped sections of stainless steel tubing from a bypass sampling loop. Reproducible lengths of the tubing will be removed with a tubing cutter, lathe, or cutoff wheel. A tubing cutter appears to be most readily adaptable to remote operation.

B. Sample Recrimper

The sample must be completely protected from the atmosphere. Prolonged exposure to any atmosphere, even one as pure as that of the cell, will be detrimental. This protection will be afforded by recrimping the stainless steel tube containing the sample. A recrimping tool will be provided for this operation.

C. Extruder

Several of the required determinations, such as total carbon, cannot be made in the presence of stainless steel and the sodium must be separated from the stainless steel jacket. An extruder is provided for this operation. Several extruder designs have been used in the laboratory but all will require modification for remote operation.

D. Balance

The sample cutter and extruder must be calibrated and their performance monitored periodically by actual sample size measurements. An automatic analytical balance (such as a Mettler) is to be provided. Additional measurements will be made chemically through the use of the dissolution and titration stations.

E. External Sample-Container Cleaner

The oxide coating which accumulates during sampling on the outside of the sample container may be detrimental to certain determinations, notably oxygen, and may have to be removed. This removal will be accomplished before the sealed sample container is opened. A mechanical buffer is to be provided at the sample preparation station for this purpose. A means is also to be provided to prevent the escape of the dust created to the rest of the facility.

2.2.3 Components for Oxygen Determination by Amalgamation

A. Amalgamation Vessels and Support Racks

Amalgamation vessels are to be made of glass, and are to be especially designed for remote operations with manipulators. Special features which are to be included to facilitate remote operation include ruggedness, sealing caps, and manipulator handles. To facilitate the separation of the impurities by

flotation and to aid in the simultaneous handling of multiple samples, racks are to be provided to hold the amalgamation vessels.

B. Mercury Supply

A supply tank and a delivery system are to be provided to deliver purified mercury in measured quantities for amalgamation. This mercury will be primarily recycled material with a small amount of make-up.

C. Amalgam Disposal Facility

A drain system is to be provided to deliver the waste amalgam to two storage tanks. One tank is to be used to receive freshly formed amalgam while radioactivity in the second tank is permitted to decay in preparation for mercury recovery.

D. Amalgam Clean-Up Facility

The mercury in the amalgam must be purified, recovered, and recycled. It will contain ^{24}Na and be highly radioactive. This radioactivity is short lived, however, and will be reduced to acceptable levels in two to three weeks. The amalgam will be stored in a tank for two weeks to allow for ^{24}Na decay and then recovered in a contact-type maintenance facility. It may be recovered immediately if the first cycle of the recovery system is placed in a shielded facility. The technical information and cost evaluations necessary to decide between immediate recovery and storage is not yet available.

E. Shaking and Draining Mechanism

Proper amalgamation and oxide separation require methods for shaking and draining the amalgamation vessel. Remote shaking and drain control devices can be designed, constructed, and tested, but present indications are that they

will not be necessary or desirable. Greater versatility in operation will be possible through the use of the master-slave manipulators in the facility.

F. Air-Lock Between Shielded Facilities

An evacuable, flushable air-lock is to be provided between the inert- and air-atmosphere shielded facilities. The amalgamation vessels containing the residual sodium oxide will be passed through this air lock prior to the dissolution and analysis of the oxide residue. The need for rapid operation of this air-lock warrants the inclusion of a high capacity, high vacuum (0.1 mm of mercury) pump suitably trapped to prevent back diffusion of oil vapors.

G. Titration Facility

Facilities are to be provided for the dissolution of the sodium oxide residue in the air-atmosphere shielded facility. An automatic pH titrator is to be provided for the measurement titration. The sensing unit, stirrer and titration vessel are to be located within the air-atmosphere shielded facility, while the electronics and recorder are to be outside the shielded facility. A special coaxial electrical cable is to be used to bring the signal from the sensing unit through the radiological shield. Facilities to eliminate atmospheric carbon dioxide from the dissolution and titration vessels may be necessary.

H. Vacuum Drying Oven Air-Lock

A vacuum drying oven air-lock is to be provided between the inert-atmosphere and air-atmosphere shielded facilities. The air-lock will permit the recycle of the amalgamation vessels and remove absorbed water from their surfaces before the vessels are inserted into the inert atmosphere. Water removal from glass surfaces is vital to the maintenance of low procedure blanks. A pump capable of producing a vacuum of less than 0.1 mm of mercury is to be provided to speed water removal. The air lock can also be heated to approximately 200 °C for the same purpose.

2.2.4 Components for Oxygen Determination by Distillation

Present planning calls for vacuum distillation (for sodium removal from the non-volatile residue) to be done "close coupled" to sampling and therefore to be performed at the sampling station. Significant benefit can be realized from this early removal of the bulk of the radioactive sodium. The close-coupling may prove undesirable, however, because of the increased complexity of the sampling station. If so, the distillation operation will be transferred to the high level analytical facility. The following distillation system components will then be necessary.

A. Glass Distillation Cell

An especially designed glass cell is to be provided for distillation of the sodium. Glass, rather than a more rugged material such as metal, is required to facilitate induction heating of the sodium. Several cell models are presently undergoing tests at LASL and ANL and an optimum design of this cell is not available at the present time.

B. Vacuum System

A high-capacity vacuum system consisting of cryogenic traps, mechanical rough pump, and pressure measuring devices is to be provided. The cryogenic traps are not conventional liquid nitrogen traps because of their location in an argon atmosphere facility. A larger-than-normal pump (425 liters/min) is to be provided to ensure adequate performance and to increase the speed of analysis.

C. Induction Heater

A 3 kVA induction heater and coupling coil are to be provided to perform the distillation. The electronics of

the heater are to be located outside the radiological shielding and a special long lead-through is to be provided to take the RF current through the shielding to the load coil, which is located on the sodium still inside the shielded facility.

D. Still-Temperature Measuring and Control Device

Close control of distillation temperature is important to prevent overheating of the residue while permitting rapid distillation. Close temperature control of the nickle crucible is vital and is to be ensured by intimate contact between the crucible and the measuring thermocouple. The output of the thermocouple is to be fed through the radiological shielding to a temperature controller in the operating gallery, which in turn controls the output of the induction heater.

E. Sample Cup Replacement Mechanism

Sample cup replacement and load-out are to be accomplished by the master-slave manipulators provided for general operation of the station. Future redesign of the still may make a semi-automatic cup removal mechanism desirable. Removal and disposal of the distilled sodium is also to be accomplished with the operations manipulators.

F. Air Lock Between Shielded Facilities

The cup containing the distillation residue will be transferred to the air atmosphere shielded facility through the air lock described in Section 2.2.2-F for the transfer of the amalgamation vessels. On many of the less highly radioactive samples it may be possible to remove this cup from the shielded facility for titration in an unshielded fume hood.

G. Sodium Disposal System

Stainless steel storage vessels will be provided for the storage of waste solid sodium prior to transfer to the air-atmosphere shielded facility for ultimate disposal.

H. Cup Washing System and Automatic pH Titrator

The solid impurities (primarily sodium oxide) in the cup will be dissolved and titrated in the facility described in Section 2.2.2-G.

I. Cup Clean-Up System

The distillation cups will be recycled to the inert atmosphere facility through the clean-up system and vacuum drying oven air lock described in Section 2.2.2-H.

J. Inert Gas Supply to Vacuum System

In order to remove the sample cup after the distillation it is necessary to refill the vacuum system with an inert atmosphere. A special inert gas supply may be required, but present plans call for the direct use of the surrounding cell inert atmosphere.

2.2.5 Components for Total Carbon Determination

A. Ultrapure Combustion Gas Supply

Cylinders of ultrapure helium and oxygen will be provided outside the radiological shielding. A system is to be provided to regulate the gas flow, mix the gases, and pass them to the gas purification train. The purification train is to consist of a copper oxide-packed quartz tube (held at 700 °C by a furnace and temperature controller) and a large unheated column of ascarite. Provisions will be made to pass the purified gas through the radiological shielding to the combustion train.

B. Combustion Train

Combustion of the sodium is to be carried out in a thick walled quartz combustion tube by a flowing stream of purified

mixed gases. The temperature of the tube is to be programmed to prevent the distillation of sodium in the early stages of combustion and to completely decompose sodium carbonate at 1100 °C in the later stages of the combustion. Because of the 1100 °C requirement a specially wound furnace is needed. Because of the need to raise and lower the combustion tube furnace temperature for each determination, a low heat capacity, high power furnace is to be used for rapid response. A power supply and temperature controller are to be supplied for the furnace and are to be located outside the radiological shielding.

C. Special Tools and Manipulators for Combustion Boat Handling

Extreme care is required to prevent the contamination of the combustion tube during sodium loading. Such contamination can occur from any source of carbon, most notably organic matter. To prevent such contamination, provisions are to be made within the shielded facility for the storage, cleanup, and use of special tools to handle the boat, extrude the sodium sample, cut it, and place the reassembled combustion tube in the furnace tube.

D. Gas Collection System

The combustion gas is to be passed through the radiological shielding to a liquid nitrogen trap in the operating gallery, through the liquid nitrogen trap and finally vented to a hood containing an absolute filter. Valving is to be supplied to permit switching the liquid nitrogen trap back and forth between the combustion gas stream and the chromatograph carrier gas stream.

E. Gas Chromatograph

A gas chromatograph and carrier gas supply are to be provided in the operating gallery to perform the measurement of the

liberated carbon dioxide. Helium carrier gas and Poropack-Q columns are to be used with thermal conductivity detection.

F. Sample Tube Preconditioning System

A sample tube preconditioning system will be provided inside the inert atmosphere shielded facility. It consists of a combustion gas supply (helium and oxygen mixture), a combustion tube, a furnace and temperature controller, and a vent system for the combustion gas. The purpose of the vent system is to prevent the contamination of the atmosphere of the inert-gas facility by the oxygen of the combustion gas mixture. All oxygen-containing combustion gas must be swept from the system before it is opened.

The tools used to handle the combustion boats (Section 2.2.5-C) will also be used to handle the preconditioned sample tubes.

2.2.6 Components for Carbon Species Determination

Methods for the determination of carbon species are not yet fully developed. The description of the components of this station will therefore have to await the results of development efforts now in progress.

The procedure presently favored by many investigators involves a three-step process with the collection, identification and measurement of the gas evolved in each step. The metal will be hydrolyzed first with the resulting solution remaining strongly basic. The aqueous solution will then be acidified and finally a vigorous aqueous oxidation will be carried out using an oxidant such as Van Slyke's reagent.

2.2.7 Components for Total Hydrogen Determination

A. Precleaned Steel Capsules for Encapsulation

Capsules for sample encapsulation will be precleaned outside the shielded facility and placed in sealed containers for transfer into the inert atmosphere. Sodium samples will be prepared and placed in these capsules at the sample preparation station (Section 2.2.2).

B. Welder

A welder will be provided in the inert atmosphere shielded facility to completely seal the steel capsules.

C. Air Lock

The sealed capsules containing the sample will be transferred to the air-atmosphere shielded facility through the air lock described in Section 2.3.6-F.

D. Vacuum System

Leak testing of the welded capsule will be performed in a vacuum system which is to be located in the air-atmosphere section of the shielded facility. Hydrogen release and measurement will also occur in this system. The major components of the systems are: cryogenic traps, diffusion pump, rough pump, pressure monitoring devices and (possibly) a palladium valve.

E. Furnace

A nichrome-wound tube furnace will be provided to heat the sample to 700 °C and effect the release and diffusion of the hydrogen.

F. Temperature Controller and Power Supply for Furnace

A temperature-sensing element and controller will be provided to monitor and control the temperature of the furnace.

The output of the controller is to switch power to the furnace on and off. 110 Volt ac power is to be supplied through a variable transformer which permits better control of furnace temperature fluctuations.

G. Palladium Valve

A palladium valve may be placed between the two sections of the vacuum system to separate the hydrogen and make the manometric measurements more specific for hydrogen.

H. Hydrogen Gas Measuring Device

The diffused hydrogen is to be measured manometrically. If future developments indicate the need for greater specificity, measurements can also be made by gas chromatography. To do this a Toepler pump and gas collection trap will be added to the vacuum system. The gas chromatograph described in Section 2.2.5-E will be adequate to perform the hydrogen measurements if a multi-column chromatograph is used.

2.2.8 Components for Hydrogen-Species Determination by Amalgamation

A. Vacuum System

A vacuum system will be provided to perform thermal decomposition of the amalgam and to handle the released gas. The main components of the system are to be a reaction vessel, cryogenic trap, rough pump, diffusion pump, and pressure monitoring device.

B. Mercury Supply

The mercury supply described in Section 2.2.3-B will be used to form the amalgam.

C. Amalgam Disposal Facility

Waste amalgam is to be disposed of through the amalgam disposal facility described in Section 2.2.3-C.

D. Induction Heater for Amalgam Reflux

An induction heater and coupling coil will be provided to reflux the amalgam. The electronic portion of the heater will be located in the operating gallery and a specially designed long lead coil will be used to transmit the RF energy through the shielding to the amalgam.

E. Gas Chromatograph

The liberated hydrogen will be trapped and measured on a gas chromatograph located in the operating gallery. It is not possible at this time to determine whether the injection port of the chromatograph should be located inside the radiological shielding or whether the relatively large number of samples required should be transported through the shielding to an external chromatograph.

F. Inert Gas Supply to Refill System

A supply of high purity argon gas will be provided for filling the vacuum system between refluxing operations. Subsequent investigation may indicate that the inert atmosphere of the facility will be adequate for this purpose and the separate supply may be deleted.

2.2.9 Components for Nitrogen Determination

A. Air Lock Between Shielded Facilities

Samples prepared for nitrogen determination will be passed into the air-atmosphere shielded facility from the inert atmosphere shielded facility by means of the air lock described in Section 2.2.3-F.

B. Kjehldahl Still

A glass Kjehldahl still with its associated ammonia collection container will be provided to perform the distillation.

C. Alcohol and Water Supply

Alcohol and water are required for the safe dissolution of sodium prior to nitrogen determination. These reagents are to be provided and the initial dissolution made at the sample dissolution station described in Section 2.2.10.

D. Heater for Kjeldahl Still

A resistance heating mantle will be provided for the still pot to provide heat for the distillation. Future developments may indicate that a steam supply will be more suitable and an extra steam generator may be added.

E. Collection Reagent Supply

Collection reagent volumes will be small and are to be supplied by passing reagent containers directly into the shielded facility.

F. Ammonia Measurement Device

The liberated ammonia will be measured by either automatic pH titration or the colorimetric Nessler's method, depending upon the impurity level of the particular sample being analyzed. The majority of samples will be low in nitride and will be measured colorimetrically using the spectrophotometer described in Section 2.2.12.

2.2.10 Components for Sample Dissolution

A. Alcohol Supply

A supply of reagent grade alcohol (ethyl or methyl) will be provided for the dissolution of solid sodium samples by a tank located outside the shielded facility. Some means of delivering measured quantities remotely are also to be provided. Visual observation will be used to determine the completeness of dissolution.

B. Distilled Water Supply

A supply of distilled water will be provided. Future developments may indicate that a mercury solvent is also needed. If so, the mercury supply described in Section 2.2.3-B can be used.

C. Acid Supply

Supplies of reagent grade hydrochloric, perchloric and sulfuric acids will be provided. These acids will be used for neutralization and pH adjustment prior to chemical separations work on dissolved samples.

D. Volumetric Glassware

Volumetric equipment is essential to quantitative measurements. Storage space for this equipment will be provided. Equipment especially adapted to remote operations is to be used.

E. Remote Micropipetter

A remotely operated, motor-driven micropipetter will be provided to perform dilutions and quantitative sample measurements. The pipetter is to be located inside the shielded facility with its controls in the operating gallery.

F. Liquid Waste Disposal System

Highly radioactive acidic and basic aqueous wastes will be generated during sample dissolution and makeup. A slurp system, drain lines, storage tanks and load out facility are to be provided to handle these wastes.

2.2.11 Components for Chemical Separations

A. Remote Micropipetter

A second remotely operated micropipetter will be provided at the separations station.

B. Volumetric Glassware

Volumetric glassware will be required at the separations station. The glassware and storage facilities provided under Section 2.2.10-D are to be used.

C. Ion Exchange Columns and Racks

Ion exchange columns and supporting racks are to be provided. Either anion or cation columns may be needed. (These are small glass analytical columns.) Vacuum and compressed air are also to be provided to aid in the control of column flow.

D. Eluent Supply

Supply tanks and delivery systems are to be provided for three eluent solutions for the ion exchange columns. Further developments may indicate that more versatility is needed, in which case eluents will be supplied from reagent bottles and the supply tanks eliminated.

E. Aqueous Waste Disposal Facility

Highly radioactive aqueous waste solutions will be generated during the separation operation. The facility for their disposal is to be completely integrated with the one described in Section 2.2.10-F.

F. Organic Solvent Supply

Supply tanks and delivery systems are to be provided for three organic solvents to be used in solvent extraction separation procedures. Further developments may alter the number of required solvents or may indicate a preference for the use of reagent bottle supplies.

G. Vortex Stirrer and Extraction Vessels

A small motor driven vortex stirrer is to be included to provide the mixing necessary for solvent extraction. Extraction vessels will be small capped vials or test tubes, requiring no special adaptation.

H. Organic Waste Disposal Facility

Highly radioactive organic waste solutions will be generated during the solvent extraction separation. Separate waste disposal facilities are required for these because of their organic nature. The exact size of these facilities has not been established but will depend primarily on the volume of waste to be handled.

2.2.12 Components for Absorption Spectrophotometry

A. Reagent Supplies

Approximately six colorimetric reagents will be required for each element to be determined spectrophotometrically. Volumes will be small and these reagents are to be taken into the shielded facility in reagent bottles as needed.

B. Volumetric Equipment

Volumetric glassware will be required during sample preparation for spectrophotometric measurements. The supply mentioned in Section 2.2.10-D is to provide any equipment needed by this station.

C. In-Cell Optical System

An in-cell optical system is to be provided. The primary components of this system are a light collimator on the spectrophotometer, some form of light channels through the radiological shielding, optical mirrors, and precisely calibrated optical cells.

The optical system is to be versatile enough to permit conversion to atomic absorption spectroscopy if this is deemed desirable. Conversion would entail the substitution of a grouping of hollow cathode lamps for the spectrophotometer light source and the substitution of a burner and flame for the absorption cell.

D. Spectrophotometer

A recording spectrophotometer is to be provided to measure and record the spectra of samples quantitatively. The instrument is to cover at least the range of 3500 to 7500 °A.

2.2.13 Components for Emission Spectrography

A. Sample Evaporators

Hot plate and infrared lamp facilities are to be provided within the air-atmosphere shielded facility for the evaporation and drying of samples for emission spectrographic measurements. Electrode racks are to be provided to aid in mounting specimens. Electrodes will be prepared in the operating gallery and transported through the shielding as required. A grinder is to be provided for electrode preparation.

B. In-Cell Optics and Arc Stand

A conventional spectrographic arc stand is to be provided within the air-atmosphere shielded facility. Special optics are to be provided to gather the source light and transmit it through the radiological shielding to the spectrograph.

C. Spectrograph

A 1.5 meter Wadsworth mounted-grating spectrograph is to be provided in an especially air conditioned room adjacent

to one end of the shielded facility. Significant sources of vibrations should not be located in the vicinity of this room.

D. Dark Room

A dark room equipped for plate development is also to be provided outside the shielded facility. The primary facilities required in the dark room are safelights, developing trays, running water, and drying racks.

E. Plate Storage Facility

A freezer unit is to be located in the dark room for the storage of unexposed spectrographic plates.

F. Power Supply for Excitation Unit

A 5 kVA dc arc power supply and a 2 kVA ac spark power supply are to be located in the operating gallery. Special provisions are to be made for the transmission of power through the radiological shielding to the source unit.

G. Densitometer

A comparative densitometer is to be provided for quantitative reading of the emission plates and comparison of the qualitative plates.

H. Alternate Approaches

Emission spectrography is neither sensitive enough nor precise enough to meet some of the needs of the installation. Several alternative survey techniques are presently under development to meet these needs. These alternative techniques include spark source mass spectrometry and atomic absorption spectrometry. The possible inclusion of these capabilities will

hinge upon the results of the development efforts now under way, upon the availability of service from similar facilities located elsewhere at PNL, and upon an evaluation of the costs involved.

2.2.14 Components for Radiochemical Analysis

A. Volumetric Sample Preparation Facilities

Volumetric sample preparation and dilution will be accomplished either at the separation station (Section 2.2.11) or at the sample dissolution station (Section 2.2.10). No special facilities are planned for this operation for radiochemical analysis.

B. Sample Mounting Facility

A remote micropipetter, infrared lamps and a fume hood are to be provided inside the air atmosphere shielded facility for the preparation and mounting of samples for counting.

C. Multichannel Gamma Energy Analyzer

A multichannel (4096) gamma energy analyzer is to be provided in the operating gallery. Associated with this instrument are to be two lithium-drifted germanium diode detectors and perhaps two sodium iodide detectors. These are to be installed outside the shielded facility. One of the germanium diode detectors is to be adjacent to the analyzer and used to count samples transported from the shielded facility. The second is to be mounted on a specially designed collimating plug in the radiological shielding and is to be used to count higher-level samples which remain within the shielding. Magnetic tape and high speed printer readout of the multichannel analyzer output is planned.

D. Isotope Standards and Standard Tape Decks

Provisions are to be made for the storage of a number of standard tape decks containing the gamma energy spectra of many of the fission and activation products. These will be used in spectrum stripping analysis.

E. Alpha Energy Analyzer

A multichannel alpha energy analyzer is to be provided outside the shielded facility, together with its associated detector.

F. Total Beta Counter

A total beta proportional counter is to be provided outside the shielded facility.

2.2.15 Components for Particulate and Total Solids Determinations

Methods for the measurement of particulate matter and total solids are presently in the development stage and an exact description of the components of this station is therefore not possible at this time. The station may include some or all of the following:

- Furnace to hold samples at 1200 °F for prolonged periods for equilibration
- Filters
- Centrifuge or ultracentrifuge
- Ammonia dissolution equipment
- Vacuum distillation equipment.

2.2.16 Components for Unshielded Sodium Analytical Facility

Secondary loop sodium samples and any primary loop samples for which time is available to permit radioactive decay are to be analyzed using unshielded facilities, since a considerable reduction in cost can be obtained by so doing. Duplicate

unshielded facilities are to be provided for the following operations:

- Oxygen determination by amalgamation
- Oxygen determination by vacuum distillation
- Total carbon determination
- Carbon species measurements
- Total hydrogen determination
- Amalgamation hydrogen determination
- Nitrogen determination
- Sample dissolution
- Sample separation
- Particulate and total solids determination
- Emission spectrographic excitation.

These facilities are to be as described below.

A. Inert-Atmosphere Glove Boxes

Glove boxes are to be used for operations requiring an inert atmosphere but no shielding. These operations are to include the following:

- Oxygen determination by amalgamation
- Sample preparation for the determination of total carbon, total hydrogen, hydrogen species (by amalgamation), nitrogen, and oxygen by vacuum distillation
- Carbon species determination.

B. Equipment for Non-Routine Determinations

Particularly versatile facilities are to be provided for non-routine determinations. These include analysis for other alkali metals, alkaline earth metals, refractory metals and halogens. The bulk of this work will be wet chemical in nature and will be performed on the benchtop or in fume hoods if radiation levels permit.

C. Facilities for Standards Preparation and Calibration

All methods are to be checked out by the use of standard samples. These standards will also be used to compare the results obtained by the newly developed remote procedures with those obtained by the more conventional procedures. A versatile glove box facility is to be provided for use in the preparation of the required working standards.

D. Dual Function Facilities

Many of the wet chemical procedures used on highly radioactive sodium call for the separation of the trace elements of interest prior to their determination. In these instances the actual determination is to be performed outside the shielded facility, and only one set of equipment is planned for both radioactive and non-radioactive work. Dual function pieces of equipment which fall into this category are:

- Spectrophotometer
- Emission Spectrograph
- Dark Room
- Densitometer
- Radiochemical Counting Equipment
- Gas Chromatographs.

2.2.17 Components for Cover Gas Analytical Facility

In-line process instruments will provide most of the information required concerning the composition of the sodium cover gas. One of the major uses of the laboratory facilities will be to assist in analyzing off-standard samples. Another use will be to provide calibration standards, or analysis facilities to verify the composition of commercial calibration

standards. The process instruments will need to be calibrated and their performance verified against such standards.

In dealing with trace or low-level impurities in inert cover gases, analysis of grab samples may be very misleading. There is always a certain amount of gaseous contaminant absorbed on the surface of the containment vessel. This may remove or contribute impurities to the sample. For this reason, the use of continuously operating process instruments is preferable since they can reach a state of equilibrium with the sample and sample lines and thus minimize effects caused by surface adsorption.

Grab samples are most useful in the analysis of off-standard samples because such samples are frequently high in the contaminating substances and therefore less perturbed by small absorption effects.

A. Gas Chromatograph

Gas chromatography affords an excellent means of routinely detecting and measuring very small concentrations of impurities in inert gases. For example, in helium it is possible to measure less than 1 ppm hydrogen, oxygen, nitrogen, methane and carbon monoxide. In nitrogen and argon, it is possible to measure concentrations as low as 10 ppm. A good gas chromatograph with several detectors and columns can also provide quantitative and qualitative information on non-routine samples containing unusual constituents or unusual amounts of normal trace impurities. In many cases the initial identification of unusual constituents can more readily be accomplished mass spectrometrically however.

B. Mass Spectrometer

Gas mass spectrometry techniques can be provided as an alternative to gas chromatography. It has some advantages,

such as greater ease of identifying unknown materials and more uniform applicability over a wide variety of matrix gases. Helium is the most easily handled matrix material in both mass spectrometry and gas chromatography. Backup support to the Chemical Analysis Laboratory can be supplied by other mass spectrographs at PNL if there is not a heavy sample load and if the samples are such that they can be transported in their containers.

Mass spectrometry has the disadvantage of being more expensive than gas chromatography in areas of both capital outlay and operating costs.

C. Gas Handling Facilities

If cylinders of calibration gas are to be prepared by the laboratory, a manifold with appropriate pressure gages and isolation valves, together with a vacuum pump and bake-out furnaces will be needed. This system should be in a controlled-temperature room.

Grab samples for introduction into the gas chromatograph or mass spectrometer will need to be handled on a manifold that provides for removal of air from the connections, lines, valves, etc. prior to introduction of the sample to the instrument inlet. Since most of these gas samples will be at atmospheric pressure, some type of pump compressor (e.g., Toepler pump) will be needed with the gas chromatograph to provide flow through the instrument. Samples can be supplied to the mass spectrometer directly without pumping.

If grab samples are to be brought to the laboratory, facilities for evacuating and cleaning the containers for these samples will be needed. Usually a mechanical vacuum pump suffices for this service but occasionally baking under vacuum is necessary.

2.2.18 Components for Inert Gas Supply and Purification

Large volumes of very high purity inert gas are required for the successful operation of the analytical facility. Because of the high purity and high circulation rates required, it is doubtful that the reactor cover gas can be used directly. If not, the following inert gas system components will be required.

A. Inert Gas Supply

The system is to be a recirculating one and only a small amount of argon will be required for make-up. The make-up may either be drawn directly from the reactor cover gas system or from clean argon storage.

B. Circulating Pump

A large volume circulating pump is to be provided to recirculate the inert gas between the operating facility and the gas purification train.

C. Radiological Filters

A low pressure-drop, high gas-flow absolute filter is to be provided between the operating cell and the gas purification train to remove all traces of particulate matter from the gas stream.

D. Gas Purification Train

A gas purification train is to be provided. Its primary components are to be a dual column molecular sieve trap, a zirconium or titanium metal hot trap and a heat exchanger.

E. Gas Purity Monitors

Purity monitors are to be provided to measure water vapor, oxygen and carbon dioxide in the inert atmosphere.

Their sensing elements will be located within the shielded facility and their electronics will be located in the operating gallery. Instruments are commercially available which are capable of measuring water vapor and oxygen in the tenths of a part per million range. Electrical connections are to be provided to bring the sensing element signals through the shielding.

F. Air Lock Entrances and Exits

All entrances to the inert atmosphere facility must be constructed so as to prevent the contamination of the atmosphere. An air lock entrance for samples has already been described in Section 2.2.1. A similar evacuable air lock, without facilities to accomodate large casks, is to be provided for placing equipment and reagents into the facility and for removing samples and some waste from the facility.

G. Manipulator and Other Seals

Because of the need to avoid air contamination of the atmosphere, sealed manipulators are to be used throughout the inert atmosphere facility. Electrical and piping entrances are to be made through hermetically sealed fittings.

H. Air Replacement in Inert Gas Facility

A mechanism is required to replace the air within the inert gas facility before start-up of the facility. A firm decision concerning the technique to be employed will depend on future design studies. Among the candidate procedures are:

- Evacuation of the entire system followed by refilling with inert gas
- Repeated inert gas flushes

- Displacement through the use of an inflatable bag or balloon

Of these techniques, evacuation is the most desirable from the standpoint of speed-of-operations.

2.2.19 Other Services

A. Electrical Power

110 and 220 V 60 cycle ac power are to be provided throughout the laboratory to operate furnaces, pumps and electronic equipment. A separate emergency power system is to be provided. An isolated 60 cycle ac power system is to be provided outside the shielded facility for the operation of sensitive electronic instrumentation.

B. Cooling Water

Process water is to be provided throughout the laboratory to act as cooling water in stills and induction heaters. Water sources and sodium storage facilities are to be located with respect to each other in such a manner as to minimize the danger from inadvertent interaction.

C. Distilled Water

Distilled water outlets are provided in the air atmosphere shielded facility and several locations throughout the laboratory. This water will be used to clean up equipment and to prepare solutions and reagents.

D. Lighting

Normal lighting is to be provided throughout the laboratory. The shielded facility will require intense lighting.

E. Vacuum

Crude vacuum (25 mm of mercury) is to be provided at various points in the laboratory and air-atmosphere shielded facility to aid in general laboratory functions such as the cleanup of equipment and ion exchange column operation. Individual vacuum systems are planned for all locations where higher vacuums are required, such as air locks and laboratory equipment. The need for individual systems arises from inability to prevent interactions in a complex multi-use high vacuum system.

F. Compressed Gases

Compressed air, oxygen, propane and nitrogen are to be provided in the laboratory, and all but propane are to be provided in the air-atmosphere shielded facility.

G. Heat Dissipation

The shielded facilities will contain several unusually large sources of heat. Most notable among these will be the lights and the heating elements associated with furnaces and distilling equipment. Because of the confined nature of these facilities special provisions will be required to dissipate this heat. The large flow of purified gas in the inert atmosphere facility is to be used to remove heat from the shielded area. The heat in turn is to be removed from the gas by the heat exchanger mentioned in Section 2.2.17-D. A temperature monitoring and alarm system to ensure adequate temperature control is to be provided. A large flow of air will be maintained in the air atmosphere shielded facility for heat removal. Blowers and absolute filters are to be provided for this air stream.

H. Decontamination Spray and Drain

Both shielded facilities are to be provided with a decontamination spray and drain system. The presence of sodium in these facilities will create a hazard when this decontamination technique is used and adequate precautions will be required to prevent use of the decontamination system when the possibility exists that metallic sodium is present.

I. Radioactive Aqueous Waste Disposal

A storage tank and piping, valving and load out facilities are to be provided which are capable of handling both acid and alkaline radioactive aqueous wastes.

J. Radioactive Sodium Waste Disposal

Waste radioactive sodium is to be dissolved in alcohol and combined with the radioactive aqueous waste.

K. Hoods and Contamination Control

Fume hoods are to be provided throughout the laboratory facility. Adequate centrifugal blowers are to be provided to ventilate these hoods through absolute filters for control of the spread of contamination. The hood system is to be integrated into the building ventilating system to provide maximum contamination control.

L. Ventilating and Air Conditioning

Adequate ventilation and air conditioning for the bulk of the laboratory are to be drawn directly from the primary facility system. A small auxiliary system is to be provided to supply special temperature and humidity control to the emission spectrographic laboratory.

2.2.20 Instruments, Controls, Alarms, and Protective Devices

Many instruments, controls, alarms and protective devices are to be provided for the safe operation of the analytical facility. Among the more important of these are:

A. Fire Detector

A fire detection and alarm system is to be provided. Normal sprinkler fire control measures are to be avoided because of the potential presence of metallic sodium.

B. Smoke Detection

Smoke detection equipment and alarms are to be provided in the air atmosphere shielded facility and conventional laboratory space. Smoke detection is one of the more useful approaches to metallic sodium fire surveillance because such fires have no flame but produce unusually large quantities of white smoke.

C. Temperature Monitor

Temperature monitoring of both shielded facilities is important particularly during periods of reduced gas circulation. The systems contain unusually large heat sources and have limited capability to dissipate this heat.

D. Pressure Monitor

Pressure buildup in the inert gas shielded facility is possible from a number of sources. Contamination control requires the maintenance of a negative system pressure in both shielded facilities. Monitoring and control devices are to be provided for this purpose.

E. Hydrogen Explosion Limit Meter

A potential exists for the generation of significant quantities of hydrogen in the shielded facilities through

the interaction of alkali metal with water or alcohol. A study should be made to determine if the buildup of an explosive mixture within the system is possible, particularly under conditions of reduced gas flow.

2.2.21 Shielding Requirements

Analytical samples will contain large amounts of ^{24}Na which is a short half-life, high-energy gamma emitter. The radiation fields produced will be hazardous and personnel will have to be protected from them.

A. General Radiological Shield

For purposes of this study it was assumed that one hundred samples (20 g each) of sodium will be stored in the analytical facility and that each gram of sample will contain 0.1 Ci of ^{24}Na . It is also assumed that as many as 10 of these samples are to be under investigation in the facility at any one time. Under these assumptions 3 ft of high density magnetite concrete will be required to reduce the radiation field on the operating face of the shielded facility to below 1 mR per hr.

B. Stainless Steel Liner

The interior of the shielded facility will become contaminated with radioactive materials during operation. The capability to decontaminate the facility to permit entrance for maintenance purposes or to permit major overhaul or replacement of equipment will be required. A stainless steel liner is to be provided to facilitate required decontamination.

C. Remote Manipulators

The analytical procedures to be carried out in the shielded facility require many complex manipulations.

Sealed master-slave manipulators are to be provided for use in carrying out this operation. These instruments are to be medium capacity, extended reach instruments with maximum versatility.

D. Viewing Windows

Visual observation of the operations carried out within the shielded facility will be absolutely essential because of their complex nature. Large high-density viewing windows are to be provided at each manipulator station to simultaneously permit close observation while shielding personnel.

3.0 SAFETY CONSIDERATIONS

3.1 HAZARDS

3.1.1 Sodium Reactivity

Metallic sodium in small quantities is apt to be handled throughout the FFTF analytical facility. It will be continuously handled in the inert atmosphere portions of the facility, both shielded and unshielded. Chemically, sodium is a very reactive material and will spontaneously react with many substances at room temperature. For example, it will spontaneously ignite when in contact with water and can detonate when in contact with chlorinated hydrocarbons such as carbon tetrachloride. Its reactive nature makes burning sodium extremely difficult to extinguish.⁽¹⁾ Common fire extinguishing agents such as carbon dioxide and water act as oxidizing agents, increasing the reaction intensity rather than extinguishing the blaze. During the burning of sodium, large quantities of dense white smoke are produced which create inhalation hazards and impede fire fighting by blocking visibility.

3.1.2 Hydrogen Buildup

Hydrogen gas is liberated during the dissolution of sodium in alcohol and as the result of the reaction of sodium with water or water vapor in the atmosphere. Accumulations of this liberated gas in the air-atmosphere shielded facility could constitute an explosion hazard. Under normal conditions, only small quantities of sodium will be dissolved at one time and a large gas flow rate will almost completely preclude the creation of hazardous conditions. Hazardous conditions may be created under abnormal conditions such as the dissolution of a large quantity of sodium during disposal operations or during a prolonged curtailment of gas circulation

through any of the confined systems such as the air-atmosphere shielded facility. It is therefore necessary to recognize the existence of sources of hydrogen within the facility and the consequent explosion hazard when designing the facility.

A similar problem may be created by the inadvertent escape of combustible gases into the air-atmosphere shielded facility. A notable source of such gas would arise if the decision were made to include atomic absorption spectroscopic facilities in the shielded facility. The flames used for excitation in this technique range all the way from propane-air flames to pre-mixed acetylene-nitrous oxide.

3.1.3 Radioactive Contamination Control

All of the samples to be analyzed will be potentially contaminated with radioactive material, including plutonium, and some of the sodium samples will be highly radioactive from the presence of large amounts of ^{24}Na . Sound, standard contamination-control equipment and procedures are to be incorporated in the design of the facility.⁽²⁾ These are to include control of all air flow directions, an absolute filter complex and several unusual construction features. Particular emphasis is to be placed on the problems of contamination control during a metallic sodium fire.

3.2 PRECAUTIONS

Most of the precautions required to minimize safety hazards resulting from the extreme reactivity of the sodium are in the area of operating procedures.⁽³⁾ However, some special design features are required to cope with this problem, e.g., the preclusion of conventional automatic sprinkler fire fighting equipment. If any molten metal is to be piped within the facility, air and water piping must be arranged so that there is absolutely no possibility of mixing these

materials with the sodium. Consideration should be given to the removal of large quantities of dense white smoke in the event of a sodium fire. Convenient space must be provided for the storage of large metal-X chemical fire extinguishers. Special considerations must be given to the problems of safe storage of alkali metal samples and to the safe disposal of metallic wastes. Drip trays are essential under any vessels or lines containing molten metal.

4.0 STATE-OF-THE-ART REVIEW

To meet FFTF analytical needs complex manipulations must be carried out remotely behind shielding in an inert atmosphere. The technology for remote operation is well developed.⁽²⁾ The technology for inert atmosphere operation is also well developed. Very limited experience has been accumulated, however, in which the two technologies have been combined. Probably the largest such facility is ANL's Fuel Cycle Facility⁽⁷⁰⁾ at the National Reactor Testing Station. Maximum use will be made of the information gained and the techniques developed by ANL at NRTS in the design and operation of the FFTF analytical facility.

4.1 SAMPLE RECEIVING AND STORAGE

Highly radioactive solid sodium samples will most likely be received from the loop sampling stations in large shielded casks.⁽⁴⁾ Transfer from these casks to the inert-atmosphere shielded facility will be complicated by the need for an air lock to prevent contamination of the inert atmosphere by air. Air lock protection can be accomplished in one of three ways:

- A separate evacuable and flushable air lock can be provided directly between the cask and the inside of the inert-atmosphere shielded facility.
- The cask can be adequately sealed to the facility wall and used as the evacuable, flushable container.
- Entry into the inert-atmosphere shielded facility can be made in two steps with transfer from the cask into the air-atmosphere shielded facility followed by a transfer into the inert atmosphere through the air lock between the two facilities.

Each of these three approaches has its advantages and disadvantages. Use of a separate air lock is the most direct and

provides for the most versatile and rapid operation. The manipulations required are complicated considerably by having to handle the sample twice; once from the outside of the radiological shielding to place it into the air lock, and once from the inside to remove it. Radiological shielding of personnel during the time the sample is in the air lock also creates a problem.

The second approach, using the cask as an evacuable container, eliminates the handling problem and simplifies the shielding problem. It does require an air-tight seal or flexible connection between the cask and the shielded facility wall, and an air-tight plug in the wall. Because of the weight of the cask and plug such seals may be difficult to operate and maintain. Provisions must also be made for the evacuation and flushing of the cask.

The third approach, inserting the sample into the air atmosphere portion of the shielded facility and then transferring it through the air lock between the two facilities, is by far the simplest from a mechanical point of view. Almost every sample will be opened initially in the inert atmosphere portion of the shielded facility, however, and the procedure therefore has the disadvantage of adding an extra transfer to the sample receiving operation.

Some critical samples will have to be stored for future reference. This need arises from two sources. It is desirable to have a "backup" sample because quite frequently during later data-digestion and interpretation phases of the investigation questionable data are encountered, and re-examination of the sample is desirable to confirm or correct the data point. A second need for "backup" samples arises in the case of more basic research when, nearing the

end of an extended series of experiments, the investigator discovers that a hitherto unrecognized, and therefore unmeasured variable is of significance. With "backup" samples available this variable can frequently be measured and thus the value of the test extensively increased.

Storage will be needed for a large number of samples. To conserve valuable in-cell space a system should be developed for removal to a storage facility outside the cell of those samples which have aged long enough for decay of the short-lived ^{24}Na . Also, an excellent system of sample cataloging will be required.

4.2 SAMPLE SECTIONING AND MEASURING

4.2.1 Cutting Methods

Representative aliquots must be removed from the as-received samples. There are at least five methods which have been used for this operation:⁽⁴⁾

- Sectioning
- Extrusion
- Coring
- Pouring
- Dipping bucket.

Of these secondary sampling techniques sectioning and extrusion are by far the most practical and are proposed as methods in the CAL.

Sectioning is accomplished by cutting off lengths of the tube section. In order to ensure a representative sample it is imperative that complete cross sections of the sample tube be taken and that the initial tube be filled in such a manner as to prevent longitudinal segregation. Section length is sometimes used as a measure of sample size during

oxygen determination by amalgamation. The absence of significant voids is assumed when this is done. When section length is used as a measure of sample size it is essential to obtain accurate, reproducible lengths. Several sectioning techniques have been suggested and used⁽⁴⁾ such as a lathe, an abrasive cut-off-wheel, a motorized two-wheel tubing cutter,⁽⁵⁾ and a conventional manually operated tubing cutter with a stop device. Because of its simplicity of operation the manually operated tubing cutter is most readily used in a remote operation and is proposed for use in the CAL facility.

4.2.2 Sealing Methods

Samples must be completely protected from atmospheric contamination. Short exposure of a few minutes to the high purity inert atmosphere of the shielded facility will not significantly contaminate the sodium analytical sample, but prolonged exposure of several hours or more to even this ultrapure atmosphere jeopardizes the sample purity. It is therefore necessary to reseal the sample tube between samplings if more than a few hours are to elapse between samplings, and to reseal all "backup" samples for storage. This can be done by either capping with a fitting (such as a Swagelock) or by recrimping the tubing where the sodium is exposed. Recrimping is the simplest operation to perform remotely and would probably create the higher integrity seal. It is therefore proposed for use in the CAL. A large amount of data has been accumulated which indicates a high potential for contamination of exposed sample ends no matter how carefully the samples are handled.^(6,7) The end sections of both new and recrimped samples will routinely be discarded during each sampling.

4.2.3 Extrusion

The stainless steel jacket must be removed from the sodium sample for many determinations such as carbon and corrosion product measurements. There are several ways to accomplish this, but the method most readily remotized is extrusion of the sodium from the jacket.⁽⁸⁾ This method has been used successfully to remove the jacket and measure the sample size simultaneously.⁽⁹⁾

There is some question concerning the validity of samples obtained in this manner, however, because a complete cross section of the tube is not taken. By its very nature, extrusion leaves a thin layer of sodium adjacent to the tubing wall. Segregation of impurities within a sodium sample is known to occur during cooling and solidification. If a significant concentration of impurity occurs adjacent to the sampler wall, a sample taken by extrusion will not be representative of the sodium being sampled. Extensive research and development is required to define the significance of the radial segregation problem and to permit complete confidence in extrusion as a sampling method.

Extruders suitable for work with the long, small diameter sample tubes obtained during bypass loop sampling are large, somewhat awkward devices but have been operated satisfactorily. Once the sample is extruded, it is usually cut with a hot wire. It can also be cut with an ordinary blade.

4.2.4 Weighing

An accurate measurement of sample size is essential if quantitative estimates of the impurity levels are to be obtained. Both the extruder and sectioner must be calibrated and their performance periodically checked against a primary

standard. Direct weighing of the sodium provides such a standard, and a balance which is easily operated remotely is provided for this operation. A second acceptable method of standardization is measurement through a chemical titration. The proposed facility also has the capability to be standardized by this second technique by passing a sample (whose size is to be measured) through the dissolution and automatic pH titration stations.

4.2.5 External Sample Cleaning

Oxide and some dirt accumulates on the outer surface of the bypass loop sample tube during handling and sampling. This oxide can react with the amalgam during oxygen determination by amalgamation, and appear as oxygen in the sodium. The effect is small normally but can be significant when samples containing low oxide content are being analyzed. A method of removing the stainless steel oxide from the surfaces of the sample tube before it is opened is therefore desirable. Such oxide can be removed chemically by an acid etch, by a fused salt etch or by heating in an amalgam. All of these operations are difficult or impossible to perform remotely in an inert atmosphere, and mechanical cleaning by straightforward buffing is proposed. Extensive effort will have to be made to confine and control the dust formed during this operation.

4.3 OXYGEN DETERMINATION BY AMALGAMATION

4.3.1 General Discussion of Oxygen Methods

Many methods have been developed for the determination of oxygen in sodium. Procedures have been published based on all of the following techniques:

- Mercury Amalgamation⁽¹⁰⁻¹⁷⁾
- Vacuum Distillation⁽¹⁸⁻²⁰⁾

- Vacuum Fusion⁽²¹⁾
- Complete Fluoridation⁽²²⁾
- Ammonia Dissolution^(23,24)
- Activation^(25,26)
- Wurtz Reaction⁽²⁷⁻³⁰⁾
- Gettering⁽³¹⁾
- Spark Source Mass Spectrometry⁽³²⁾
- Isotope Dilution.⁽³³⁾

Of these many methods, only amalgamation and vacuum distillation are being widely used and accepted at this time. A similar conclusion is reached in several recently published review articles on the analytical chemistry of sodium.⁽³⁴⁻³⁸⁾ Rejection of the other methods is based on the fact that their blanks are high or that either their sensitivity or specificity is inadequate. Capability for the determination of oxygen by both amalgamation and vacuum distillation is to be included in the facility in an effort to provide the maximum amount of information concerning oxygen-containing impurity species, and to permit ready cross checks with other facilities which have only one of the methods in operation. If future developments indicate a need, minor modifications will permit the preparation of samples for activation analysis or mass spectrometry and these procedures can readily be adopted.

4.3.2 Procedure Description

Sodium is highly reactive and will "getter" oxygen from the atmosphere, water vapor, or metallic oxides on equipment surfaces. The "gettered" oxygen subsequently appears as sodium oxide and results in an incorrect estimate of the oxygen content of the sample. Extreme care is therefore necessary during the entire procedure to protect the sample from these

sources of contamination. All operations in which sodium or sodium amalgam are present are therefore to be carried out in a highly purified inert atmosphere and the amalgamation vessels and all equipment with which the amalgam has contact are to be scrupulously cleaned.

Samples are to be prepared by cleaning the oxide from their outer surfaces mechanically. A small section at the end of the sample is then cut off and discarded. A measured section is cut off and placed in an amalgamation vessel, the stainless steel tubing of the bypass loop normally being left on the sample. Considerable disagreement exists between investigators concerning the need for removal of oxide which may be present on the stainless steel jacket prior to amalgamation. The resolution of this disagreement will have to await the results of laboratory investigations.

The vessel is agitated and amalgamation allowed to proceed to completion. Considerable heat is liberated during amalgamation and the vessel becomes quite warm. The maximum temperature of the amalgam must be held within reasonable limits to prevent errors arising from the thermal decomposition of NaH and the liberation of oxygen from the glass of the amalgamation vessel by the attack of sodium. Temperature limitation is accomplished by controlling the rate of amalgamation, the rate of heat dissipation, and the size of the mercury heat sink. In glove-box operations, this temperature rise is sufficient to create handling difficulties.

Once amalgamation is complete, the vessel is allowed to stand quiescent in the upright position until the sodium oxide has floated to the top of the amalgam. As much as

possible of the amalgam is then drained away and the remaining amalgam separated from the oxide by repeated mercury washes. All impurities which are insoluble in mercury are isolated by this technique and may or may not be interpreted as sodium oxide impurity depending on the method used for the final measurement.

Two techniques are commonly used to measure the oxide after separation is complete. They are acid-base titration and flame photometry. Acid-base titration is recommended for FFTF application because it is by far the easiest to remove and because the equipment necessary is required for other determinations and will therefore be available. This procedure is an aqueous titration and cannot take place in an inert atmosphere without seriously contaminating that atmosphere. The amalgamation vessel is therefore transferred to the air-atmosphere portion of the shielded facility through an air lock. Here, water is added to the amalgamation vessel and any soluble material is completely washed into a titration vessel. The solution is titrated with standard acid using an automatic titrator. Only the sensing unit of the titrator is placed inside the radiological shielding; the electronics and recorder remain in the operating gallery.

After washing, the amalgamation vessel has significant amounts of water adsorbed on its surfaces. This adsorbed water will react with the amalgam of the next sample and appear as an error in the determination unless it is removed. The vessel is therefore returned to the inert atmosphere portion of the shielded facility through a special air lock which is also a vacuum drying oven. A supply of dried vessels will probably be stored in this chamber.

The mercury contained in the amalgam must be recovered. Recovery can be performed while the amalgam contains

significant amounts of ^{24}Na , and is therefore highly radioactive, if a remote recovery system is provided. However, it will probably be desirable to provide sufficient storage space to allow a two to three week period for the ^{24}Na to decay prior to recovery and thus permit the reprocessing of the amalgam in an unshielded facility.

4.3.3 Advantages

The amalgamation method has the following points to recommend it as a procedure for use on highly radioactive sodium.

- Recent work has markedly reduced the blank size and improved the detection limit.⁽¹⁰⁾
- It is a long-standing, well proven method for non-radioactive sodium analyses.
- It is the simplest of the methods to remotize.
- It is capable of handling a large number of samples.
- A large backlog of experience has been accumulated with the method on non-radioactive sodium with which to compare results.
- The method is capable of precise reproducible results.

4.3.4 Disadvantages

Amalgamation has the following disadvantages or weak features when applied to highly radioactive systems:

- Large amounts of mercury are used. This requires considerable clean-up effort and creates a significant hazard from reagent contamination.
- The method is non-specific; many impurities appear as sodium oxide.
- It is an indirect measurement. A quantity related to the oxygen rather than the oxygen itself is measured.

4.3.5 Expected Capability

Under exacting conditions amalgamation is capable of determining oxygen concentrations in sodium down to 1 ppm with a precision of about ± 1 ppm at the lower concentration levels. With reasonable equipment and some support in amalgamation clean-up, one operator can perform as many as six determinations per shift. Some information concerning oxygen-containing impurity species can be obtained by combining information obtained by amalgamation with that obtained by vacuum distillation.

4.4 OXYGEN DETERMINATION BY VACUUM DISTILLATION

4.4.1 Need for Vacuum Distillation

Vacuum distillation has been used for some time as an impurity concentration technique for oxygen determination in alkali metals. It is usually close-coupled to the sampling operation to permit the early removal of the sodium and to minimize the possibility of contamination during sample handling and transfer. It has sufficient merit as a general separations technique, however, to warrant its application in other than a close-coupled manner. Capability for vacuum distillation separation is provided for several reasons: the method can yield additional information regarding oxygen species, it may be needed to obtain comparison numbers with other locations using the method, and it has applicability to the isolation of impurities for purposes other than oxygen determination.

4.4.2 Procedure Description

The sample for vacuum distillation is either taken in or placed in a nickel cup which in turn is placed inside a glass distillation vessel. The distillation vessel is attached to a vacuum system and evacuated to a pressure of 10^{-5} mm of

mercury or less. The sodium is then distilled while closely monitoring its temperature. To date much of the work with distillation has been done with resistance heating but extensive difficulty has been encountered in obtaining adequate heat transfer to the nickel cup and sodium. Induction heating overcomes this problem and permits a much more rapid distillation.

Careful control of sodium temperature is required to both ensure complete distillation (which is obviously essential) and to prevent loss of impurities by volatilization due to overheating. This control is obtained through monitoring coupled with control of the energy input. Provisions must be made to condense the distilled sodium and to prevent sodium vapor from reaching the vacuum pump.

Once distillation is complete, the distillation vessel is disassembled and the cup containing the separated oxide removed from the vacuum system. For most applications, the sample need not be protected from atmospheric contamination from this point on. The actual quantitative measurement of the separated oxide is performed exactly as is done when the amalgamation separation is used and is described in Section 4.3.2. The distilled sodium must be removed from the distillation vessel and the vessel cleaned and baked out for the next separation.

4.4.3 Advantages

The vacuum distillation method for oxygen determination in sodium has the following advantages or strong points to recommend it for use on highly radioactive samples:

- Distillation is a very "clean" separation technique; e.g., no reagents or solvents are required and therefore the chances of contamination of the sample

from this source are small.

- The method can be used for the separation and identification of impurities other than oxide.
- Distillation is effective in precipitating and retaining dissolved oxygen.
- Radioactivity can be removed early in the sample handling process.

4.4.4 Disadvantages

The vacuum distillation procedure has the following weaknesses or disadvantages when applied to the analysis of radioactive sodium.

- The apparatus and manipulations required are quite complex and therefore difficult to remotize.
- The method is non-specific, and impurities other than oxygen appear as oxide. It does not measure oxygen but measures a related variable such as alkalinity or sodium.
- Loss of carbon and oxygen may occur during distillation, by volatilization or entrainment.
- Errors are possible and even probable from incomplete distillation.
- Extensive difficulty has been encountered in obtaining an accurate measure of the sample size when a dipping cup sample is used.
- The procedure is time consuming and the operation of simultaneous multiple systems difficult. Therefore, the number of determinations possible in a given span of time is limited.
- Thermally unstable impurities may be destroyed or altered during the distillation process.

4.4.5 Expected Capability

Vacuum distillation is capable of determining oxygen concentrations in sodium down to 5 ppm with a precision of about $\pm$ 5 ppm. With a single apparatus, a maximum of two determinations per shift is possible. The method or equipment can also be used to concentrate or separate impurities for special investigations such as species identification by X-ray diffraction or infrared absorption.

4.5 TOTAL CARBON DETERMINATION

4.5.1 General Discussion of Carbon Methods

Several methods have been developed for the determination of total carbon in sodium. Procedures have been published based on the following techniques:

- High-temperature combustion^(39,40,41)
- Low-temperature combustion⁽⁴²⁾
- Wet oxidation⁽⁴³⁻⁴⁵⁾
- Flux oxidation^(46,47)
- Isotope dilution mass spectroscopy⁽⁴⁸⁾
- Activation analysis.⁽⁴⁹⁾

The first four methods are based on the conversion of carbon to a gaseous form, generally carbon dioxide, followed by the quantitative measurement of the gas produced. Several techniques have been used for the gas measurement such as gas chromatography, mass spectroscopy, infrared analyses, gravimetry, manometry, and chemical titration. The most sensitive and specific is gas chromatography and it is recommended for CAL application.

The isotope dilution mass spectrometric method is complex, relatively new, and offers no outstanding improvements in performance. Activation analysis, particularly

using high energy particle activation, offers the possibility of considerable improvement in detection limit, but the method is by no means adequately developed at this time. Future developments may dictate the inclusion of this technique in FFTF capabilities. The blanks associated with the wet oxidation method are prohibitively high for total carbon determinations on good quality sodium.

Of the remaining methods (low-temperature oxidation, high-temperature oxidation and flux oxidation) there is little difference in the operations required to carry out an analysis.

The difficulties which may be encountered in placing any of these methods in a remote facility are very similar and no firm selection is required at this time. High-temperature oxidation is arbitrarily selected and discussed in this report as typical.

4.5.2 Procedure Description

The stainless steel tubing in which sodium samples are normally taken naturally contains carbon which must be removed before carbon analysis. The sodium is removed by extrusion from the jacket. Future developments may dictate the substitution of another dejacketing method which does not leave behind a thin layer of sample adjacent to the tubing walls. This layer leaves some doubt concerning the validity of the extruded sample because of possible segregation of carbon along the tubing walls during sample solidification.

Extruder plunger movement may be used as a measure of sample size. The extruded sodium is placed in a preconditioned quartz boat which is then packed into a quartz sample tube using quartz wool. The sample tube is then loaded into the combustion tube. All of these operations are carried out in a carbon dioxide-free atmosphere to prevent sample contamination. Specially cleaned and maintained tools are required to

prevent contamination by organic material. The sample tubes and boats are preconditioned in a separate furnace because of the long times required. Combustion is carried out using oxygen diluted with helium, dilution being required to control the rate of the reaction and to prevent the liquification of oxygen during the trapping of the carbon dioxide at liquid nitrogen temperatures. Large volumes of gas are used during combustion and it must be of extremely high purity if blanks are to be maintained at acceptable levels. It is therefore passed through a purification train upstream of the combustion furnace to remove carbon-containing materials. The purification train consists of a column of heated copper oxide followed by a column of ascarite. The copper oxide catalyzes the oxidation of any organic material present and the ascarite removes the carbon dioxide produced during the oxidation.

Combustion of the sodium is carried out at a low temperature to prevent distillation of sodium to colder portions of the apparatus prior to complete combustion. Once combustion is complete, the furnace temperature is raised to 1100 °C and held for a minimum of one-half hour to completely decompose any sodium carbonate which may be formed during the combustion by the reaction between carbon dioxide and sodium oxide. During the high temperature ignition the sodium oxide also reacts with the surrounding quartz boat and wool which act as an acidic flux and aid in the liberation of the carbon dioxide.

Carbon dioxide is scrubbed from the combustion gases with an efficient trap held at liquid nitrogen temperature. This trap is plumbed into both the combustion train and the carrier gas train of a gas chromatograph in such a manner that it can readily be switched from one to the other.

Once all the CO_2 from the combustion is collected, the trap is switched to the carrier gas stream, warmed, and the released CO_2 measured chromatographically. Many other methods have been used to measure the carbon dioxide formed but the chromatographic method has a higher sensitivity and greater specificity than the others and is therefore recommended.

4.5.3 Advantages

The combustion method has been widely used and accepted for the determination of carbon in sodium. Therefore a large backlog of data is available for comparison of results. The method is specific for carbon, it has a high sensitivity, and it ensures the measurement of the total carbon content of the sample.

4.5.4 Disadvantages

The high-temperature combustion procedure has the following weaknesses or disadvantages when applied to the analyses of radioactive sodium:

- The apparatus and manipulations required to perform an analysis are quite complex and difficult to remotize.
- The maintenance of low blanks is difficult.
- No information concerning carbon species is provided by the method.
- The procedure is lengthy and with a single experimental setup, a maximum of two determinations per shift can be handled.
- Danger of incomplete recovery of carbon dioxide arises from two sources: gettering by sodium oxide formed in the cooler portions of the tube from the oxidation of sodium which inadvertently distilled during combustion; and incomplete decomposition of the sodium carbonate in the melt.

4.5.5 Expected Capability

The combustion method is capable of determining total carbon in sodium down to 5 ppm with a precision of about $\pm$ 5 ppm at the lower levels. With a single set of equipment, a maximum of two determinations per shift is possible. Little or no information can be obtained to identify carbon species.

4.6 CARBON SPECIES DETERMINATION

4.6.1 Need for Species Identification

Combustion techniques will provide a good measure of the total carbon content of sodium samples. There is significant evidence to indicate that the variable which is important in mass transport and carbonization may not be total carbon concentration, but rather the concentration of a particular species of carbon impurity.^(37,38,39) Methods for the identification and quantitative measurement of carbon species are therefore needed and are presently under development in several laboratories.

4.6.2 General Discussion of Present State-of-the-Art

The species of carbon-containing impurity which contribute most significantly to carbonization and carbon transfer by sodium have not yet been identified. Such identification is difficult without the aid of analytical procedures which are specific for each species. The more probable species of carbon impurities present in sodium are dissolved carbon, elemental carbon, sodium carbide, sodium cyanide, sodium carbonate and possibly a few of the more thermally stable organic compounds. Many attempts are being made to develop analytical methods which are specific for

each of these species.⁽⁵⁰⁾ Most methods depend upon aqueous hydrolysis followed by the identification and measurement of the hydrolysis products. A typical scheme might be outlined as follows:

- Dissolution (without neutralization) in water, and identification and measurement of the off-gases (usually chromatographically). In this step, carbide carbon is converted to acetylene, methane and other hydrocarbons.
- Acidification of the solution and identification and measurement of the off-gases. In this step, carbonate and cyanide are liberated.
- Wet oxidation of the remaining solution with a reagent such as Van Slyke's reagent. In this step, forms of elemental carbon and any more stable organic compounds are converted to carbon dioxide and measured.

Many of the methods of carbon species measurement now in the development stage do not require highly specialized equipment and can be adapted to remote operation.

4.7 TOTAL HYDROGEN DETERMINATION

4.7.1 General Discussion of Hydrogen Methods

Hydrogen has been determined in sodium by thermal decomposition,^(51,52) by isotope dilution,^(53,54) and by a modified amalgamation procedure.⁽⁵⁵⁾ The oldest and most widely used method is that of thermal decomposition followed by diffusion of the hydrogen out of a thin-walled sealed container (usually steel) and finally, quantitative measurement. Measurement has been performed both chromatographically and manometrically.

The isotope dilution technique is sensitive and specific. When deuterium is used as the diluent, mass spectrometric measurements are required and these add to the complexity

and cost of the method. When tritium is used as the diluent, low-level counting is required for the determination. The high radiation backgrounds caused by the large amount of ^{24}Na present in FFTF coolant will make this counting difficult if not impossible. Thermal decomposition is therefore suggested as the primary method to be used to determine hydrogen. A modified amalgamation technique has recently been developed⁽⁵⁵⁾ in an effort to provide additional information regarding the hydrogen-containing impurity species present. This information is of considerable value to the corrosion and materials investigators and the method is therefore included as a backup hydrogen method. The amalgamation procedure is quite lengthy and therefore each determination costly. Unless multiple experimental assemblies are employed, only a limited number of determinations are possible.

4.7.2 Thermal Decomposition Procedure Description

The sodium is removed from the stainless steel tubing of the sampling loop, usually by extrusion or melting, and placed in a precleaned steel container. An inert atmosphere is required for this and the subsequent welding operations. A lid is placed on the container and the container hermetically sealed with a weld. The container is loaded into a glass vessel which can be attached to a vacuum system and the vessel closed in the inert atmosphere. The remainder of the operation may be carried out in an air atmosphere.

The sealed vessel is attached to a vacuum system and the entire system evacuated and degassed. Tests are performed to be certain the steel capsule is hermetically sealed. The system is evacuated and the portion of the vacuum

system containing the bulb and a pressure measuring device is isolated from the pump. The steel capsule containing the sodium is heated to decompose the hydrogen-containing compounds and to permit the diffusion of the liberated hydrogen through the steel. The pressure rise in the isolated portion of the system is observed and used as an indication of the completion of the diffusion process. Frequently, it is assumed that hydrogen is the only gas capable of diffusing through the steel container and the amount is estimated directly from the manometric measurements. Many investigations require a method which is more specific for hydrogen. Several measuring techniques have been developed to obtain this specificity. One is to pump the liberated gas, using a Toepler or similar pump, into a gas chromatograph sampler valve and measure the hydrogen content chromatographically. Specificity is thus obtained from the chromatographic separation coupled with the column retention time measurement.

4.7.3 Advantages

The thermal decomposition method has been widely used and accepted as a method for the determination of total hydrogen in sodium. Therefore, a large backlog of data is available for comparison of results. It can be made quite specific by the use of chromatographic measurement. It has good sensitivity and fair precision. A reasonable number of samples can be handled with a single equipment setup. It is simple and the most readily remotized of hydrogen methods.

4.7.4 Disadvantages

The thermal decomposition method for total hydrogen in sodium is subject to high blanks, and early workers questioned

the recovery it achieves. Recent work by Dutina⁽⁵⁶⁾ and associates has dispelled much of the doubt about recovery, however. The method provides no information concerning the chemical species of hydrogen impurity present.

4.7.5 Expected Capability

With the proper detection system, the thermal decomposition method is capable of determining hydrogen in sodium down to the 1 ppm level with adequate precision. The decomposition step is time-consuming and as a consequence, only three to four determinations can be made per shift with a single apparatus.

4.8 HYDROGEN DETERMINATION BY AMALGAMATION

4.8.1 Need for Method

The amalgamation method of hydrogen determination permits the determination of total hydrogen and also distinguishes between that combined as hydroxide and that which is present in a non-hydroxide form.⁽⁵⁵⁾ It thus provides extensive information concerning the hydrogen-containing impurity species as well as the hydrogen content.

4.8.2 Procedure Description

A specially designed amalgamation vessel is attached to a vacuum system and preconditioned by refluxing an amalgam in it until hydrogen is no longer liberated. It is then transferred to an inert atmosphere box, opened, and a 5 g sample of the sodium to be analyzed is placed in a side arm above the amalgam. The vessel is reattached to the vacuum system, evacuated to under 1 μ pressure and the system tested for leaks. The sodium is dropped into the amalgam and the amalgam refluxed for 5 min at 200 °C. The amalgam is allowed to cool and the vessel filled

with argon, a 1 ml sample of gas is taken and its hydrogen content determined gas chromatographically. If hydrogen is detected, the reflux cycle is repeated until no additional hydrogen is evolved. Three cycles are usually required to complete the removal of hydrogen. The sum of the amounts of hydrogen liberated in this series of refluxes is considered non-hydroxide hydrogen.

Using the same vessel and amalgam, hydroxide hydrogen is evolved and measured by performing a similar series of reflux cycles at an argon pressure of 500 mm of mercury and a temperature of 370 °C.

4.8.3 Advantages

Considerable information is obtained during this analysis about the hydrogen-containing impurity species present in the sample.

4.8.4 Disadvantages

The primary disadvantage of this procedure is that it is complex and will therefore be difficult to remotize. It is also time-consuming and therefore will be expensive.

4.8.5 Expected Capability

The amalgamation procedure can differentiate between and determine hydroxide and non-hydroxide hydrogen at concentrations down to 2 ppm with a standard deviation of better than ± 1 ppm. The method is time-consuming, and only one determination can be made per shift per equipment setup.

4.9 NITROGEN DETERMINATION

4.9.1 General Discussion of Nitrogen Methods

The only widely used method for the determination of nitrogen in sodium is by Kjeldahl distillation.⁽⁵⁷⁾ This

method measures only nitride nitrogen. Recently, considerable interest has developed in other nitrogen containing impurity species, notably cyanide. This interest has prompted some efforts to extend the Kjeldahl method to include other species.⁽⁵⁸⁾

4.9.2 Procedure Description

Sodium reacts violently with water. Therefore, for safety reasons, it is frequently dissolved in ethyl alcohol and the alcoholic solution diluted with water. The nitrogen is distilled from the basic aqueous solution as ammonia and trapped in a boric acid solution. No addition of base or reducing agent is required as the metallic sodium of the unknown performs both functions.

The distilled ammonia may either be titrated with standard hydrochloric acid or determined colorimetrically using Nessler's reagent.⁽⁵⁹⁾ Facilities for both forms of quantitative measurement are provided. It is hoped that the method may be extended to other nitrogen species, notably cyanide, by acidification of the residue followed by a second distillation and colorimetric determination of the distilled cyanide.

4.9.3 Advantages

Kjeldahl distillation is a sensitive, precise and quantitative method for the determination of nitride nitrogen in sodium. It is time consuming, but simultaneous multiple determinations can readily be made. The method has been used extensively for the determination of nitrogen in alkali metals and a large backlog of experience and data is therefore available for the comparison of results. The required operations are simple enough to permit ready remotization.

4.9.4 Disadvantages

The method measures only nitride nitrogen and does not see other species such as dissolved nitrogen and cyanide. These species may be important variables in the test run. It requires an aqueous dissolution of metallic sodium and therefore entails the safety hazards associated with such a dissolution.

4.9.5 Expected Capability

The Kjeldahl distillation method can measure nitride nitrogen in sodium over the concentration range from 1 to 100 ppm with a standard deviation of about 1 ppm. Simultaneous multiple distillations will permit one operator to make up to five determinations per shift.

4.10 SAMPLE DISSOLUTION

4.10.1 General Discussion

Many analyses and separations are best accomplished in the aqueous system. Sodium is a highly reactive metal and its direct dissolution in water is hazardous. Several techniques, most of which involve an intermediate or indirect step, have been developed to minimize these hazards.

The technique which is best suited to analytical requirements is an initial dissolution in alcohol followed by dilution of the alcoholate with water. Ethanol, methanol or isopropanol can be used but 95% ethanol is the most generally accepted reagent.

During dissolution, care must be taken to ensure quantitative recovery of the sodium and its impurities if a quantitative measure of the original impurity level is to be obtained. Splattering and fog carry-over must be avoided. Volatilization

of impurity hydrolysis products must also be avoided. This latter requirement is almost impossible to meet in some instances as there are a number of potentially volatile species. For instance, carbon can be lost either as volatile hydrocarbons formed by the reaction of carbide with water or as hydrogen cyanide or carbon dioxide formed during the acidification of an aqueous solution. Nitrogen can also be lost as ammonia or cyanide.

Dissolution involves the use of relatively large quantities of reagents. This fact, combined with the fact that impurity levels in reactor grade sodium are very low, requires the reagents to be of extremely high purity in order to avoid significant sample contamination.

4.10.2 Procedure Description

Pepkowitz⁽⁶⁰⁾ first described a method for the safe dissolution of sodium for analytical purposes. Since the initial publication, many variations on the technique have been developed.⁽⁶¹⁾ A typical procedure might be outlined as follows:

- The dissolution vessel is cleaned and dried.
- The dissolution vessel is filled with argon gas, the sodium sample placed in it, and the vessel closed. Frequently, operations up to this point are carried out in an argon atmosphere box.
- A slow continuous purge of argon gas is passed through the vessel to remove hydrogen as ethyl alcohol is added.
- When the reaction is complete, the vessel contents are washed into a volumetric flask with water, neutralized and made to volume.

4.10.3 Expected Capability

Dissolution is quite rapid and multiple simultaneous operations are readily performed. A single operator will

be able to handle as many as 20 to 30 samples per shift if proper equipment is available.

4.11 CHEMICAL SEPARATIONS

4.11.1 Need for Separations

Some impurities have a marked influence on sodium chemistry and corrosion at very low concentration levels. Therefore, trace level determinations are essential to the interpretation of test results. In order to determine trace levels of impurities, it is almost always necessary to first perform a concentration and separation step. This step has two useful functions; first, it concentrates sufficient trace material to permit measurement and secondly, it removes the bulk of the interfering materials.

4.11.2 General Discussion of Separations Techniques

Two of the more powerful means of separating impurities from sodium, namely, amalgamation and vacuum distillation, have already been discussed. They are extensively used because they avoid conversion to the aqueous system and the attendant danger of sample contamination by reagent addition. These methods, however, are not adequate for the separation of all the trace impurities which must be determined. Conversion to the aqueous phase, therefore, becomes desirable. In the aqueous system two very versatile separations methods become available--solvent extraction and ion exchange.

4.11.2 Ion Exchange Separation Procedure

Many metallic elements can be separated by ion exchange. The separation of the rare earth elements is a notable example of the power of the technique. The reagents and conditions required for the isolation of each element or group of elements

differ, but the primary operations in all the separation processes are similar. They may be outlined as follows:

- The oxidation state, acid concentration, and complexing nature of the solution containing the ions to be separated, are adjusted.
- The ion of interest is loaded on the top of a resin column.
- A chromatographic separation and elution of the materials on the column is effected with the aid of an eluent solution.
- The eluent is monitored as it comes off the column and the portion containing the ion of interest separated mechanically.
- The column is reprepared for the next separation.

In analytical work, the columns used are very small and the flow rate is frequently adjusted by applying air pressure or vacuum to the system. The separation factors are usually quite high and frequently little or no use is made of the chromatographic effect.

4.11.4 Solvent Extraction Separation Procedure

The reagents and conditions required for the solvent extraction separation of each element or group of elements are different but the primary operations involved in all the separations are similar. They may be outlined as follows:

- The valence state, acid concentration and complexing nature of the solution containing the ions to be separated are adjusted.
- The aqueous phase containing the ion to be extracted is contacted with a small amount of organic solvent containing the extracting agent. Vigorous contacting is required for a significant (five min) length of time.

- The two phases are allowed to disengage and are carefully separated.
- The aqueous phase is washed several times with small amounts of the organic extractant and the wash solution added to the original extractant.
- Frequently the organic phase is diluted to a predetermined volume and quantitative measurements performed directly on it. In other determinations, the element of interest is stripped back into an aqueous phase before measurement. Stripping is accomplished by a process very similar to the original organic extraction.

In analytical solvent extraction separations, small volumes are involved and systems with extremely high separation factors are chosen wherever possible. This separation factor is usually limited by the ability of the operator to separate the two phases. Phase separation is particularly difficult in a remote operation where good visibility and fine mechanical controls are frequently lacking.

4.11.5 Expected Capability

Both ion exchange and solvent extraction analytical separations are rapid, and multiple simultaneous operations can readily be performed. A single operator will be able to handle as many as 20 identical separations in a shift, or 10 each of two different separations. Most determinations of fission products, corrosion products, and fuel materials will be preceded by an aqueous separation step. In some instances, separation will provide sufficient decontamination from radioactivity to permit the removal of the sample from the shielded facility for the remaining steps in the analyses.

4.12 SPECTROPHOTOMETRIC DETERMINATIONS

4.12.1 General Discussion of Spectrophotometric Methods

Very sensitive, highly specific colorimetric procedures have been developed for most of the elements of the periodic chart, through the use of the proper separation procedure combined with the proper colorimetric reagent.⁽⁶²⁾ Although most methods have not been specifically applied to the sodium system nor adapted to remote operation, they are simple enough that remotization can be readily accomplished. Spectrophotometric instruments have been developed specifically for remote use on highly radioactive solutions⁽⁶³⁾ and much of this development work may be directly applicable to FFTF needs.

4.12.2 Typical Procedure Description

A separate spectrophotometric determination is almost always required for each element to be determined. These determinations will differ slightly from element to element primarily in the preliminary separation and in the color developing reagent. The basic operations required for most determinations are the same, however, and can be outlined as follows:

- After the separation step (which was described in the previous section) the valence state, degree of complexity, and the acid content of the sample are readjusted if this is necessary for the formation of the correct colored complex.
- A colorimetric reagent is added and the color developed.
- The sample is placed in one cell of a dual beam spectrophotometer and its absorbance compared to that of a standard. The standard is usually a reagent blank.

- A calibration curve, which is prepared and periodically checked by running standards through the analytical procedure, is used to relate the measured absorbance to the trace impurity concentration.

4.12.3 Advantages

Absorption spectrophotometry is a well developed art with an abundance of published procedures which can be adapted to FFTF needs. The operations required to perform an analysis are simple and readily adapted to remote use. It has adequate sensitivity, high precision, and can be made quite specific.

4.12.4 Disadvantages

Absorption spectrophotometry is not a survey type operation. Separate determinations are required for each element of interest. As a consequence, when multiple trace metal impurity surveys are desired, the spectrophotometric approach is time-consuming and costly.

4.12.5 Expected Capability

Spectrophotometric procedures are capable of determining all of the stainless steel corrosion products, nitrogen, and most other non-radioactive trace-metallic impurities with the sensitivity and precision required for FFTF loop tests. The number of survey type samples which can be handled will definitely be limited because of time and cost considerations. As a consequence, most impurity surveys will be made by emission spectrographic techniques. Only those samples which show unusual impurity concentrations or behavior will be handled spectrophotometrically.

Considerable interest is presently being shown in the use of atomic absorption spectroscopy for the determination of trace-metallic impurities in sodium.⁽⁶⁴⁾ The technique does not completely solve the problem of survey type determinations,

as an individual hollow cathode lamp is required for each element. The method offers sensitivities and precisions equal to or greater than spectrophotometry in some instances, however, and its adoption may become advisable. Modification of the equipment installed for spectrophotometry to permit atomic absorption measurements can readily be accomplished by replacing the light source with a bank of hollow cathode tubes and the absorption cell with the proper burner. The operation of a flame within the shielded facility will create significant problems and hazards, however, and proper engineering will be required to overcome these.

4.13 EMISSION SPECTROGRAPHIC DETERMINATIONS

4.13.1 General Discussion of Emission Spectrographic Techniques

Trace metallic impurity surveys are routinely made by emission spectrographic techniques, particularly in the ferrous and aluminum metallurgy fields. Such surveys on alkali metals are complicated by the highly reactive nature of the metal. To avoid this reactivity problem, the general practice has been to convert the metal to the chloride and perform the analyses on the salt or to separate the impurities by carrier precipitation.⁽⁶⁴⁾ This conversion requires significant amounts of reagents (such as alcohol, distilled water, and hydrochloric acid) and the problem of sample contamination by reagent, particularly at very low impurity levels, is great. Significant danger of contamination also exists because of the lengthy sample handling required.

Emission spectrographic techniques are inherently limited to precisions of about $\pm 30\%$ unless special excitation and plate handling techniques are employed. This

precision may not be adequate for some coolant analytical applications.

4.13.2 Typical Procedure Description

The steps required to perform a typical emission spectrographic determination of trace impurities in sodium may be outlined as follows:

- A weighed sodium sample is dissolved in alcohol, neutralized with hydrochloric acid, and evaporated to dryness under a heat lamp.
- A portion of the sodium chloride is mounted in a graphite electrode, the electrode is mounted in the source unit, and the sample is then burned using a dc arc.
- The light produced during the excitation is passed through the radiological shielding to the slit of the spectrograph, dispersed, and photographed in the spectrograph.
- The photographic plate is developed under rigidly controlled conditions and the line intensities read, using a densitometer.
- The measured intensities are interpreted with the aid of calibration curves.

A single plate shows lines of most elements of interest and the measurement of an additional element requires only the measurement of an additional line intensity.

4.13.3 Advantages

The emission spectrographic method is rapid and simultaneously produces information concerning the concentrations of a large number of elements. In the case of sodium the sample preparation step is time-consuming. Good standards are available in the form of spiked samples of ultrapure sodium chloride. Remote operation of a spectrographic source unit has been carried out at several locations, notably ORNL.

4.13.4 Disadvantages

For many elements, spectrographic procedures are not sensitive enough to meet the exacting demands of a test program using very high purity sodium. Variations in source excitation and photographic recording limit the precision of the method to about $\pm 30\%$ unless special measures are taken to maintain stability. This level of precision may be inadequate for initial test runs. Spectrographic equipment is expensive, delicate, and requires special care for optimum performance.

4.13.5 Expected Capability

A single spectrograph will suffice to survey a large number of samples for trace metallic impurity levels. Multiple simultaneous sample preparation will be required if a large number of samples are to be handled. Primary limitations will be on meeting the sensitivity and precision requirements.

4.13.6 Alternate Approaches

Several methods are under development which are potentially capable of determining trace levels of metallic impurities in sodium with either greater sensitivity or precision than emission spectrography. Notable among those with greater precision are spectrophotometry (discussed in Section 3.12) and atomic absorption spectroscopy.⁽⁶⁴⁾ Both require individual determinations for each element of interest and are therefore not well suited for survey type work. Atomic absorption has the added disadvantage of requiring instrumentation which is awkward to operate in a remote, shielded, sealed facility.

Spark source mass spectroscopy has been considered as a means of improving the sensitivity and detection limits

for trace impurities in sodium metal.⁽⁶⁵⁾ An improvement in detection limit of at least one and probably two orders of magnitude can be expected through the use of this approach. The equipment required is expensive, however, and no improvement in precision can be expected over that obtained by emission spectroscopy as the two methods have the same inherent precision-limiting features.

The method is in its early stages of development and if this development proves satisfactory it will be desirable to apply spark source methods to selected FFTF sodium samples.

4.14 RADIOCHEMICAL DETERMINATIONS

4.14.1 General Discussion of Radiochemical Methods

Many trace impurities in the reactor and test loop sodium and cover gas will be radioactive. Of these impurities, the concentrations of fission and activation products will be of interest to the test engineer. By far the most sensitive methods for the determination of these isotopes are based on radiochemical counting techniques. High specificity can be obtained by a combination of chemical separation and particle energy analysis.

Because of the high sensitivity of the counting instruments, it will be possible to handle and count many samples with little or no radiological shielding once the proper chemical separation is completed. In the few cases in which the sample activity is high enough to require shielding, counting will be performed through the wall of the shielded facility with the aid of a special beam collimating device.⁽⁶⁶⁾

The ^{24}Na isotope is a high energy gamma-emitter with a short half-life. Because of the high neutron fluxes encountered in the FFTF, large quantities of ^{24}Na will be present in coolant

samples. ^{24}Na will contribute an overwhelming radiation background unless excellent separations techniques are used. In fact, adequate separation may be impossible for some of the isotopes which are low-energy emitters or which are present in low concentrations.

4.14.2 Typical Gamma Energy Analysis Procedure Determination

The steps of a typical gamma energy analytical procedure may be listed as follows:

- After separation and dilution, the sample is placed in a small vial or other container of fixed reproducible geometry and the vial placed inside the well of a crystal detector.
- The scintillations from the detector are then observed for a predetermined time using photomultiplier tubes.
- The output pulses from the photomultiplier tubes are fed into a multichannel analyzer which sorts, counts and records them as a function of energy.
- The resulting spectrum is interpreted either visually or electronically. Frequently, when complex mixtures of gamma emitters are involved, spectrum stripping techniques are used in this interpretation.

When higher resolution is required, a lithium drifted, germanium diode detector may be substituted for the sodium iodide crystal.

4.14.3 Typical Alpha Energy Analysis Procedure Description

The steps in a typical alpha energy analysis procedure are similar to those of a gamma energy analysis except for the preparation of the sample and the detection of the alpha particles. Great care is required in sample preparation to avoid problems with self-absorption of the alpha particles.

4.14.4 Typical Total Beta Count Procedure Determination

The beta activity of ^{24}Na prohibits the practical use of β -counting techniques on loop sodium directly out of the reactor. Some useful data on long-lived β -emitters may be possible after sufficient time has been allowed for the decay of ^{24}Na (roughly two weeks). A typical procedure which might be used to obtain the data follows:

- The sodium sample is allowed to "cool" for from 10 days to two weeks to permit the decay of ^{24}Na .
- A known weight of sodium is then dissolved in a small volume of ethanol. As water is added, the solution is made acid with nitric acid, and the sample is brought to volume with distilled water.
- An aliquot of the solution is direct mounted on a stainless steel plate.
- The beta activity of this plate is counted with a beta proportional counter.

4.14.5 Expected Capability

Radiochemical determinations are among the most sensitive measuring techniques; therefore radioactive impurities can be determined at extremely low concentration levels. Gamma emitters in particular can be measured at low levels provided ^{24}Na interference can be overcome. Gamma energy analysis also lends a high degree of specificity to the radiochemical methods. The methods are rapid and sample counting should not limit the sample throughput rate, although some of the required chemical separations are lengthy and may place a limit on the sample throughput.

Radiochemical determinations will be particularly useful in measuring fission product, activation product and fuel material concentrations in loop sodium and cover gas.

4.15 PARTICULATE AND TOTAL SOLIDS DETERMINATION

Methods have not yet been fully developed for the isolation and identification of particulate matter contained in sodium. Several general approaches to the problem of particulate isolation are being investigated. The most obvious of these is the mechanical separation of the particles of interest by means of a device such as a filter or ultracentrifuge.^(67,68) Mechanical separation has the distinct advantage that it is the method least likely to change the chemical nature of the particles under investigation.

Dissolution of the sodium followed by filtration is also being investigated as a means of particle isolation. Ammonia,⁽⁶⁹⁾ mercury, and water have all been used as solvents. A major disadvantage of the dissolution approach is that it presents considerable hazard of changing the chemical nature of the particulate matter under investigation. Finally, distillation of the sodium has been used as a means of particle isolation. This approach isolates material in addition to particulate matter and may also alter the nature of the particles through heating.

Once the particles have been isolated, their identification is by no means straightforward. They are quite small and may be highly radioactive. Infrared absorption spectroscopy, X-ray diffraction, chemical analyses, and optical microscopy have all been applied to their identification in nonradioactive sodium.

4.16 UNSHIELDED SODIUM ANALYSIS

4.16.1 Need for Capability

Remote operations within large radiological shields are costly and time-consuming; therefore only operations in which radiation levels are high should be performed in

such facilities. Wherever possible, analyses should be performed in simpler contact-operated equipment. The FFTF analytical laboratory will be required to handle large numbers of sodium samples which are not highly radioactive and therefore a complete unshielded analytical facility will be required in addition to the shielded facility already described. The samples requiring little or no shielding will arise primarily from two sources: those which have not yet been exposed to a high neutron flux and those for which sufficient time is available to permit the decay of ^{24}Na before analysis.

4.16.2 Typical Glove Box Facility

Many of the operations to be carried on in the unshielded analytical facility will require an inert atmosphere, and conventional inert atmosphere glove boxes are proposed. High capacity recirculating gas purification systems capable of maintaining oxygen, water and CO_2 contents below 2 ppm are to be associated with each glove box. The purity of each atmosphere is monitored with an automatic in-line monitor. In many cases, the boxes will serve the additional function of radioactive contamination containment, particularly for alpha-containing samples. The proper performance of this function will require special filters and other devices in the gas purification trains.

4.16.3 Facilities Desired

Operations essentially parallel to all those performed in the shielded facility will also be performed in a more or less conventional manner on samples which do not have a high radiation field associated with them. Those requiring inert atmospheres will be performed in glove boxes and the rest will be done on the laboratory bench top or in fume hoods.

Many operations will require the use of small portable forms of radiological shielding such as drilled lead brick sample holders and portable lead glass window shields. Most will require contamination control measures because of the potential presence of particulate radioactive materials.

4.16.4 Dual Function Facilities

The facility is designed to locate most of the more complicated analytical instrumentation outside the contamination and radiation zones. These instruments can therefore perform dual functions in the sense that a single piece of equipment can be used for the analysis of the highly radioactive and the non-radioactive samples, thus permitting a significant saving in capital investment.

4.16.5 Expected Capability

The unshielded sodium analytical facility will be capable of performing the following determinations on non-radioactive or slightly radioactive sodium:

- Total oxygen by amalgamation
- Total oxygen by vacuum distillation
- Total carbon by combustion
- Total hydrogen by diffusion
- Hydrogen species by amalgamation
- Nitrogen by Kjeldahl process
- Metallic impurities survey by emission spectrography
- Many trace impurities by absorption spectrophotometry or atomic absorption spectroscopy
- Fission product, activation product, and fuel materials determination by radiochemical techniques
- Particulate and total solids identification
- Sample preparation for nonroutine investigations

- Many nonroutine determinations such as the concentrations of other alkali metals and halogens
- The preparation of secondary analytical standards.

Sample loads have not yet been defined and the facility is therefore not "sized" in this study. When sufficient information becomes available to permit sizing of the facility, it must be remembered that sample throughputs for most determinations can be varied only in fixed increments as each new physical setup is added to the system.

4.17 COVER GAS ANALYSIS

4.17.1 Need for Capability

Experience to date with commercial samples of low level (<10 ppm) gas mixtures has not been wholly satisfactory. If commercially available mixtures are to be used as the reference gases on the process instruments, periodic verification of their composition will be necessary.

It is expected there will be times when the process instruments indicate the gas composition is out of the normal range. At such times, it is desirable to be able to collect samples of the gas and test it on instruments other than the process one. This use plus the one above indicate the Chemistry Analysis Laboratory should have the capability to analyze sodium cover gas samples for impurities.

4.17.2 Typical Procedure Description

The sample cylinder whose contents are to be analyzed is connected to the sample inlet manifold (through a pressure reducing valve if it is a high pressure sample). The portion of the system, between valves, that has been exposed to air is evacuated, closed off from the vacuum pump, and the sample admitted. The inlet valve to the instrument is opened and

the gas allowed to flow for 10 minutes (or whatever period is necessary to reach equilibrium). Then the sampling valve is actuated and an instrument reading or scan is obtained. This is repeated over a period of time to verify establishment of equilibrium. From the instrument reading and the calibration curve the concentration of the measured constituents is calculated.

4.17.3 Expected Capability

It is expected that this part of the laboratory will be set up with such instrumentation as necessary to verify the composition of the reference standards used with the process instruments and to confirm the occurrence of off-standard samples when it is necessary to confirm process instrument indications of trouble. This capability can be provided by a gas handling system and a general purpose instrument such as a gas chromatograph or a mass spectrometer.

5.0 MAINTENANCE

5.1 PREVENTIVE MAINTENANCE

The CAL facility will require normal preventive maintenance such as pump oiling, belt replacement, valve seat replacement, jet cleaning and replacement, instrument maintenance, etc. The operations involved are not expected to be unusual nor to markedly affect the conceptual design of the system.

5.2 GENERAL MAINTENANCE

General maintenance of the CAL will involve several unusual operations because of the combined requirements of shielding and atmosphere control. Notable among these operations will be the changing of such components as CWS filters, lights, manipulators, and air lock seats. The design of the facility must permit any in-cell maintenance to be readily accomplished with manipulators and special tools. Provisions must be made to ensure the integrity of the inert atmosphere during any maintenance which involves entry into that atmosphere. Such components as vacuum pumps, motors, electrical equipment, and electronic instrumentation are to be located on the outside of the shielding wherever possible.

In the case of manipulator breakdown requiring the temporary or permanent replacement of a manipulator, a distance of 18 ft free of obstructions will be required from the front face of the cell to permit the removal of the manipulator. In most cases (unless a major facility shutdown is planned) a bag-in and bag-out procedure will have to be employed in order to maintain the integrity of the inert gas atmosphere and to provide contamination control. A minimum free space of 15 ft will be required at the rear of the cells for the purpose of

building contamination control "green houses" when it is necessary to open main cell doors for the replacement of equipment on major decontamination and maintenance.

The design of the facility should make maximum use of these areas required for maintenance purposes during normal periods of operation. Most of the laboratory operations which do not require shielding can be performed in the area in front of the operating cells. Much of the storage space required for operation can be provided by the area behind the operating cells.

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APPENDIX A

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APPENDIX B

DESIGN AND DEVELOPMENT REQUIREMENTS

DESIGN AND DEVELOPMENT REQUIREMENTS

Areas in which design and development effort will be required are numerous. They can be divided into two major categories: (1) the area in which basic information is lacking and additional research is required; and (2) areas in which the basic technology is available but in which a development effort must be made to adapt the technology to the unusual requirements of the FFTF program. It is expected that the latter effort will be made primarily at PNL while the bulk of the former effort will be expended elsewhere in the LMFBR program.

The primary need for the development effort arises from the need for some analytical numbers a short time after coolant sampling, thus requiring the analysis of highly radioactive sodium. The analytical procedures recommended in this study have all been developed and tested on non-radioactive sodium using contact or glove box operations. Virtually none of them have been undertaken successfully in a remotely operated shielded facility. Therefore intensive design and development effort will be required to ensure that safe, reliable, and adequate systems, procedures, and equipment are available for remote operation by the time of FFTF startup. This program will have to entail the following major steps:

- The laboratory mockup and bench testing of equipment and procedure designed to perform each determination remotely.
- The testing and modification of each procedure in a remote mockup using remote techniques. This initial testing will be performed in the absence of sodium, radioactivity, and shielding.

- Continued testing through the performance of actual analysis in a second mockup which is capable of handling sodium and which is as exact a duplicate as possible of the shielded facility except for the shielding. Enough sodium analyses should be performed to develop any "art" required. This activity will require the presence of an inert atmosphere. Sufficient experience should be accumulated in the final cell mockup to permit the writing of operational manuals and procedures for remote work.
- The design of the actual high level facility will be based on the information gained during these activities.
- Most of the equipment should be transferred directly from the final test facility to the shielded operating facility and placed in actual service.

In addition to the design data the development program will generate answers to numerous questions concerning procedures and conditions. Typical examples of the information which will be obtained might be:

- An evaluation of the various total carbon methods for remote operation and the selection of the most desirable one.
- The definition of the most advantageous mercury cleaning procedure, the major choice being between a cooling period followed by cleaning in a contact maintenance plant or immediate cleaning in a remote maintenance plant.
- Selection of a location for the sampling ports in the various chromatographic analysis. Again the major choice is between location within the shielding or directly on the chromatographic column.

- An evaluation of the merits of locating the spectrophotometer inside the shielded facility.
- The definition of the alcohols and acids most required for routine operation and of the best manner in which to supply these reagents, the primary candidates being reagent bottles or larger tank facilities.
- Information concerning the operation of an atomic absorption flame within an enclosed shielded facility, particularly with regard to practicality and safety.

There are many areas in which existing technology is inadequate to meet program needs, and basic research is desirable. Programs designed to satisfy most of these needs are presently underway in the existing LMFBR program. Maximum use will be made of any new and improved technologies developed. Typical improvements which can be anticipated are:

- The development of more sensitive methods for the interstitial elements, particularly carbon, hydrogen, and oxygen to permit precise measurements to be made in the 0.1 to 10 ppm concentration range.
- The development of the technology necessary to measure particulate matter and total solids in the sodium.
- The development of the technology necessary for the measurement of carbon species concentration in the very low ranges.
- A clearer definition of the sampling problems created by longitudinal and radial segregation of impurities within the sample and the development of methods of overcoming or minimizing these problems.
- The development of more sensitive methods for the determination of metallic impurities, particularly the corrosion products. Emphasis should be placed on the

development of survey techniques such as spark source mass spectrometry.

- The development of techniques for the preparation of primary and working analytical standards for each of the constituents of interest.

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APPENDIX C

INTERFACE REQUIREMENTS

INTERFACE REQUIREMENTS

Sampling

One of the most important interfaces between the CAL and the FFTF facilities will be that with the sodium sampling system. The sampling system to be employed will have a significant impact on reactor cost and is under study.⁽⁴⁾ Firm decisions regarding its nature have not been made but three alternatives seem likely:

- Individual samplers at each loop with large transfer casks to transport the samples to the CAL.
- One or more centralized sampling locations at the reactor with a specialized system to transport samples to the CAL. Transportation could be based on casks, pneumatic tubes or gravity.
- A centralized sampling system integrated with the CAL.

Each approach has its advantages in terms of capital investment, ease of operation, complexity and quality of resulting samples.

This study assumes that sampling will occur at the reactor as in the first two instances and that samples will be transported to the analytical facility--probably by a cask system. The assumption is dictated by the complexities involved in thermostating and maintaining the long sample lines required for an integrated system and on the almost certain reduction in sample validity the use of such long lines would entail. Assuming the cask system of sample transfer, the primary interfacial point will be the sample receiving air lock. Principal problems will be shielding during sample entry, contamination control and inert atmosphere quality control. They are described in Section 2.2.1.

Information Feedback

A prime function of the CAL is to obtain data defining irradiation environment. This data of course must be closely correlated with the individual experiment. Several similar sets of samples will be generated simultaneously. In-cell operations are difficult and complex and individual samples will be difficult to follow. A simple, clearly defined, and readily read labeling and cataloging system must be used at the sampling station to ensure unequivocal sample identification. A careful cataloging and accounting system will be used during analysis to ensure the correct correlation and reporting of the data obtained.

Inert Atmosphere

A large flow of highly purified argon gas will be required to maintain the quality of the atmosphere in a portion of the CAL. Normally in facilities of this nature this flow is supplied by a recirculating gas purification system with a small makeup flow.

There will be an interface between the reactor inert gas system and the CAL inert gas supply. The extent of this interface is not defined at this point.

Waste Disposal

Highly radioactive acidic and basic aqueous waste solutions will be generated in the CAL. Radioactive organic and solid sodium wastes will also be generated. There will be a significant interface between the CAL waste disposal mechanisms and those of the reactor system.

General Facilities

All general facilities such as electrical, air conditioning, water, and heat will have normal interfaces with those of the reactor system.

BILL OF MATERIAL

For

ANALYTICAL FACILITY

SK-3-12703

<u>Part No.</u>	<u>Req'd</u>	<u>Description</u>	<u>Remarks</u>
1	1	<u>SHIELDED INERT GAS CELL - FUNCTIONS:</u> Sodium Sample Preparation and Storage, Oxygen Amalgamation, Carbon Determination, Vacuum Distillation	119 Ft ² Approx.
2	1	<u>SHIELDED AIR ATMOSPHERE CELL - FUNCTIONS:</u> Hydrogen Determination, Hydrogen Amalgamation, Separation, Dissolution, Absorption Spectrophotometric, Modified Kjeldahl Nitrogen, Radiochemical Counting.	126 Ft ² Approx.
3	1	<u>AIR CONDITIONED ROOM (UNSHIELDED)</u>	144 Ft ² Approx.
4	1	<u>DARK ROOM (UNSHIELDED)</u>	108 Ft ² Approx.
5	1	<u>MANIPULATOR STORAGE AND REPAIR</u>	378 Ft ² Approx.
6	1	<u>FRONT FACE AREA AND COLD LAB</u>	936 Ft ² Approx.
7	1	<u>AIRLOCK</u>	120 Ft ² Approx.
8	1	<u>REAR FACE AREA AND GENERAL STORAGE</u>	1,110 Ft ² Approx.
9	As Req'd	<u>WALLS - Hi Density Magnatite Concrete, Typical Thickness 3 ft. In-between cells, 18 in. thick.</u>	
10	2 Sets	<u>MANIPULATOR - Sealed, Model E, Med. Duty, Extra Reach.</u>	\$40,000/Set Approx.
10A	2 Sets	<u>MANIPULATOR - Model E, Med. Duty, Extra Reach</u>	\$20,000/Set Approx.
11	4	<u>WINDOWS - Hi Density With Non-Darkening Glass Inner Face.</u>	

Part No.	Req'd	Description	Remarks
12	1	<u>AIRLOCK</u> - Sealed, Evacuable, for Sodium Sample Transfer.	
13	1	<u>CELL DOOR</u> - MEEHANITE, 18 in. Thick, Sealed	
14	1	<u>CELL DOOR</u> - MEEHANITE, 18 in. Thick, Non-Sealed	
15	1	<u>AIRLOCK</u> (GENERAL PURPOSE) - Sealed, Evacuable	
16	1	<u>AIRLOCK</u> (GENERAL PURPOSE) - Non-Evacuable.	
17	As Req'd	<u>WORKING TRAY AND SUPPORT STAND</u>	
18	1	<u>SODIUM SAMPLE STORAGE</u> - Automatic Indexing, 108 Samples, Shielded, Inert Gas Purging.	
19	1	<u>COUNTERBALANCE WEIGHT</u> - For Storage Lid	
20	1	<u>BALANCE</u> - Grams	
21	1	<u>FUNNEL</u> - Mercury Drain	
22	2	<u>MERCURY DRAIN AND DECAY TANKS</u>	
23	1	<u>MERCURY PURIFIER</u>	
24	1	<u>MERCURY SUPPLY TANK</u>	
25	2	<u>RACK</u> - For amalgam Vessels, Oxygen Determination	
26	1	<u>VACUUM DISTILLATION APPARATUS</u>	
27	2	<u>POWER SUPPLY</u> - For Induction Heater	
28	1	<u>HELI-ARC WELDER</u> - For Hydrogen Determination	
29	1	<u>WELDING GENERATOR</u> (Power Supply)	

<u>Part No.</u>	<u>Req'd</u>	<u>Description</u>	<u>Remarks</u>
30	As Req'd	VACUUM PUMP - Needed for Parts No. 11-14-36-37	
31	1	FURNACE - Preconditioning for Carbon Determination	
32	2	FURNACE - Combustion for Carbon Determination	
33	3	CHROMATOGRAPH COLUMN OVEN	
34	3	CHROMATOGRAPH ELECTRONICS	
35	1	TOOL RACK - For Carbon Determination	
36	1	AIRLOCK (GENERAL PURPOSE) - In-Cell Transfer W/Transfer Mechanism, Sealed, Inert Gas, Evacuatable, Heated	
37	1	AIRLOCK (GENERAL PURPOSE) - W/Transfer Mechanism, Sealed, Inert Gas, Evacuatable.	
38	3	HELIUM GAS SUPPLY - Carrier gas supply for Chromatograph	
39	2	HELIUM TANK SUPPLY - For Carbon Combustion	
40	2	OXYGEN TANK SUPPLY - For Carbon Combustion	
41	2	GAS HEATER AND MIXER - For Carbon Train	
42	2	GAS PURIFICATION TRAIN - For Carbon Train	
43	1	AUTOMATIC TITRATOR	
44	1	ALCOHOL TANK SUPPLY - For Dissolution.	
45	1	DISTILLED WATER SUPPLY TANK - For Solution. Make-up.	
46	1	INDUCTION HEATER - For Hydrogen.	

<u>Part No.</u>	<u>Req'd</u>	<u>Description</u>	<u>Remarks</u>
47	1	<u>ORGANIC WASTE SLURP LINE</u>	
48	1	<u>AQUEOUS WASTE SLURP LINE</u>	
49	2	<u>VACUUM TRAIN RACK - For Hydrogen Determination</u>	
50	1	<u>GLASSWARE SUPPLY</u>	
51	1	<u>SPECTROPHOTOMETER</u>	
52	1	<u>SPECTROPHOTOMETER CELLS AND OPTICS</u>	
53	1	<u>SPECTROPHOTOMETER LIGHT SOURCE</u>	
54	1	<u>VORTEX MIXER - For Solvent Extrac- tion Separation.</u>	
55	1	<u>IN-CELL GAMMA SPECTROMETER SOURCE</u>	
56	As Req'd	<u>VACUUM PUMP - Needed for Parts No. 26-49-68-69-70 and 71.</u>	
57	1	<u>EMMISION SPECTROGRAPH</u>	
58	2	<u>ARC-SPARK STAND</u>	
59	1	<u>SODIUM IODIDE WELL CRYSTAL DETECTOR - Lead Brick Shielded</u>	
60	1	<u>MULTI-CHANNEL ANALYZER AND AMPLIFIER</u>	
61	1	<u>DENSITOMETER</u>	
62	1	<u>ALPHA ENERGY ANALYZER</u>	
63	1	<u>ALPHA ENERGY DETECTOR</u>	
64	1	<u>PLATE DEVELOPER</u>	
65	1	<u>REFRIGERATED COLD STORAGE CHEST - For Plates.</u>	
66	1	<u>SINK CABINET</u>	

<u>Part No.</u>	<u>Req'd</u>	<u>Description</u>	<u>Remarks</u>
67	6	<u>WORK BENCH</u>	
68	1	<u>CARBON DETERMINATION - GLOVE BOX</u>	
69	1	<u>OXYGEN DETERMINATION - GLOVE BOX</u>	
70	1	<u>NON-ROUTINE DETERMINATION - GLOVE BOX</u>	
71	1	<u>GAS SAMPLE HANDLING RACK</u>	
72	2	<u>CABINET</u>	
73	As Req'd	<u>TRACK</u>	
74	1	<u>HOIST (ELECTRIC) 2-TON</u>	
75	1	<u>HAND AND FOOT COUNTER</u>	
76	2	<u>FUME HOOD</u>	

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APPENDIX D

DRAWINGS

D-1

Neg 0682486-1

SK-3-12703

Preliminary Design Analytical Facility

BNWL-821

STEP #1

SODIUM SAMPLE IS CUT INTO DESIRED LENGTHS

STEP #2

CUT SPECIMEN IS DROPPED INTO A AMALGAMATION FLASK CONTAINING MERCURY

STEP #3

FLASK IS CAPPED THEN SHAKEN AND ROTATED SIMULTANEOUSLY

STEP #4

THE FLASK IS PLACED IN A RACK AND THE Na_2O (SODIUM OXIDE) FLOATS TO THE TOP THE Na AMALGAM IS DRAINED OFF A SMALL PORTION OF THE AMALGAM + Na_2O IS LEFT IN THE FLASK.

TO MERCURY CLEANUP AND RECYCLE

TANKS ARE USED ALTERNATELY AS WASTE AND DECAY VESSELS

STEP #5

THE Na_2O IS WASHED 3 TIMES WITH FRESH MERCURY. OPERATIONS OF STEPS 3 & 4 ARE REPEATED. THE THIRD MERCURY WASH IS CHECKED FOR SODIUM.

STEP #6

PASS FLASK THRU AIRLOCK

SHIELDED INERT ATMOSPHERE

SHIELDED AIR ATMOSPHERE

STEP #7

FLASK IS CLEANED AND RETURNED THRU VACUUM DRYING AIRLOCK

WASH OXIDE FROM FLASK

PUT IN TITRATION VESSEL

UNSHIELDED AIR ATMOSPHERE

TITRATE WITH AUTO-MATIC TITRATOR

Neg 0682486-2

Amalgamation Oxygen Determination

D-2

BNWL-821

STEP #1

SODIUM SAMPLE IS CUT TO DESIRED LENGTHS IN SHIELDED INERT ATMOSPHERE.

STEP #2

SODIUM IS EXTRUDED FROM A CUT SECTION INTO A NICKEL CUP.

STEP #3

NICKEL CUP WITH SODIUM SAMPLE IS PUT INTO A VACUUM DISTILLATION APPARATUS. HERE IT IS INDUCTION HEATED AND DISTILLED IN A VACUUM.

STEP #4

VACUUM DISTILLATION APPARATUS IS DISASSEMBLED AND THE NICKEL CUP REMOVED.

STEP #5

SHIELDED INERT ATMOSPHERE

PASS NICKEL CUP THRU AIRLOCK

SHIELDED AIR ATMOSPHERE

THE RESIDUE LEFT IN THE NICKEL CUP IS WASHED WITH DISTILLED WATER INTO A GLASS BEAKER.

RETURN NICKEL CUP THRU VAC. DRYING AIRLOCK

PUT IN TITRATION PROBES

UNSHIELDED AIR ATMOSPHERE

TITRATE ON AUTO-MATIC TITRATOR

STEP #1

QUARTZ BOAT IS ASSEMBLED INSIDE A QUARTZ LINER AND PACKED WITH QUARTZ WOOL

STEP #2

QUARTZ LINER IS THEN PRE-CONDITIONED BY PLACING IT IN A FURNACE AND HEATING TO 1100°C IN A STREAM OF PURIFIED OXYGEN AND HELIUM.

STEP #3

MEASURED SODIUM SAMPLE IS EXTRUDED INTO THE QUARTZ BOAT

STEP #4

THE QUARTZ BOAT CONTAINING THE SODIUM SAMPLE IS RE-ASSEMBLED IN THE QUARTZ LINER.

← SHIELDED INERT ATMOSPHERE →

QUARTZ LINER IS INSERTED INTO THE COMBUSTION TUBE OF THE FURNACE. THE SODIUM IS BURNED AND THE CO₂ COLLECTED.

THE COLLECTED CO₂ IS MEASURED ON A GAS CHROMATOGRAPH

D-4

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Neg 0682486-7

Total Carbon Determination

D-5

BNWL-821

Neg 0682486-11

Hydrogen Determination

D-6

BNWL-821

Neg 0682486-5

Amalgamation Hydrogen Determination

OR DETERMINE NH_3 SPECTROPHOTOMETERICALLY USING NESSLER'S REAGENT

Neg 0682486-9

Modified Kjeldahl Nitrogen Determination

D-7

BNWL-821

PASS SODIUM SAMPLE
THRU AIRLOCK

EXTRUDE SODIUM FROM
TUBING INTO A
ERLENMEYER FLASK

DISSOLVE IN
ALCOHOL

ADD DISTILLED
WATER

NEUTRALIZE
WITH ACID

STEP #6

MAKE TO VOLUME IN
VOLUMETRIC FLASK

Neg 0682486-12

Sample Dissolution

D-8

BNWL-821

D-9

BNWL-821

Neg 0682486-5

Chemical Separations

STEP-#1

RECEIVED SODIUM SAMPLE FROM
DISSOLUTION OR SEPARATION STATION

STEP-#2

ADJUST CONCENTRATION AND
VALENCE STATE OF SAMPLE

← SHIELDED AIR ATMOSPHERE →

STEP-#3

ADD COLORIMETRIC
REAGENT

STEP-#4

Neg 0682486-8

Absorption Spectrophotometric Determination

D-10

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STEP #1

SODIUM SAMPLE RECEIVED
FROM DISSOLUTION STATION

STEP #2

STEP #3

EVAPORATE TO DRYNESS
← SHIELDED AIR ATMOSPHERE →

MOUNT SALT IN A
GRAPHITE ELECTRODE

STEP #4

MOUNT ELECTRODES IN SPECTRO-
GRAPHIC SOURCE AND EXCITE

STEP #5

DEVELOPE PLATE
IN DARK ROOM

STEP #6

READ THE DEVELOPED
PLATE ON A DENSITOMETER

D-11

BNWL-821

Neg 0682486-10

Emission Spectrographic Determinations

STEP #1

RECEIVED SODIUM SAMPLE FROM
DISSOLUTION OR SEPARATION STATION

STEP #2

DILUTE WITH ACID

STEP #3

PASS VOLUMETRIC FLASK
THRU AIRLOCK

STEP #4

COUNT AND GAMMA ENERGY ANALYZE
SCANTILATIONS ARE PICKED UP BY PHOTOMULTIPLIER
TUBES WHICH SENDS THEM TO A PULSE HEIGHT
ANALYZER.

Neg 0682486-6

Radiochemical Counting

D-12

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