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RDT Standard

SODIUM REMOVAL PROCESSES

APRIL 1977

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SODIUM REMOVAL PROCESSES

1. SCOPE

This standard establishes requirements for acceptable processes for sodium removal from components or systems intended for reuse. The sodium removal methods described herein are applicable to removal of radioactive sodium, but additional requirements regarding the handling of radioactive components and process streams are not covered in this standard.

2. APPLICABLE DOCUMENTS

The following documents are a part of this standard to the extent specified. The issue of a document in effect on the date of application, including any amendments or other published changes also in effect, shall apply unless otherwise specified. Where this standard appears to conflict with the requirements of the reference document, such conflict shall be brought to the attention of the user for resolution.

2.1 RDT Standards.

RDT F 2-2	Quality Assurance Program Requirements
RDT F 3-2T	Calibration Program Requirements
RDT F 4-20T	Operation and Maintenance Manual
RDT F 5-1T	Cleaning and Cleanliness Requirements for Nuclear Components

2.2 Federal Specifications.

26CFR212	Denaturing of Alcohol and Rum (Code of Federal Regulations)
O-E-00760	Federal Specification for Ethyl Alcohol

2.3 U.S. Bureau of Mines Publications.

Bulletin 503	Limits of Flammability of Gases and Vapors, H. F. Coward and G. W. Jones
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2.4 American Society for Testing and Materials (ASTM) Publications.

ASTM D 2029	Water Vapor Content of Electrical Insulating Gases by Measurement of Dew-Point
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3. DEFINITIONS

3.1 Required Sodium Removal Documentation.

3.1.1 Component Sodium Removal Maintenance Procedure (CSRMP). The document which defines requirements for component disassembly, requalification, and selection of the Sodium Removal Process in accordance with RDT F 4-20. (See 6.2.)

3.1.2 Sodium Removal Facility Operating Instruction (SRFOI). The operating instructions for the facility operator to safely process the component consistent with the CSRMP and this standard. (See 6.3.)

3.1.3 Sodium Removal Process Specification (SRPS). A section of this standard containing requirements for process operations. (See 6.1 and Sections 7 and 8.)

3.2 Frost. Vapor-deposited sodium metal or compounds.

3.3 Inerting. The process of substituting inert gas, such as argon or nitrogen, around sodium-wetted surfaces to prevent reaction with air, oxygen, or water vapor.

3.4 Oscillation. An oscillation is a periodic variation in hydrogen concentration caused by corresponding variation of the water-sodium reaction rate.

3.5 Process Control Criteria. The specific variables and their limits that will allow a controlled reaction.

3.6 Process Vessel. The envelope in which the process takes place. It can be a component shell, a loop, or a container.

3.7 Forms of Residual Sodium.

3.7.1 Crevice Sodium. Sodium remaining in crevices [narrow gaps less than 0.025 in. (0.64 mm) in width] after draining.

3.7.2 Pool Sodium. Sodium accumulating in heels or pockets that have not drained. It has a linear surface dimension greater than 0.025 in. (0.64 mm) and a depth greater than the surface film [>0.002 in. (0.05 mm)].

3.7.3 Surface Film Sodium. The sodium adhering to vertical and inclined surfaces after draining. On a vertical surface drained at 400°F (204°C), the film typically is 1 lb/100 ft² (48.9g/m²) or 2 mils (0.05 mm) thick.

3.8 Reuse. The return of a component to sodium service after sodium removal processing.

4. GENERAL REQUIREMENTS

4.1 Documentation of Processing Requirements. Sodium and sodium compounds shall be removed from components and equipment in accordance with an approved Sodium Removal Process Specification (SRPS), Component Sodium Removal Maintenance Procedure (CSRMP) and Sodium Removal Facility Operating Instructions (SRFOI) as described in Section 6 of this Standard.

4.2 Processing of Installed Components. Unless approved by the facility owner, sodium removal processing of installed components or systems is prohibited.

4.3 Control of Hazardous Materials. Sodium removal facilities shall be equipped and operated to provide for safe, controlled handling and disposal of toxic, flammable, explosive, or other hazardous materials in accordance with local, state, and federal regulations. A dry inert gas atmosphere shall be maintained over metallic sodium that would otherwise be exposed to an oxidizing environment.

4.4 Storage.

4.4.1 Sodium-Wetted Components. All items that contain bulk sodium or surface-adherent sodium shall be stored in a dry argon or nitrogen (<50 ppm water) atmosphere using a storage container, sealed packaging, or by sealing the openings in components that contain sodium. If plastic bags are used for storage of sodium-wetted components, continuous drying or purging of the cover gas is required because of water vapor permeability of plastic bag materials.

4.4.2 Processed Components. All items from which sodium has been removed that are to be requalified and returned to sodium service shall be stored to preclude the introduction of moisture and maintain the state of cleanliness in accordance with the requalification requirements of the CSRMP.

4.5 Requalification. All items from which sodium has been removed and that are to be returned to sodium service shall be requalified in accordance with the requirements of the CSRMP.

5. PROCESS REQUIREMENTS

5.1 Process Description. The basic process steps for sodium removal are:

1. Removal of bulk sodium by draining.
2. Inerting and cooling.
3. Reaction of sodium with the cleaning media.
4. Rinsing of reaction products from the component.
5. Drying of liquid from the component.

Supplementary steps may include mechanical disassembly following reaction of accessible gross sodium, manual removal of sodium, and reprocessing following intermediate disassembly. The specific process sequence for a particular component or equipment should be selected on the basis of minimizing exposure of the component to the caustic environment and the elevated metal temperatures associated with the sodium reaction step.

5.2 Draining. Bulk sodium shall be drained from the item at as high a temperature as practical.

5.3 Inerting and Cooling. The as-drained item shall be inerted and cooled to a temperature range required by the SRPS for the sodium reaction step. The cooling rate shall not exceed the maximum rate specified in the applicable CSRMP.

5.4 Reaction of Sodium. The residual sodium on or in the as-drained item shall be reacted in accordance with the requirements of the selected SRPS. Temperature-indicating devices shall be available on the item to be processed if required by the CSRMP. The concentration of the evolved hydrogen shall be continuously measured and the reaction rate controlled to prevent the occurrence of explosive or flammable concentrations.

5.5 Rinsing. All residual processing fluids and reaction products from the sodium reaction step shall be removed by rinsing in accordance with the requirements of the SRPS, using the fluids specified therein.

5.6 Drying. Subsequent to rinsing the item shall be thoroughly dried by evacuation, hot inert gas purge, or both in accordance with the requirements of the SRPS or CSRMP.

5.7 Reprocessing. Items may be reprocessed through the sodium reaction, rinsing, and drying steps or the reaction step may be interrupted for disassembly operations and then resumed. The total time of exposure to the sodium reaction products shall not exceed any limit specified in the SRPS. (See footnote page 14 and Appendix B.)

Manual removal of sodium can be applied to local surfaces.

5.8 Manual Removal of Sodium. Manual removal of sodium from equipment surfaces by scraping or other mechanical means may be performed in accordance with CSRMP guidance.

6. PROCESS DOCUMENTATION

6.1 Sodium Removal Process Specification (SRPS). The SRPS describes a complete sodium removal process. The SRPS shall include:

1. Process parameters and operating limits for the reaction step.
2. Requirements for processing materials.
3. Process control requirements.
4. Criteria for in-process determination of completion of the reaction step, rinsing step, and the drying operation.
5. Criteria for acceptable level of sodium removal from the processed item.
6. Precautions required to assure safety from process-related hazards.

6.2 Component Sodium Removal Maintenance Procedure (CSRMP). The CSRMP shall specify the sodium removal process and describe how the selected process is to be applied to a specific component. The CSRMP shall include:

1. Requirements for the preparation of the component for sodium removal processing such as the method of draining sodium, provisions for drainage and removal of reactants and reaction products during processing, disassembly required, removal of nonmetallic parts, isolation of connected parts, maximum cooling rate from draining temperature, and installation of process instrumentation. Schematic illustrations or reference to assembly drawings shall be provided as required.
2. Handling requirements.
3. Any requirements for manual removal of sodium before and after disassembly.
4. Requirements for packaging and sealing after processing, storage, or handling.
5. Requalification requirements and acceptance criteria for reuse in sodium, including requirements for further action if bleed-out of sodium occurs after processing.
6. Preparation required for return to service as a result of sodium removal processing, replacement of expendable parts, reassembly, and leak testing.

7. Requirements for process control specimens, if used. (See Appendix F).
8. All hold points where review and approval are required for continued processing, inspection, or other disposition. As a minimum, the concurrence of the quality assurance representative shall be required for continuance of processing after completion of a processing operation, final release for items to be requalified, and resumption of processing following a hold for nonconformance.
9. Identification of location of difficult to drain pockets or crevices.

6.3 Sodium Removal Facility Operating Instructions (SRFOI). The instructions shall reiterate all requirements of the applicable SRPS and the CSRMP and provide detailed instructions for performing all processing operations of the facility in accordance with these requirements. General work rules, precautionary requirements, and equipment operating instructions that are documented for routine facility use may be cited by reference. Quality assurance documents such as process control sheets, process control specimens where required (see Appendix F), and inspection records may be cited by reference or integrated directly into the operating instructions.

6.4 Responsibility for Documentation. The organization responsible for the component to be cleaned shall have primary responsibility for the preparation, review, concurrence, any revisions, and approval of the CSRMP. The organization responsible for the cleaning facility has corresponding responsibility for the SRFOI.

6.5 Records. Copies of the applicable SRPS, CSRMP, SRFOI, process completion records, inspection records, process data records (temperature and exposure times), and nonconformance documentation shall be compiled as a record file by the organization responsible for the component. This record file shall be maintained for the life of the component.

7. WATER VAPOR-NITROGEN SODIUM REMOVAL PROCESS SPECIFICATION

7.1 Process Description. Water vapor in nitrogen gas reacts with residual sodium in a closed, pressure vented vessel or system in the water vapor-nitrogen (WVN) process in accordance with Equation 1.

Other inert gases may be used, but the most experience has been acquired with nitrogen. The reaction rate and the heat of evolution are controlled by limiting the water available for reaction in accordance with the mass action law. The reaction rate is monitored by measuring the hydrogen evolved in the vessel. The inlet temperature is adjusted to maintain a steady state reaction and to avoid thermal excursions which might promote caustic corrosion cracking (see Appendix B). Completion of the vapor phase reaction is determined by cessation of the evolution of hydrogen.

The rinse operation uses hot recirculating water to react sodium and its compounds in crevices and to remove reaction products from the component. Rinsing is repeated until the resistivity of the effluent water reaches a specified level.

The drying operation uses hot inert gas or a vacuum until a specified low moisture content is attained.

A typical schematic diagram of the process apparatus is shown in Fig. 1.

7.2 Process Conditions. The parameters and required criteria for control of the WVN process are summarized in Table 1.

7.3 Equipment Requirements. The process requires a closed system using nitrogen for inerting.

In addition, the system must be capable of metering water vapor into the recirculating nitrogen gas under the conditions specified in Table 1.

The system must conform to OSHA and local safety requirements for flammable hydrogen.

7.3.1 Process Vessel. It is desirable to construct the vessel with an alloy capable of resisting corrosive attack by caustic solutions generated from the reaction of the sodium with water and applicable decontamination acids. Satisfactory durability has been experienced using austenitic stainless steel for sodium removal processing. The vessel shall be designed for at least 17 psia (120 kPa absolute) pressure, and temperature service of at least 250°F (121°C).

Fig. 1. Typical Schematic for WVN Process.

Table 1. Water Vapor-Nitrogen Process Parameters and Control Criteria

	Startup	Vapor Phase Reaction	Reaction Termination	Rinse	Dry
<u>System Feed</u>					
N ₂ (vol. %)	nm	nm	nm	-	100
H ₂ O (vol. %)	1	5 to 10	15	100	-
H ₂ (vol. %)	nm	nm	nm	nm	-
O ₂ (vol. %)	nm	nm	nm	nm	-
Temp. °F	160	160 to 190	160 to 190	180	200 to 250
°C	71	71 to 88	71 to 88	82	93 to 121
<u>Discharge Gas</u>					
N ₂ (vol. %)	nm	nm	nm	nm	100
H ₂ O, dew pt., °F (°C)	nm	nm	nm	-	<-40 (<-40)
H ₂ (vol. %)	<4	<4	<0.0003 ^a	<4	-
O ₂ (vol. %)	<2	<2	<2	<2	-
Temp. °F	m/nc	m/nc	≤190	-	200 to 250
°C	m/nc	m/nc	≤88	-	93 to 121
<u>Discharge Rinse Water</u>					
Resistivity, Ω-cm ^b	-	-	-	>10 ⁵	-
Temp., (°F)	-	-	-	nm	-
(°C)	-	-	-	nm	-
pH	-	-	-	<10	-

^a = hydrogen evolution rate, cubic feet per minute

^b = Ω-cm x 10⁻² = Ω·m

nm = not monitored

m/nc = monitored, but not controlled.

The bottom of the vessel should be designed with a catch tray such that process residue of dripping caustic or sodium will not affect drainage, recirculation, or increase local corrosive attack. The vessel discharge should be processed to be consistent with local environmental requirements.

A lockout system is required to prevent accidental introduction of rinse water into the system before the vapor phase stage of the reaction is complete.

The vessel shall be designed for hot wall construction to avoid condensed moisture dripping on unreacted sodium.

7.3.2 Gas Recirculation System. A blower is required to recirculate the inert gas load of the process vessel every 5 min (300 s) or less.* A heat exchanger to heat or cool is required to control the recirculating gas temperature to a steady state of $\pm 3^{\circ}\text{F}$ ($\pm 2^{\circ}\text{C}$) over the range of 160 to 190 $^{\circ}\text{F}$ (71 to 88 $^{\circ}\text{C}$). The cooler may be used to dry recirculating gas during the drying cycle.

7.3.3 Ventilation System. A system of blower fans or ducts is required to provide air circulation to dissipate accidental accumulation of hydrogen at the top of the building.

7.3.4 Nitrogen Gas System. A nitrogen system is required to supply inerting gas for the sodium removal system. The system shall include a storage tank, converter unit, pressure regulators, flowmeters, safety relief valves, control valves, and piping to provide a flow of dry purge gas to the system for dilution of hydrogen to safe operating levels during inerting, filling, and rinsing operations.

A feed and bleed rate of about 5 percent/min of the vessel volume is recommended.

7.3.5 Instrumentation. Equipment is required to measure and record the following:

1. The temperature of the recirculating inert gas, rinse water, and drying atmosphere.
2. The concentration of the water vapor in the inert gas, the hydrogen in the vessel, and the oxygen in the vessel.
3. The pressure of the gas in the vessel.
4. The level of the rinse liquid in the vessel.
5. The electrical resistivity and the pH of the recirculating rinse water.

*For large vessels, economics of inert gas quantity dictates a recirculating system.

The data should be displayed so that the operator can coordinate the response of the hydrogen evolution rate with the temperature of the recirculating gas system. A system of lighted panels to indicate process status (on normal, on emergency, off) and a system of distinctive alarms is recommended to indicate the approach of off-standard conditions.

The required ranges and accuracy are given in Table 2.

Table 2. Measurement Requirements

Instrument	Range	Accuracy
Hydrogen Detector	0.1 to 6 vol. %	±5% of full scale, each range
Hydrogen Monitors	1 ppm to 5 vol. %	±5% of full scale, each range
Oxygen Monitor	0.1 to 25 vol. %	±5% of full scale, each range
Water Vapor Meter	0.1 to 20 vol. %	±5% of full scale, each range
Moisture Detector	10 to 10,000 ppm	±5% of full scale, each range
Pressure Indicator	12.7 psia (87 kPa) to max ^a	±1/2 psi (3.5 kPa)
Temperature	32 to 300°F (0 to 149°C)	±3°F (±2°C)
Liquid Level Ind.	(Varies with facility)	±1 in. (25 mm)
Resistivity	5,000 to 10,000,000 ohm-cm ^b	±5% of full scale
pH Meter	5 to 14	±0.2 units

^aDesign pressure of vessel

^b50 to 100 000 Ω·m.

7.3.5.1 Hydrogen Detector. This instrument shall detect and actuate an alarm when accumulations of hydrogen approach the lower flammability limit (specified in 7.6.2) for a mixture of hydrogen in air. The detector shall be located in the upper portion of the building facility to avoid explosive concentrations.

7.3.5.2 Hydrogen Monitor. One such instrument is required to measure low-level concentrations of hydrogen in the reaction vessel, to control the reaction rate of the process, and to indicate the end point of the reaction.

7.3.5.3 Oxygen Monitor. This instrument is required to measure the oxygen concentration in the reaction vessel.

7.3.5.4 Water Vapor Meter. An instrument is required to measure and record the water vapor concentration of the recirculating inert gas during the sodium-water vapor reaction phase of the process. It is recommended that this unit have built-in redundancy of two probes since experience has shown that these units may need to be cleaned at least once per day.

7.3.5.5 Moisture Detector. An instrument is required to detect moisture in the vessel during drying to determine drying end point.

7.3.5.6 Pressure Monitors. Indicators are required to measure the process vessel pressure during processing, filling, and rinsing operations to assure maintaining a positive pressure.

7.3.5.7 Temperature Indicator. Instrumentation is required to record temperatures during the reaction phase, rinse operation, and drying.

7.3.5.8 Liquid Level Indicator. This device shall be capable of reading the water level in the vessel to the nearest inch (25 mm) during filling, cleaning, and draining operations to 6 in. (150 mm) below the component to indicate fluid level.

7.3.5.9 Water Resistivity Meter. An instrument is required to measure the electrical resistivity of the rinse water to determine the end point of the rinsing operation.

7.3.5.10 pH Meter. An instrument is required to measure the pH of the discharge rinse water to confirm the electrical resistivity measurements.

7.3.6 Water Supply. This system shall be capable of delivering Grade B water in accordance with RDT F 5-1 for rinsing operations and water vapor supply.

7.3.7. Auxiliary Equipment. All vessels and lines carrying high-temperature water vapor should be insulated and have external heating as required to prevent condensation of moisture and disruption of the water vapor concentration in the feed of the reaction vessel.

7.3.8 Storage Tanks. A system of tanks is required that can hold rinse solutions of the process until disposal can be accomplished. These tanks should be constructed of materials capable of containing various concentrations of caustic.

7.3.9. Pumps. System pumps are required to transfer fluids (water, caustic, and acids) into the reaction vessel, to recirculate water in the reaction vessel during the reaction, to recirculate water during rinse operations, and to transfer fluid wastes to storage tanks.

7.3.10 Steam Generator. A boiler is required to furnish water vapor for injection into the nitrogen supply line.

7.4 Materials.

1. Water, Grade B in accordance with RDT F 5-1.
2. Nitrogen, 99.995 percent or greater with a maximum dew point of -55°F (-48°C).
3. pH indicator paper, Fisher Alkacid or equivalent with pH range of 2 to 10.

7.5 Procedure.

7.5.1 Preprocessing Operations.

1. Review the component drawings, service history, and the planned draining procedure to define the requirements and constraints on the process by the component configuration. This should include thermal change rates, optimum locations for instrumentation application and rinse spray, flooding or special channeling fixtures, potential sodium frost or pools, gas pockets, and location and size of crevices and shrink fits.
2. Calculate the amount residual sodium to be processed (see Appendix C).
3. Estimate the processing time for vapor phase reaction, rinsing, and drying operations (see Appendix C).
4. Verify that the component will fit the processing vessel to determine if there are any unusual loading requirements.
5. Prepare the SRFOI to meet requirements of the CSRMP and this standard. This should include checklists and operational logs. Facility operations should be based on facility calibration tests as given in Appendix D.
6. Verify that the vessel valves and joints are tight in accordance with the SRFOI.
7. Verify that the water line is disconnected or closed with a positive lockout.
8. Purge or evacuate the vessel and fill it with dry nitrogen until the oxygen concentration is less than 2 vol. percent of the vessel discharge gas.

9. Verify the functional operation of all monitoring and alarm systems. Test the sensitivity of the hydrogen monitors as described in Appendix D.

7.5.2 Loading Operations.

1. Transfer and load the component into the reaction vessel under an inert atmosphere.
2. Load process control specimens if required by the CSRMP.
3. Purge the system with inert gas until the oxygen content is less than 2 vol. percent.

7.5.3 Reaction Operations.

1. Preheat the system and component to process temperature [160°F (71°C)] by circulation of hot nitrogen in accordance with the SRFOI.
2. Introduce water vapor generated from Grade B water into the feed gas at 1 ± 0.5 vol. percent water vapor content for a minimum of 2 h (7.2 ks).
3. Monitor the hydrogen concentration in the effluent gas. Shut down the water vapor feed if the hydrogen concentration exceeds 4 vol. percent. When the hydrogen level is reduced significantly below 4 vol. percent, continue the water vapor feed.
4. Increase the water vapor concentration in carefully controlled increments from 1 vol. percent to a maximum of 15 vol. percent in accordance with the requirements of the SRFOI (see Table 1).
5. Observe the hydrogen concentration in the effluent gas for any oscillations. After one oscillation [a deviation away from normal (as determined by calibrations, Appendix D), and a return to the previous level (up or down)] is observed, increase the feed gas temperature 10°F (5.5°C) immediately. If oscillation reoccurs, increase the recirculating gas temperature 10°F (5.5°C). If further oscillations of hydrogen concentration or temperature are observed, repeat the increases of the feed gas temperature in 10°F (5.5°C) increments, up to a maximum of 190°F (88°C)*.

*This "feedback control" technique has been repeatedly demonstrated to be effective in producing stable reactions. However, there is a substantial margin of safety if the specified temperature is exceeded. Corrosion results show that exposure to caustic at 200°F (93°C) for approximately 10 days can be tolerated without danger of caustic stress corrosion cracking (see Appendix B).

6. Continue reacting the sodium until the reaction is complete as indicated by a hydrogen evolution rate of 0.0003 cfm ($1.4 \times 10^{-7} \text{ m}^3/\text{s}$) or less. This evolution rate corresponds to a reacting surface of 5 in.² (0.003 m^2).
7. Increase the water vapor concentration by filling the vessel with hot Grade B water [180°F (82°C) maximum] to a depth not closer than 6 in. (150 mm) below the bottom of the component.
8. Sparge nitrogen through the hot water and monitor the hydrogen evolution rate. If the hydrogen evolution rate remains below 0.0003 cfm ($1.4 \times 10^{-7} \text{ m}^3/\text{s}$) for 4 h (14.4 ks), proceed with rinsing operations. If the hydrogen evolution rate increases, continue sparging with nitrogen until the reaction is completed, as indicated by the hydrogen level being maintained at or below 0.0003 cfm ($1.4 \times 10^{-7} \text{ m}^3/\text{s}$) for at least 4 h (14.4 ks). Proceed with rinse operation.

7.5.4 Rinsing Operation.

1. Terminate the water vapor addition, but continue the recirculation of the inert atmosphere.
2. Stabilize the temperature of the system and component at 180°F (82°C).
3. Adjust the nitrogen sparge feed to maintain a positive pressure in the system.
4. Start filling the reaction vessel with 180°F (82°C) Grade B water in accordance with RDT F 5-1. If a spray is used, start at the bottom and move upwards with special care to keep the liquid level below the component.
5. Monitor the temperature of the gas and the hydrogen concentration in the exit gas with special emphasis as the liquid level approaches inverted cavities. If the hydrogen concentration exceeds 1 percent, stop filling the vessel with water. Continue rinsing at that water level in accordance with Step 8 of 7.5.3.
6. After the component is completely immersed in 180°F (82°C) Grade B water and the hydrogen evolution rate reaches 0.0003 cfm ($1.4 \times 10^{-7} \text{ m}^3/\text{s}$) or less, pump out the initial rinse water and refill with 180°F (82°C) Grade B water and recirculate for 2 h (7.2 ks) or until the pH or water resistivity is unchanged.

7. Measure the water resistivity and repeat the rinse cycle until the resistivity is equivalent to the inlet resistivity [approx. 100,000 ohm-cm (1000 $\Omega \cdot m$)] or greater for storage, requalification, or immediate disassembly. Hold time shall be as specified in the SRF01.

7.5.5 Drying Operation. Purge hot 200 to 250°F (93 to 121°C) inert gas <100 cfm (<0.048 m³/s) until the discharge dew point is at least as low as -40°F (-40°C) for storage or for immediate disassembly or in accordance with the CSRMP for hands-on maintenance.

7.5.6 Unloading, Storage, and Inspection.

1. Cool the component and cleaning assembly per the SRF01.
2. Open the reaction vessel and remove the component in accordance with the SRF01.
3. Visually inspect the stored component for bleedout or other evidence of incomplete sodium removal in accordance with the schedule specified in the CSRMP. Storage in air is expected to generate bleedout faster than storage in dry, inert gas. If bleedout occurs, take action in accordance with the CSRMP.

7.6 Safety Requirements. The safety practices shall be consistent with and in accordance with OSHA requirements and additional local requirements.

7.6.1 Personnel Training. Personnel involved in sodium removal operations shall be trained in handling sodium (frost, liquid, and solid), caustic, hydrogen, and the hazard of inert or oxygen-deficient atmospheres. They should be thoroughly familiar with SRPS CSRMP, SRF01, checkoff lists, precautionary procedures, potential hazards of the operations, and emergency measures. Operating personnel shall be instructed in the use and performance of all process and safety apparatus. Personnel shall be trained on the location and correct use of all safety equipment including clothing, glasses, showers, eyewash, tools, fire alarms, and extinguishers. Personnel shall be instructed on rules for limiting access, no smoking, control of open flames, and the safety regulations for the cleaning area.

7.6.2 Hydrogen Hazards. Hydrogen evolved by the sodium-water reaction can form a flammable mixture in air between 4 and 74 vol. percent hydrogen as specified in U.S. Bureau of Mines Bulletin 503. The atmosphere in the reaction vessel shall be inerted with nitrogen to less than 4 vol. percent hydrogen. As a redundant precaution, the oxygen content of the reaction shall be maintained at less than 2 vol. percent oxygen. Hydrogen and oxygen concentration shall be monitored by hydrogen and oxygen meters.

Since inerted vessels have an oxygen-deficient atmosphere hazard, self-contained breathing equipment, positive ventilation, safety harnesses, and oxygen alarms shall be used if entry is required.

7.6.3 Caustic Hazards. Caustic (sodium hydroxide) as solid or in a solution can chemically burn tissue of personnel. Since caustic can occur as vapor, aerosol or liquid droplets on exposed surfaces, special protection shall be provided for eyes, face, and hands. Face shields, protective gloves, and protective clothing shall be used while working on components or the processing facility until a surface pH of 10 or less has been established by swabbing with wet indicator paper. Eye protection should be maintained until a pH of 8 or less is attained. Safety showers and eye wash apparatus shall be available near the work station.

8. ETHANOL SODIUM REMOVAL PROCESS SPECIFICATION

8.1 Process Description. Hot denatured ethanol (ROH) is reacted with residual sodium in a closed vessel, component housing, or system in accordance with Equation 2.

The reaction rate of ethanol with sodium is controlled by adjusting the temperature of the alcohol. Completion of the reaction is determined by the cessation of the hydrogen evolution and by a stable sodium ion concentration.

An alternate method for the removal of sodium uses acidified, denatured ethanol, and the composition of the ethanol is adjusted to provide the following composition:

1.5 vol. percent glacial acetic acid maximum
 $\leq$ 0.5 vol. percent water.

This acidic ethanol solution neutralizes the basicity and thus eliminates any caustic which may be formed. A lesser amount of acetic acid may be added to the ethanol, but the amount must be sufficient to neutralize the solution and still maintain an acidic solution at the end of the cleaning process.

Caution must be used in applying this alternate method because of the lower solubility of the reaction products in acidified ethanol. The problem of saturation in confined spaces should be carefully assessed because it could result in formation of insoluble precipitates in the component. (See Step 2 of 8.5.1 and Step 6 of 8.5.3.)

Rinse operations may use either recirculated fresh hot ethanol or hot water. Rinsing is repeated until the sodium concentration of the wash solution is unchanged.

The drying operation uses hot inert gas or evaporation until a specified dew point or alcohol vapor content is attained.

A typical schematic diagram of the process apparatus is shown in Fig. 2.

8.2 Process Conditions. The parameters and criteria for control of the process are summarized in Table 3.

8.3 Equipment Requirements. The process requires a closed system using inert gas.

Processing of radioactive components will impact the facility design. Requirements for containment, decontamination, corrosion by

Fig. 2. Typical Schematic for Ethanol Process.

Table 3. Ethanol Process Parameters and Control Criteria

	Fill	Steady State	Termination	Rinse	Dry
<u>System Feed</u>					
N ₂ , vol. %	nm	nm	nm	nm	100
O ₂ , vol. %	nm	nm	nm	nm	nm
H ₂ , vol. %	nm	nm	nm	nm	--
Ethanol, %	100	100	100	100	--
Water, %	<0.15 <0.5 ^a	<0.15 <0.5 ^a	<0.15 <0.5 ^a	100 --	-- --
Temperature, °F	Ambient ^b	140°	140	140° (ROH) 180° (H ₂ O)	200 to 250
, °C	Ambient ^b	60°	60	60° (ROH) 82° (H ₂ O)	93 to 121
<u>Discharge Fluid</u>					
Ethanol	nm	nm	nm	nm	--
Water	nm	nm	nm	nm	--
Na g/l	nm	≤30	≤30	remain constant	--
Na g/l (acetic acid option)	≤3.5	≤3.5	≤3.5	remain constant	--
<u>Discharge Vapor</u>					
Ar or N ₂ , vol. %	nm	nm	nm	--	100
O ₂ , vol. %	<2	<2	<2	<2	--
H ₂ , vol. %	<2	<2	<2	<2	--
Ethanol, %	<3 ^c	<3 ^c	<3 ^c	<3 ^c	0.01
H ₂ O(v) dew pt, °F	--	--	--	--	-40
, °C	--	--	--	--	-40
<u>Spurge Gas</u>					
Ar or N ₂ cfm/lb Na/h 6		nm	nm	nm	--
H ₂ O ppm (vol.)	<50	nm	nm	nm	--
O ₂ ppm (vol.)	≤50	nm	nm	nm	--

^aWhen using the alternate acetic ethanol process.

^bWhen ethanol fill temperatures above 85°F (29°C) are used, high hydrogen and alcohol vapor evolution rates (spikes) can be expected if substantial quantities of sodium frost are concentrated in localized areas of the component.

^cmore restrictive limits may apply in some locations.

nm = not monitored.

decontamination acids, and disposal should be defined early in the design cycle.

The system must conform to OSHA requirements and local safety codes for flammable hydrogen and ethanol fumes.

8.3.1 Process Vessel. It is desirable to construct the vessel with an alloy capable of resisting corrosive attack by solutions generated from the reaction of sodium with ethanol, rinse water, and if applicable, decontamination acids. The vessel shall be designed for ambient pressure plus the alcohol head, and temperature service of at least 150°F (66°C) and preferably to 250°F (121°C) (see Appendix E).

The bottom of the vessel shall be designed such that process residue will not affect drainage and recirculation.

A lockout system is required, if the water rinse option is used, to prevent accidental introduction of rinse water into the system before the ethanol stage of the reaction is complete.

8.3.2 Heat Exchanger. A unit is required which is capable of heating ethanol to 140°F (60°C), or water to 180°F (82°C).

8.3.3 Ventilation System. A system of blower fans or ducts is required to provide adequate air circulation to dissipate accidental accumulation of hydrogen which will collect at the high spots and the ethanol vapors which will collect at the low spots.

8.3.4 Inert Gas System. A system is required to supply inerting gas for the sodium removal system. The system shall include a storage tank, converter unit, pressure regulators, flowmeters, safety relief valves, control valves, and piping to provide a flow of dry purge gas to the system for dilution of hydrogen and ethanol to safe operating levels during inerting, filling, and rinsing operations.

An inert gas purge capacity is required to flush out excess hydrogen.

8.3.5 Instrumentation. Equipment is required to measure and record the following:

1. The inlet and outlet temperatures of the recirculating ethanol, rinse solutions, and drying atmospheres.
2. The concentration of hydrogen and oxygen in the vessel.
3. The level of alcohol and rinse water, if used, in the vessel.
4. The sodium ion concentration in the alcohol and rinse water, if used.

A system of lighted panels to indicate process status (on normal, on emergency, off) and a system of distinctive alarms is recommended to indicate the approach of emergency conditions.

The required ranges and accuracies are given in Table 4.

Table 4. Measurement Requirements

Instrument	Range	Accuracy
Hydrogen Detector	0.1 to 6 vol. %	±5% of full scale, each range
Ethanol Detector	0.1 to 10 vol. %	±5% of full scale, each range
Oxygen Monitor	0.1 to 25 vol. %	±5% of full scale, each range
Hydrogen Monitor	1 ppm to 5 vol. %	±5% of full scale, each range
Sodium Detector	10 ppm to 30 g/l	±5% of full scale, each range
Resistivity	5,000 to 10,000,000 ohm-cm ^a	±5% of full scale, each range
Liquid Level Ind.	As req. per tank size	± 0.5 in. (13 mm)
Pressure Monitor	12.7 psig (87 kPa) to max. ^b	±0.5 psi (±3.5kPa)
Temperature	32 to 300°F (0 to 149°C)	±3°F (±2°C)

^a50 to 100 000 Ω·m

^bDesign pressure of vessel.

8.3.5.1 Flammable Vapor Detector. This instrument(s) shall detect accumulations of hydrogen, ethanol vapors, or both which approach the lower flammability limit for mixtures of hydrogen and ethanol in air and to actuate an alarm. The detector(s) shall be located in appropriate portions of the building facility.

8.3.5.2 Oxygen Monitor. One instrument is required to measure the oxygen concentration in the reaction vessel exhaust.

8.3.5.3 Hydrogen Monitor. One such instrument is required to measure low-level concentrations of hydrogen in the reaction vessel gas outlet to indicate the reaction rate and end point of the reaction.

8.3.5.4 Sodium Detector. A means should be provided for promptly determining the sodium ion concentration in the recirculating ethanol and rinse solutions.

8.3.5.5 Water Resistivity Meter. An instrument is required to measure the electrical resistivity of the rinse water to determine the end point of the rinsing operation if water is used.

8.3.5.6 Liquid Level Indicator. A device shall be capable of reading the ethanol and rinse solution level in the vessel.

8.3.5.7 Pressure Monitors. Indicators are required to measure (but not record) the process vessel pressure during processing, filling, and rinsing operations.

8.3.5.8 Temperature Indicators. Instrumentation is required to monitor the temperature of the alcohol, rinse water, if used, and the drying atmosphere.

8.3.6 Water Supply*. This system shall be capable of delivering Grade B water in accordance with RDT F 5-1 for rinsing operations.

8.3.7 Storage Tanks. A system of tanks is required that can hold rinse solutions of the process until disposal can be accomplished. The ethanol process solutions are:

1. A mixture of ethanol with sodium alcoholates.
2. Caustic in rinse water*.

8.3.8 Pumps. System pumps are required to transfer fluid (water and ethanol) into the reaction vessel, to recirculating ethanol in the reaction vessel during the reaction operation, to recirculate water during rinse operations, and to transfer fluid wastes to storage tanks.

8.3.9 Vapor Condensor. This unit is required to condense ethanol swept from the processing vessel by hydrogen and sparging nitrogen in the exhaust line. The condensate shall be returned to the ethanol circulation system.

8.3.10 Safety Tools. Special non-sparking tools are required in the vicinity of the ethanol processing facility. Area access and personnel shall be monitored to prevent introduction of improper tools.

8.3.11 Fire Suppression System. A system is required to contain or divert the entire ethanol inventory in the event of a vessel or line rupture. A system is also required to put out a fire should it occur.

8.4 Materials.

1. Water, Grade B, in accordance with RDT F 5-1.
2. Inert gas, $<-67^{\circ}\text{F}$ ($<-55^{\circ}\text{C}$) dew point.

*If 180°F (82°C) water rinse is used.

3. Ethanol. Ethanol shall be procured as Type III ethyl alcohol in accordance with Interim Federal Specification 0-E-00760 which has been denatured in accordance with Formula 3-A of 26CFR212 using 200 proof denatured ethanol. Five gal (0.019 m³) of methyl alcohol shall be added to each 100 gal (0.38 m³) of ethanol in accordance with Formula 3-A of 26CFR212. Formula 3-A shall be further denatured by the addition of 10 gal (0.038 m³) of isopropyl alcohol anhydrous, and 1 gal (0.0038 m³) methyl isobutyl ketone to every 1000 gal (0.38 m³) of ethyl alcohol anhydrous to provide:

85.8 vol. percent ethyl alcohol anhydrous
4.3 vol. percent methyl alcohol
9.0 vol. percent isopropyl alcohol anhydrous
0.9 vol. percent methyl isobutyl ketone

4. Glacial acetic acid, reagent grade, 99.7 percent minimum purity with the following contaminants:

Chloride	0.4 ppm (maximum)
Sulfate	0.4 ppm (maximum)

5. Acidic ethanol, denatured alcohol as in item 3 above, with 1.5 percent acetic acid.

8.5 Procedure.

8.5.1 Preprocessing Operation.

1. Review the component drawings, service history, and planned draining procedure to define requirements and constraints on the process by the component configuration. This should include thermal change rates, optimum locations for instrumentation applications and rinse spray, flooding or special channeling fixtures, potential sodium frost or pools, gas pockets, and location and size of crevices and shrink fits.
2. Calculate the amount of residual sodium to be processed. (See Appendix E.)

The total sodium concentration in the ethanol at the process completion should not exceed 225 lb/1000 gal (30 g/l). If the optional acidified alcohol is used, the limit for the initial sodium ion content shall be low enough to permit the removal to be completed without exceeding an upper limit of 30 lb/1000 gal (3.5 g/l) of sodium.

3. Estimate the processing time for the ethanol reaction phase, rinsing, and drying operations (see Appendix E).

4. Verify that the component will fit the processing vessel and determine if there are any unusual loading requirements.
5. Prepare the SRFOI to meet requirements of the CSRMP and this standard. This should include checklists and operational logs.
6. Verify that the vessel valves and joints are tight in accordance with the SRFOI.
7. Verify that the water line is disconnected or closed with a positive lockout.
8. Purge or evacuate the vessel and fill it with dry inert gas until the oxygen concentration is less than 2 vol. percent of the vessel discharge atmosphere.
9. Verify the functional operation of the monitoring and alarm systems and fire protection equipment. Test the sensitivity of the hydrogen monitors as described in Appendix D.
10. Assure the absence of ignition sources. Verify the presence of limited access barriers, warning signs, and no smoking signs. Assure the availability of personnel protective equipment.

8.5.2 Loading Operation.

1. Transfer and load the component into the reaction vessel under an inert atmosphere.
2. Load process control specimens if required by the CSRMP.
3. Purge the system with inert gas until the oxygen content is less than 2 vol. percent.

8.5.3 Reaction Operation.

1. Start the sparge gas flow at approximately 6 cfm/lb of sodium [$6.24 \times 10^{-3} \text{ m}^3/(\text{s} \cdot \text{kg})$ of sodium] estimated to be reacted each hour.
2. Initiate monitoring of the hydrogen and ethanol content in the stack gas.
3. Fill the vessel with ambient temperature ethanol. Maximum hydrogen evolution usually occurs during the fill. Extra sparge of inert gas may be needed to ensure that the gas mixture being vented is below the flammability limit. However, during the initial fill, hydrogen spikes exceeding the flammability limit may occur.

4. Start ethanol recirculation.
5. While monitoring and controlling the hydrogen and ethanol content in the stack gas to meet facility safety requirements, increase the ethanol temperature to the cleaning temperature as specified in the SRF01. As shown in Appendix E, the preferred temperature for crevice cleaning is between 140°F (60°C) and the boiling point of the ethanol for the cleaning site location. However, ethanol temperatures exceeding the boiling point may be used for special cleaning purposes, as described in Appendix E. At temperatures above the boiling point, the reaction rate is greatly reduced.
6. Monitor the sodium ion concentration periodically. If necessary, add fresh ethanol to maintain the sodium ion concentration within acceptance limits (≤ 30 g/l).

If the optional acidified alcohol is used, the sodium ion content shall not exceed 3.5 g/l. [The solubility of sodium acetate in the solution is equivalent to 30 lb/1000 gal (3.5 g/l) of sodium.]

7. Reduce the sparge gas to the minimum needed to maintain the hydrogen gas concentration in the vent gas to less than 2 vol. percent and still maintain the vessel at a positive pressure.
8. Continue the process for the time estimated to clean the component. This condition is reached when the sodium ion content becomes stable and the hydrogen content of the vent gas is stable and less than 0.0003 cfm (1.4×10^{-7} m³/s) for a period of at least 2 h (7.2 ks).
9. Shut off the heat exchanger after the reaction is completed. Maintain a sparge to keep a positive pressure on the vessel.
10. Transfer the used ethanol from the vessel to the storage tank.

8.5.4 Rinsing (If Used).

1. Rinsing is optional if the sodium ion concentration in the ethanol cleaning solution is less than 1 g/l.*
2. Fill with rinse media.

*When the component is not rinsed, a harmless residue of sodium compounds may be visible, especially in areas where pools of liquid may form. This residue may be removed by brushing or rinsing with water. At concentrations greater than 1 g/l, the residue will be heavier and more difficult to remove although harmless to the component.

3. A maximum of 160°F (71°C) ethanol or 180°F (82°C) water shall be used.
4. Monitor the sodium ion content and continue rinsing until the sodium ion content is stable.
5. Upon completion of the rinse, remove the rinse fluid from the vessel while maintaining a slight positive pressure with inert gas.

8.5.5 Drying.

1. If ethanol is used, continue the purge with dry inert gas to sweep out the ethanol to a concentration of 0.01 vol. percent. Alternately, residual ethanol may be removed by evaporation under vacuum.
2. If a water rinse is used, see 7.5.5.

8.5.6 Unloading, Storage, and Inspection.

1. Cool the component and cleaning assembly per the SRFOI.
2. Open the reaction vessel and remove the component in accordance with the SRFOI.
3. Visually inspect the stored component for bleedout or other evidence of incomplete sodium removal in accordance with the schedule specified in the CSRMP. Storage in air is expected to generate bleedout faster than storage in dry inert gas. If bleedout occurs, take action as specified in the CSRMP.

8.6 Safety Requirements. Personnel involved in sodium removal shall be trained as specified in 7.6.1. Hydrogen evolved by the ethanol process shall be handled by the same safety criteria as specified in 7.6.2. The caustic solutions generated by the process shall be handled with the precautions specified in 7.6.3. If the optional acidified ethanol is used, caustic ethanol solutions are eliminated. The mildly acidified ethanol shall be treated as ethanol.

Liquid ethanol (denatured) contact with eyes, skin, or ingestion can cause blindness, irritation, or death. Protective equipment, such as face shields, rubber gloves, and safety showers shall be provided. Design of the facility needs to accommodate accidental discharge of liquid ethanol.

Ethanol vapor can form an explosive mixture with air and explosion-proof electrical switchgear, meters, and other electrical equipment are required. The vessel vapor discharge shall be inerted to below flammable limits to provide additional safety for reactor or other sensitive sites.

9. QUALITY ASSURANCE REQUIREMENTS

9.1 Quality Assurance Surveillance. Implementation of the operating instructions shall be monitored by a quality assurance representative in accordance with RDT F 2-2.

Quality assurance personnel shall review the CSRMP and the SRPS, and verify that the critical parameters specified in the SRFOI are maintained. In addition, quality assurance personnel shall verify that instruments used to monitor critical process parameters identified in Tables 2 and 4 are calibrated in accordance with RDT F 3-2.

9.2 Process Verification. Verify the following process steps as appropriate to the selected sodium removal process. Acceptable verification shall be indicated by a signature or initial and the date on the process specifications or check sheets.

9.2.1 Verify that engineering requirements documents, system operating procedures, and component documentation or status are consistent with control parameters, safe operation, and quality control acceptance.

9.2.2 Verify that the component to be cleaned has been drained to conform to the requirements of the CSRMP for the specific component.

9.2.3 Verify that the sodium removal system is ready, that tanks of appropriate cleaning fluids are adequate, and that acceptable packaging materials are available.

9.2.4 Verify that the handling equipment has been qualified for the prospective use.

9.2.5 Verify that dimensional interfaces of handling equipment and the component to be cleaned have been checked.

9.2.6 Verify that the operators are qualified.

9.2.7 Using the SRFOI, verify:

1. Proper positioning of the component in the sodium removal vessel.
2. Component temperature monitors have been correctly located and attached.
3. Radioactivity control methods (if appropriate) are being followed.
4. Process control specimens have been installed per the CSRMP.

5. The system (including tank joints and closures) is leak tight at the specified test pressure.
6. The hydrogen and oxygen monitors are functional.
7. The system pressure regulators and relief valves are correctly set.
8. The spray water line is interrupted or valved out and locked.
9. The process control system has been activated.
10. The operator is aware of the estimated quantity of sodium remaining and the predicted processing time schedule.

9.2.8 Periodically during the sodium removal process, verify that the requirements are being met.

9.2.9 Verify that rinsing and spray controls (e.g. valve lock-out) are correct and properly sequenced for safety.

9.2.10 Verify that the rinse water resistivity or alcohol sodium ion content matches the endpoint requirements.

9.2.11 Verify that the process control specimens are cleaned and inspected. (See Appendix A.)

9.2.12 Review the component historical records package to verify that the requirements for records have been met. The following records shall be maintained as minimum requirements:

1. Process SRPS.
2. Completed SRFOI with sign-off's.
3. Records, reports, or charts of system data such as, feed rates, temperature, and evolved hydrogen plots.
4. Records of sodium draining or redraining operation if performed during the sodium removal process.
5. Visual inspection and nondestructive examinations of the major component and of any test samples.
6. Records of tests and inspections to verify requalification for use or storage, or to justify disposal.

9.3 Acceptance Criteria. Acceptance criteria shall be as specified in the CSRMP.

APPENDIX A

MOIST GAS PROCESS (AS USED AT EBR-II)

This process is used to remove residual sodium from drained fuel subassemblies from the core or blanket of LMFBR's with residual heat of not more than 1.2 kW or temperatures not exceeding 340°F (171°C). The process removes sodium from exposed surfaces but not from crevices. Fuel subassemblies cleaned by this process are not qualified for reuse in a reactor without further disassembly and inspection.

A.1 PROCESS DESCRIPTION

Water vapor in gas is reacted with the residual sodium in a cask at a wash station in accordance with Equation 3.

The process is used for radioactive, contaminated, experimental, driver and suspected failed fuel subassemblies. The reaction is controlled by cooling with flowing gas to remove the evolved heat.

Moist air is used to process driver fuel and argon is used to process experimental fuel.

After a subassembly has been removed from the reactor core with its normal residue of sodium, pure argon is circulated to remove gamma-induced heat and to prevent melting of fuel or cladding. Sodium is removed in a cask (see Fig. 3) at a wash station.

Sodium is removed by introducing humidified air or argon to the recirculating argon coolant. The amounts of water and air are gradually increased as the recirculating cooling argon is removed by bleedoff. The reaction rate is monitored by thermocouples in the reaction chamber, and the temperature is maintained below 300°F (149°C) by adjusting the moist argon or air flow.

After 15 min (900 s) flow of humid gas or a drop of temperature, the moist flow is increased with the same restriction on maximum process temperature for an additional time.

At the end of this period the flow of humid air and recirculating argon are terminated and the subassembly is rinsed with 25 gal (0.095 m³) of pure water. The subassembly is dried by forced circulation of dry air, purged and recirculated with argon, and a fission gas sample is taken.

Experimental subassemblies are processed without air using moist argon. The subassemblies are then rinsed in water and dried in dry argon.

Fig. 3. Simplified Schematic of a Sodium Removal Processing Cask and Wash Station.

A.2 PROCESS CONDITIONS

The parameters and required criteria for control of the process are summarized in Table 5.

A.3 EQUIPMENT REQUIREMENTS

1. Cask for removing sodium from radioactive fuel subassemblies.

The cask shall be constructed gas-tight of stainless steel and lined with lead.

2. Blower for cooling subassemblies in the cask.
3. Scrubber, turbo-compressor venturi-type, to remove radioactivity from discharge effluent.
4. Waste tank to collect contaminated rinse solutions.
5. Retention tank to collect discharge solutions.
6. Water supply tank to store deionized water.
7. Water purity meter to determine resistivity of ion exchange water.
8. Ion exchanger, backup unit on high-purity water supply.
9. Rotometers, to measure gas flowrate.
10. Panelboard to centralize valves and control.
11. Flowmeters to measure fluid flow.
12. Gas sample line or cask to provide sample for radioactivity analysis.

A.4 MATERIALS

1. Water, deionized Grade A, in accordance with RDT F 5-1.
2. Argon, 99.96 percent with -76°F (-60°C) dew point.

A.5 PROCEDURE

A.5.1 Preprocessing Operations.

1. Verify that safety requirements of A.6 have been met, that radiological levels are acceptable to local requirements, and that setting of valves, blowers, and argon flow are correct.

Table 5. Moist Argon Process Parameters

Unit Operation		Initial		Final		Rinse	Dry
Variable	Prior to Processing	React Na with Air	React Na with moist Argon	React Na with Air	React Na with Argon		
Cask Temp °F (°C)	<340 (<171)	125 to 300 (52 to 149)	<300 (<149)	<300 (<149)	<300 (<149)	-	-
Cask Voltage, volts	>27	-	-	-	-	-	-
Water, resistivity megohm-cm	>10*	-	-	-	-	-	-
flow, gpm (m ³ /s)	-	-	-	-	-	5 (3.2x10 ⁻⁴)	-
Argon Flow, dry, cfm (m ³ /s)	-	-	20 to 30 (9.4 to 14x10 ⁻³)	-	20 to 30 (9.4 to 14x10 ⁻³)	-	-
wet, cfm (m ³ /s)	-	-	0.4 for 60 min (1.9x10 ⁻⁴)	-	7 (3.3x10 ⁻³)	-	-
Air Flow, dry, cfm (m ³ /s)	-	-	-	-	-	-	30 (1.4x10 ⁻²)
wet, cfm (m ³ /s)	-	0.4 (1.9x10 ⁻⁴)	0.4 (1.9x10 ⁻⁴)	7 (3.3x10 ⁻³)	7 (3.3x10 ⁻³)	-	-
Final Na Conversion	-	Greater of 10°F loss or 5 min	-	-	-	-	-
Reaction Time, min.	-	-	60	25	30	-	-
Oxygen, ppm	-	-	-	-	<200	-	-
Exhaust Line, psig After 1 min (kPa)	3 (21)	-	-	-	-	-	-

* < 10 000 Ω·m

2. Move the cask to wash station and review checklist.
3. Alert analytical laboratory for receiving fission gas samples. Take samples prior to connecting lines at wash station. Hold operations until results of gas analysis are available and acceptable to the responsible engineer. If sample radioactivity is 1.5 times that of reactor cover gas, use the procedure for suspected failed fuel subassembly.
4. Start the scrubber and assure that the waste tank and exhaust lines from the cask are empty.
5. Connect instrumentation recorder leads to cask for temperature indication. With the cask operating on internal d-c power, record the voltage. If the voltage is below the acceptable level, recharge the batteries in accordance with the SRF01.
6. Vent the water supply tank and the line to the cask. Close all valves to seal the system. Fill the water supply tank with water of the grade specified in the CSRMP and sample the water supply in accordance with the CSRMP. Close the vent and inlet valve on top of the water supply tank.
7. Vent the exhaust lines to the scrubber and after 1 min (60 s), reset the pressure to that required in the SRF01.
8. Purge the water supply line and feed hose with argon. Open the exhaust line of the wash station. Connect hoses and open appropriate valves as specified in the SRF01.

A.5.2 Reaction Operations.

1. Start argon flow for initial reaction in accordance with the SRF01.
2. Increase the moisturized argon flow to 7 cfm ($3.3 \times 10^{-3} \text{ m}^3/\text{s}$). If the temperature exceeds 300°F (149°C), reduce the moist argon flow.
3. After 30 min (1.8 ks) of moist argon flow, shut off the argon and draw an oxygen sample for analysis, if required for multiwall assemblies.

A.5.3 Rinse Operation.

1. Proceed to rinse with concurrence of the responsible engineer.
2. Secure the blower and adjust valves in accordance with the SRF01 to obtain 5 gpm ($3.2 \times 10^{-4} \text{ m}^3/\text{s}$) water flow.

3. Sample the cask effluent after 20 gal (0.094 m³) of water have passed through the cask. Flush the sample line prior to taking a sample.
4. Record the water resistivity. If the resistivity is greater than 1.25 megohms and the sodium ion concentration is ≤ 0.4 ppm, proceed to the drying operation in accordance with the SRF01. If the above conditions are not obtained, continue rinsing until the conditions are met. If resistivity level requirement is still not met after using 100 gal (0.4 m³) of rinse water, advise the responsible engineer.

A.5.4 Drying Operation.

1. When the rinsing is complete, stop the water rinse.
2. Dry with dry argon for 1 h (3.6 ks).

A.6 SAFETY REQUIREMENTS

1. Face shield, rubber gloves, and lab coats shall be worn when connecting or disconnecting either the sample blowdown line or the hoses to the cask.
2. The operator must remain with the cask at all times when washing a subassembly with water. The operator shall monitor the cask frequently during the initial humidification of an experimental subassembly and during the subassembly drying operations.
3. All samples shall be checked for contamination before they are sent to the analytical laboratory.
4. When moving or lifting casks, appropriate safety requirements should be imposed upon the crane equipment and tackle requirements. Inspection and other requirements of OSHA regarding safety of lifting and handling of heavy items shall be followed.
5. A radiation survey shall be made prior to moving the cask to ensure control of contamination and radiological hazards.
6. Air masks shall be available to personnel handling the cask..
7. Approval shall be obtained prior to release of radioactive krypton, xenon, cesium and iodine through the venturi scrubber system and stack. The operator shall remain with the cask during this entire operation.

APPENDIX B

CAUSTIC STRESS CORROSION CRACKING IN STAINLESS STEELS

Stress corrosion cracking of stainless steel can occur under certain combinations of caustic (sodium hydroxide and water) concentration, stress, temperature, and time. At 500°F (260°C), for example, cracking has occurred in less than 1 h in a 50 percent sodium hydroxide-water solution.

Much of the development of the water vapor nitrogen process (WVN) has been concerned with determining process conditions that will avoid corrosion damage to the components being cleaned. The results of this work are summarized in Fig. 4. This chart can be used as a guide in assessing the degree of component safety associated with specific processing conditions.

Fig. 4. Times in Days to When SCC was Observed in 50 wt% NaOH-Water Solution Sleeved 304 Stainless Steel Tensile Specimens Stressed to 100% of 0.2% Offset Yield.

APPENDIX C

PROCESS CALCULATIONS FOR THE WVN PROCESS

C.1 SODIUM QUANTITY ESTIMATES

The amount of sodium to be removed can be calculated. As an example, Fig. 5 shows a simple cylindrical shape containing typical types of surfaces encountered in sodium-wetted components. Referring to Fig. 5, the individual areas are calculated and totaled as shown in Table 6.

Dimensions in inches

Fig. 5. Cylinder Used to Estimate Process Duration.

Table 6. Surface Area Calculations

Surface	Calculation		Area		Sodium Depth*	
			in. ²	m ²	in.	mm
1	12 x π 13	=	490.1	0.3161	.002	.05
2	$\pi/4$ (12.1 ² - 12.0 ²)	=	1.9	0.0012	.002	.05
3	12.3 x π x 14	=	541.0	0.3490	.002	.05
4	$\pi/4$ (12.5 ² - 12.3 ²)	=	3.9	0.0025	.002	.05
5	12.5 x π x 27	=	1060.3	0.6840	.002	.05
6	$\pi/4$ (13.5 ² - 13 ²)	=	10.4	0.0067	.002	.05
7	13.5 x π x 1	=	42.4	0.0273	.002	.05
8	$\pi/4$ (13.5) ²	=	143.1	0.0923	.002	.05
9 (lower crevice)	$\pi/4$ (13 ² - 12.5 ²)	=	10.0	0.0064	0.25	6.35
10 (upper crevice)	$\pi/4$ (12.5 ² - 12.30 ²)	=	3.8	0.0025	1.00	25.4
11 (Pool)	$\pi/4$ x 12	=	113.1	0.0730	0.50	12.7
Total			2420.0	1.561		

*Sodium thickness is assumed to be 0.002 in. (0.05 mm) on well drained surfaces.

C.2 REACTION RATE ESTIMATE

For a reaction phase using 5 percent water vapor concentration at 160°F (71°C), Appendix C.2 gives 0.01 in./h (7.5×10^{-5} m/s) as the expected reaction rate which generates hydrogen at the rate of 0.000045 ft³/min/in² (3.3×10^{-5} m³/2·s) of reacting surface. The reaction rate for the various surfaces is shown in Table 7.

Table 7. Summary of Sodium Reaction Rate

Surface	Area		Depth		Reaction Time		H ₂ Evol. Rate	
	in. ²	m ²	in.	mm	h	s	ft ³ /min	m ³ /s
1 to 8	2293.1	1.479	0.002	0.05	0.2	720.	0.103	4.9×10^{-5}
9	10.0	0.0065	0.25	6.35	25	9×10^4	0.00045	2.1×10^{-7}
10	3.8	0.0025	1.00	25.4	100	3.6×10^5	0.00017	8.0×10^{-8}
11	113.1	0.0730	0.50	12.7	50	1.8×10^5	0.0051	2.4×10^{-6}
	2420.0	1.561					0.109	5.1×10^{-5}

With the information from Table 7, the theoretical hydrogen evolution rate is given in Table 8.

Table 8. Time Versus Sodium Reaction Rate

Time Span, h	Rate	
	ft ³ /min	m ³ /s
0 to 0.2	0.109	5.1×10^{-5}
0.2 to 25	0.00572	2.7×10^{-6}
25 to 50	0.00527	2.5×10^{-6}
50 to 100	0.00017	8.0×10^{-8}

The amount of undrained sodium on horizontal surfaces could be as much as 0.3 in. (8 mm) thick at a low drain temperature of 290°F (143°C). This amount of sodium can prolong processing operations.

If the reaction is started at 3 scfm (1.4×10^{-3} m³/s) of nitrogen and then decreased to 0.5 scfm (2.4×10^{-4} m³/s) at the 5-hour (18 ks) mark, the theoretical hydrogen content is given in Fig. 6. Because of diffusion, vessel size, etc., the actual evolution will probably follow the dotted line.

Fig. 6. Example of Hydrogen Evolution Rate.

The rinse cycle should be started after the hydrogen content falls to about 0.03 percent at 1 scfm [$4.7 \times 10^{-4} \text{ m}^3/\text{s}$] of nitrogen (equivalent to 0.0003 cfm of hydrogen). This would be after about 60 h (216 ks) of reaction time.

C.3 SODIUM RECESSION RATES FOR THE WVN PROCESS

The sodium recession rate for the WVN process using $160 \pm 20^\circ\text{F}$ ($71 \pm 9^\circ\text{C}$) feed gas and 1 to 10 volume percent water vapor is given by Equation 4.

$$K = 1.28 \times 10^{-8} (C_{\text{H}_2\text{O}})^{1.2} \quad (4)$$

where

K = sodium recession rate in $\text{m}^3/\text{m}^2 \cdot \text{s}$

$C_{\text{H}_2\text{O}}$ = volume percent of water vapor in feed gas.

APPENDIX D

SODIUM REMOVAL FACILITY CALIBRATION

New facilities shall be calibrated to provide data on systems response times and instrumentation characteristics. This information will assist operations personnel in interpreting and responding to process signals, particularly hydrogen oscillations. The following calibration tests should be performed, as a minimum.

D.1 Hydrogen Sensitivity Test. This test should be performed for both Sections 7 and 8 as follows:

1. The system shall be started up and brought to the conditions of temperature, pressure, flow rate, etc., where sodium removal processing would normally take place. (See Table 1 as appropriate.) The alcohol process facility calibration may be accomplished prior to alcohol addition by using only the inert gas purge system.
2. A quantity of hydrogen shall be injected as a pulse into the vessel to raise the free gas volume to a 1 percent concentration level.
3. Operating conditions shall be maintained until the observed hydrogen concentration has decreased to undetectable levels.
4. The hydrogen decrease as a function of time should be recorded.
5. Steps 2, 3, and 4, above, should be repeated for low, medium, and high inert gas purge rates.

D.2 Bulk Sodium Reaction Test and Hydrogen Oscillation Measurements. This test should be performed for Section 7 as follows:

1. Two containers holding bulk sodium are required and are described as follows:
 - . One 8-inch (200 mm) diameter by 4-inch (100 mm) deep containing sodium to a depth of 1 in. (25 mm) [1.75 lb (0.070 kg)].
 - . One 12-inch (300 mm) diameter by 4-inch (100 mm) deep containing sodium to a depth of 0.5 in. (13 mm) [1.96 lb (0.89 kg)].

Both containers are to be sodium-wetted before testing by heating to 1000°F (538°C) for 48 hours (173 ks) minimum in an inert atmosphere.

2. The containers, instrumented with thermocouples, shall be mounted horizontally in the process vessel at different elevations.
3. The containers are then processed through reaction, rinsing, and drying cycles.
4. Information is to be obtained on instrument response as noted in Table 2. Reaction progress is to be monitored as specified for the process.
5. The hydrogen evolution rate and reaction temperature should be plotted against time to indicate reaction rates and magnitude of the oscillations.

APPENDIX E

PROCESS CALCULATIONS FOR THE ETHANOL PROCESS

E.1 SODIUM QUANTITY ESTIMATES

The amount of sodium to be removed should be estimated by the methods outlined in Appendix C.

E.2 REACTION RATE ESTIMATES

Using the cylinder example as shown in Fig. 5, the reaction rates of alcohol and sodium illustrated in Fig. 7, and assuming the alcohol temperature to be 140°F (60°C), the following approximate reaction times may be anticipated in Table 9.

Table 9. Estimated Sodium Reaction Times

<u>Surface No. in Fig. 5.</u>	<u>Time</u>	
	<u>hours</u>	<u>seconds</u>
1 to 8 Vertical	0.001	3.6
9 Crevice	0.125	450.0
10 Crevice	0.500	1800.0
11 Bulk	0.250	900.0

The total estimated reaction time is approximately 30 min (1.8 ks).

The reaction rates for crevices and various-sized specimens [0.013 to 4 in. (0.33 to 100 mm) diameter] are given in Fig. 7 and 8. Fig. 9 shows the effect of attitude on the reaction rate. The effect of the addition of 1.5 percent acetic acid on the reaction rate is shown in Fig. 10.

If during the cleaning operation blocked passages are identified, it may be advantageous to react out the majority of the sodium and then heat the system under pressure to a temperature above the melting point of sodium [208°F (98°C)]. The hot, pressurized alcohol may be used to force the melted sodium from the passages during the reaction. Fig. 8 shows the reaction rates for the denatured ethanol, both below and above

Fig. 7. Reaction Rates of Sodium in Alcohol.

Fig. 8. Reaction Rates of Sodium with Denatured Ethyl Alcohol.

Fig. 9. Reaction Rate of Sodium in Alcohol with Various Crevice Orientations.

Fig. 10. Comparison of the Reaction Rates of Denatured Ethanol and Acidified Denatured Ethanol with Sodium Pellets.

the normal boiling point. As may be seen, the rate at the higher temperatures [up to 250°F (121°C)] is less than the reaction rate at the normal 140°F (60°C) temperature. The sodium alcohol reaction remains stable through the full temperature range investigated.

APPENDIX F

PROCESS CONTROL SPECIMEN

Since nondestructive examination methods have not been demonstrated to be adequate to detect caustic stress corrosion cracking, a traveler specimen is exposed to the sodium removal process along with the item. The specimen is then examined metallographically for evidence of caustic stress corrosion cracking. A typical process control traveler specimen is shown in Fig. 11.

SPECIMEN PROCESSING

1. ASSEMBLE REPRESENTATIVE ALLOY IN FIXTURE.
2. WET IN SODIUM AT 1050°F/2 WEEKS.
3. TENSION SPECIMEN TO 0.2% OFF SET YIELD STRESS OF ALLOY.
4. ATTACH TO COMPONENT IN INERT ATMOSPHERE.
5. PROCESS THROUGH Na REMOVAL PROCESS.
6. MEASURE DEPTH AT 100X OF ANY CRACKS ON EDGE OF ETCHED METALLOGRAPHIC LONGITUDINAL SECTION PER SKETCH A.

SKETCH A - LONGITUDINAL METALLOGRAPHIC SECTION

Fig. 11. Process Control Specimen.