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**COMPLETION OF EXPERIMENTAL BREEDER REACTOR-II SODIUM PROCESSING  
AT ARGONNE NATIONAL LABORATORY**

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**ABSTRACT**

The Experimental Breeder Reactor - II (EBR-II) at Argonne National Laboratory - West (ANL-W) was shutdown in September 1994 as mandated by the United States Department of Energy. Located in eastern Idaho, this sodium-cooled reactor had been in service since 1964, and was a test facility for fuels development, materials irradiation, system

and control theory tests, and hardware development. The EBR-II termination activities began in October 1994, with the reactor being maintained in an industrially and radiologically safe condition for decommissioning. With the shutdown of EBR-II, its sodium coolant became a waste necessitating its reaction to a disposal form.

A Sodium Process Facility (SPF), designed to convert sodium to 50 wt% sodium hydroxide, existed at the ANL-W site, but had never been operated. The SPF was upgraded to current standards and codes, and then modified in 1998 to convert the sodium to 70 wt% sodium hydroxide, a substance that solidifies at 65°C (150°F) and is acceptable for burial as low level radioactive waste in Idaho. In December 1998, the SPF began operations. Working with sodium and highly concentrated sodium hydroxide presented some unique operating and maintenance conditions. Several lessons were learned throughout the operating period. Processing of the 330 m<sup>3</sup> (87,000 gallons) of EBR-II primary sodium, 50 m<sup>3</sup> (13,000 gallons) of EBR-II secondary sodium, and 290 m<sup>3</sup> (77,000 gallons) of Fermi-1 primary sodium was successfully completed in March 2001, ahead of schedule and within budget.

Keywords: sodium processing, sodium hydroxide waste

## **SODIUM REACTION PROCESS**

The SPF Flow Diagram is shown in Fig. 1. The process begins with molten sodium at 120°C (250°F) being introduced into the sodium storage tank at the SPF. Sodium is available from two sources, although electrical interlocks only allow one source to be used at a time. The first source is the 210 liter (55 gallon) Fermi-1 primary sodium barrels. The barrels are moved to the barrel melt and drain room where they are placed into the melting stations. There are two manifolds each consisting of four barrel melting stations. Once the barrels are in place, they are opened, and the nitrogen purge lines, heaters, and insulation blankets are attached. The barrels are heated to 120EC (250EF) to melt the sodium. Dip tubes are inserted through the top of the barrel and the molten sodium is vacuum transferred to the 19 m<sup>3</sup> (5000 gallon) carbon steel sodium storage tank. The melting stations have a mechanism to tilt the barrels to maximize the removal of sodium.

The second source of sodium for the sodium storage tank is from the EBR-II by way of the sodium transfer line. A transfer line was built from the drain tank system in the EBR-II sodium boiler building to the SPF sodium storage tank as shown in Fig. 2. The transfer line is constructed of 4 cm (1½ inch) stainless steel pipe, approximately 250 m (820 feet) long. To allow surveillance during sodium transfer, the transfer line is located outside and above ground. It is also heat-traced and insulated to maintain sodium temperatures during transfer.

From the sodium storage tank, molten sodium is transferred to one of two carbon steel day tanks by pressurizing the sodium storage tank. Each of the day tanks holds 3 m<sup>3</sup> (730 gallons) of sodium, which allows for approximately one day's operation based on design throughput. By using the day tanks, the sodium storage tank is free to receive sodium from the barrel melt and drain system or the transfer line.

Prior to reacting the sodium, the nickel reaction vessel is charged with 50 wt% sodium hydroxide from the caustic cooling tank. A day tank is pressurized to flow sodium through a sodium injection nozzle, into the 50 wt% sodium hydroxide solution in the reaction vessel. Nitrogen is injected into the process through an annulus around the sodium injection nozzle to atomize the sodium and move the chemical reaction into the working fluid toward the center of the reaction vessel. As sodium is injected into the reaction vessel, the 50 wt% sodium hydroxide is concentrated to 70 wt% by the sodium reaction and the evaporation of water. The sodium hydroxide solution in the reaction vessel is maintained at saturation (boiling) temperature, which is directly related to the sodium hydroxide concentration. Through a separate nozzle, water is injected into the reaction vessel to maintain the temperature at approximately 185EC (367EF) and the sodium hydroxide solution at approximately 70 wt%. The sodium hydroxide being generated in the reaction vessel is continuously circulated to maintain a homogenous solution.

Sodium hydroxide of the appropriate concentration is pumped off into specially fabricated square 269 liter (71 gallon) drums that are lined with 90 mil thick high-density polyethylene. The square drums occupy the same volume as the standard 208 liter (55 gallon) cylindrical drums; however, the square drums optimize the space on a pallet and the landfill space required for disposal. After the drums are filled, they are grouped four to a pallet and transferred to an area where they are cooled down to 65°C (150°F). When the drum contents are solidified, they are shipped off-site for burial at the Radioactive Waste Management Complex at the Idaho National Engineering and Environmental Laboratory.

The sodium reaction process creates gaseous hydrogen and steam which are removed by the off-gas system. Off-gas from the reaction vessel flows through a mesh demister, is cooled in a condenser to remove the water vapor, flows through a scrubber to remove any entrained sodium hydroxide vapors, flows through a moisture eliminator, and is filtered through pre-filters and high efficiency particulate air (HEPA) filters to remove any particulate contamination. The remaining gaseous nitrogen and hydrogen are discharged through a facility stack.

Water from the off-gas system is collected in a suspect water holding tank and reused in the sodium reaction process. This reduces the quantity of contaminated waste water which would need to be treated for disposal.

During operation, all of the sodium and sodium hydroxide components are heated. After an operating run, the 70 wt% sodium hydroxide in the reaction vessel is diluted to 50 wt%. This prevents 70 wt% sodium hydroxide from solidifying in components. All sodium and hydrogen bearing systems and tanks are purged continuously with nitrogen gas.

Sodium inventories were processed from the lowest radiation level to the highest. The Fermi-1 primary and EBR-II secondary sodium contained only low levels of radiation, while the EBR-II primary sodium had radiation levels of

approximately 40 millirem per hour at 1 meter. Approximately 145 m<sup>3</sup> (38,000 gallons) of Fermi-1 primary sodium were processed initially to gain operator experience with the process. Next, all of the 50 m<sup>3</sup> (13,000 gallons) of EBR-II secondary sodium were processed.

Prior to processing the EBR-II primary sodium, the SPF was shutdown to undergo upgrades. Process system upgrades included increasing the operating temperature from 177°C (350EF) to 191°C (375EF), and installing physical and computer controls to prevent inadvertent water addition. A Quality Assurance/Quality Control Program for producing sodium hydroxide at the SPF was also implemented to verify the sodium hydroxide product is greater than 70 wt%. Samples were taken from every drum and were regularly analyzed. The sample results were tracked on a Shewhart control chart to indicate process shifts.

Approximately 106-125 m<sup>3</sup> (28,000-33,000 gallons) of Fermi-1 primary sodium were processed to validate modifications made to the SPF. Next, all of the 330 m<sup>3</sup> (87,000 gallons) of EBR-II primary sodium were processed. Finally, the remaining Fermi-1 primary sodium, approximately 20-39 m<sup>3</sup> (5000-10,000 gallons) were processed as a flush of the SPF systems. All the Fermi-1 primary and EBR-II primary and secondary sodium processing was completed in March 2001.

## **OPERATIONAL AND MAINTENANCE PROBLEMS**

Throughout the operation of the SPF, six major and continuing problems were encountered. These include the reaction vessel circulation pump failures and associated sodium hydroxide leaks; HEPA filter failures caused by off-gas moisture and sodium hydroxide carryover; electrical heater failures on sodium and sodium hydroxide systems; nozzle and line plugging; corrosion of carbon and stainless steel components in sodium hydroxide systems; and reaction vessel vibration.

### Reaction Vessel Circulation Pump Problems

Continuing problems occurred with the reaction vessel circulation pump. The original, magnetically-coupled gear pump was rebuilt by the manufacturer for the higher temperature operation. However, the polyether etherketone (PEEK) containment cup for the pump magnet ruptured and the PEEK drive shaft had severe wear causing the pump to fail on two separate occasions. The pump was replaced with a magnetically-coupled centrifugal pump having Hastelloy C wetted parts and a zirconium oxide containment cup. This pump also performed poorly over the operation of the facility. The containment cup failed from wear and gaskets failed causing numerous leaks. Solids from the sodium reaction process collected in the pump causing it to seize when shutdown. A second pump was procured and the two pumps were alternately repaired and replaced as they failed, requiring considerable continuing maintenance. The magnetically-coupled pumps proved unsuitable for high temperature, high

sodium hydroxide concentration, and solids-containing processes, but were used through the completion of the sodium processing.

### Off-gas System Problems

The single biggest challenge to sustained operation of the SPF was protection of the final off-gas HEPA filter from premature failure. Failure was caused by loading of the filter with entrained moisture in the off-gas stream and by sodium hydroxide attack. Several modifications were performed on the off-gas system in an attempt to extend filter life and thus the continuous operation time of the facility. The modifications that proved to be successful are as follows.

In order to prevent the entrained moisture from overloading the final filter, parallel pre-filters were installed. Different filter media was tested; however, the final choice was to use a polypropylene fiber layered in a 0.64 cm (1/4-inch) thick mat. These pre-filters were used as an impaction demister for very small droplets. The polypropylene mats, attached to frames and installed into the pre-filter housings, were not susceptible to sagging and tearing when they became saturated. The pre-filter frames used a gel seal to fit them to the housings. This gel was susceptible to attack by the sodium hydroxide solution. Eventually, sodium hydroxide carryover bypassed the pre-filters through the deteriorating seals. This bypassed moisture and sodium hydroxide loaded the final filters causing the pressure drop to increase. When the final HEPA filter pressure drop increased to 9 mm Hg (5 inches water gauge), the filters were replaced. Drains were also installed on the pre-filter housings to remove liquid collected off the polypropylene mats.

The original HEPA filter media was borosilicate glass, which is not sodium hydroxide resistant. Initial investigation into sodium hydroxide resistant filter media proved to be unsuccessful; however a Teflon® filter media was finally found. Parallel HEPA filter housings were installed with the Teflon® filter media in an attempt to extend the operating time between filter changes. With the pre-filter and final filter modifications, filter life was extended from a few days to approximately 1 to 1 1/2 months.

### Electrical Heater Problems

Electrical heaters were installed on sodium and sodium hydroxide tanks and lines, and off-gas lines and filter housings to keep them hot. Premature heater failure was a significant problem.

All strip heaters used on tanks were replaced because they were incorrectly installed and did not make sufficient contact with the tank. Insufficient contact meant poor heat transfer, high heater temperatures, and early heater failure. For better contact to the tanks' walls, studs were welded to the tanks to attach the replacement heaters.

Cable heaters on process piping often overheated and failed. This problem was corrected by installing more instrumentation and/or dividing up the heater into more zones.

### Nozzle and Piping Plugging Problems

Maintaining sodium and sodium hydroxide lines free flowing was another continuing problem. Keeping hot temperatures in the lines and equipment was the major determinant. Restarts following shutdowns caused most sodium system problems, especially when temperatures cooled to ambient. In addition, the 70 wt% sodium hydroxide concentration caused problems throughout the sodium hydroxide systems.

Even though pure sodium melts at 98°C (208°F), the reaction was difficult to start unless all the filters, valves, flow meters, and connecting purge lines on the sodium delivery system were above 149°C (300°F). Once sodium filled the lines after the initial flow, it was easier to raise the temperature of the sodium feed lines to a uniformly high temperature above 149°C (300°F). The heat capacity and conductivity of the sodium in the piping aided heat transfer. Nonetheless, impurities in the sodium, inadequately wrapped heaters, failed heaters, and inadequately insulated components necessitated heating some components to above 204°C (400°F). The sodium in the daytanks was heated to 177°C (350°F), and once it started flowing through the piping and components, distribution of the thermal energy throughout the system was assured. The system has a significant thermal capacitance and required a waiting period of routinely more than 12 hours to heat up.

The sodium injection nozzles also plugged with sodium hydroxide. Some maintenance necessitated that nitrogen purges to the nozzles/annuli be shut off while the reaction vessel still contained sodium hydroxide. Sodium hydroxide backed up into the nozzle and purge lines. If the plugs were extensive, the purge lines had to be taken apart and cleaned or replaced. If the sodium hydroxide was at the nozzle, heaters on the nitrogen lines and repeated steaming the nozzle helped. Persistence on the part of the operating crews by steaming, heating, and purging the nozzles was the key to getting sodium flow established.

As a result of increasing the operating temperature from 177°C (350°F) to 191°C (375°F), plugging of the drum fill line became a major problem. Prior to filling the square 269 liter (71 gallon) drums, the sodium hydroxide is cooled to 93°C (200°F) to prevent damage to the high-density polyethylene liner inside the drum, to reduce the time for the sodium hydroxide to cool and solidify, and to minimize operator exposure to high sodium hydroxide temperatures during drum filling operations. Over cooling of the drum fill line resulted in solid sodium hydroxide plugging the line. Unplugging the line required taking the piping and valves apart to melt and dissolve out the solid sodium hydroxide. Reducing the reaction vessel operating temperature from 191°C (375°F) to 186°C (365°F) and increasing the drum fill line cooling temperature set point above 93°C (200°F) alleviated the drum fill line plugging problem, but still ensured a solidified waste product.

During the EBR-II primary sodium processing campaign, problems with the circulation pump forced immediate shutdowns of the reaction system prior to diluting the 70 wt% sodium hydroxide to 50 wt%. As a result of the immediate shutdowns, 70 wt% sodium hydroxide solidified in the pump and circulation line. Damage to the pump occurred when attempting to liquify the sodium hydroxide to facilitate pump disassembly or to open the plugged suction line. Heaters used on the circulation line also failed when operators tried to get the system hot enough to melt the sodium hydroxide. This continued to be a problem until the end of operations. It was concluded that this pump was not appropriate for our application.

### Corrosion Problems

The sodium hydroxide systems were fabricated mainly of nickel. The off-gas system was constructed primarily of carbon steel and stainless steel. These materials were selected for use in a 50 wt% sodium hydroxide system which the SPF was originally designed for. When the process was changed to produce 70 wt% sodium hydroxide, material compatibility was evaluated. It was determined that although carbon steel and stainless steel were not the best materials for use in 70 wt% sodium hydroxide systems, they were acceptable for their limited life, and they should continue to be regularly inspected.

After approximately five months of operation, a leak occurred in the stainless steel demister housing which is installed on top of the reaction vessel. Visual inspection revealed several cracks on the demister housing, and the stainless steel type 304 demister mesh effectively disappeared. The 0.376 cm (0.148 inch) thick stainless steel demister housing was replaced with a 0.927 cm (0.365 inch) thick carbon steel housing containing a Monel 400 mesh. Over one year later, a leak was noticed on a welded fitting on the demister housing. Inspection revealed corrosion at the heat affected zone on the internal welds and significant general corrosion in the wetted flow areas of the housing. The demister housing was replaced with a 0.927 cm (0.365 inch) thick nickel housing. The Monel 400 mesh was reused.

Originally, the off-gas piping from the reaction vessel to the condenser was 10.2 cm (4 inch) by 0.602 cm (0.237 inch) carbon steel. Corrosion rates were determined by quarterly ultrasonic thickness measurements and ranged from 5 to 18 mils per year. After 20 months of operation, the carbon steel off-gas piping was replaced with nickel piping.

There was some concern about corrosion of the off-gas condenser since its shell and tubes are fabricated from carbon steel and stainless steel, respectively. Visual and liquid penetrant inspections of the inlet nozzle shell weld, random liquid penetrant inspections of the tubes, pneumatic test of the shell, and hydrostatic test of the tubes revealed no problems. When the off-gas piping was upgraded to nickel, ultrasonic thickness measurements of the shell showed adequate thickness. However, a visual inspection revealed corrosion

on the inlet piping which was replaced. Three months later, a leak was discovered on the off-gas condenser inlet piping flange which was replaced. Ultrasonic thickness measurements revealed adequate shell thickness. One month later, a leak was noticed on the inlet nozzle weld to the shell of the condenser. At that time, the shell was replaced with a new carbon steel one and the inlet nozzle was welded directly to the shell. A nickel shell was not used since the limited life of a new carbon steel shell was adequate to complete the processing of the remaining sodium.

Clearly, the combined conditions of increasing the sodium hydroxide concentration to 70 wt% and raising the operating temperature to 186°C (365°F), caused corrosion problems in the off-gas system. Stress corrosion cracking was found in the stainless steel components. In the carbon steel components, general corrosion was evident on wetted flow areas and localized corrosion was observed in the heat affected zones.

#### Reaction Vessel Vibration

The sodium-water reaction in the reaction vessel caused violent banging and vibration during the early startup tests. Increased atomizing nitrogen flow to the sodium injection nozzles greatly diminished the violence of the reaction and with it the amount of vibration. Even so, after approximately one week of operation, a fatigue crack developed in a machined fitting that was welded to one of the sodium injection lines, resulting in a small sodium leak. The sodium piping supports were originally designed for static conditions. The original design for monitoring reaction vessel level was continuous load cell measurement of the weight of the reaction vessel and its contents. Inadequate dynamic support of the sodium piping and reaction vessel allowed excessive relative movement of both, which resulted in overstress of the sodium injection piping and subsequent cracking. The load cells were abandoned as level indicators due to inaccuracies caused by temperature changes and rocking of the reaction vessel. The reaction vessel support legs were firmly bolted at the base to reduce vibration of the system components, and the sodium injection tubing was redesigned to provide improved flexibility. Additional structural supports were installed on the sodium and sodium hydroxide system piping.

After approximately two years of operation, a leak was discovered at the off-gas nozzle on the reaction vessel. Inspection revealed that the nickel off-gas nozzle weld had a fatigue crack caused by the vibration of the reaction vessel relative to the supported off-gas piping. Other nozzles on the reaction vessel were inspected using liquid penetrant examinations and showed no rejectable indications. Since only three weeks of operating time was needed to process the remaining sodium, only the reaction vessel off-gas nozzle was repaired.

#### Recommended Improvements

Prior to processing additional sodium through the SPF, some equipment and administrative modifications are

recommended. If a significant amount of sodium were to be processed, the equipment modifications include replacing the magnetic-drive circulation pump with a more durable pump; replacing the pre-filter gel seals with elastomer seals; replacing the existing heaters with more robust heaters and installing them with better contact to the tanks and piping; and replacing the existing carbon steel off-gas condenser with a nickel shell. In addition, the existing reaction vessel piping configuration should be analyzed to eliminate/minimize the fatigue situation with the reaction vessel nozzles. Administrative modifications include thoroughly heating the sodium systems to 177°C (350°F) and steaming the sodium injection annulus during processing and restarts to prevent nozzle plugging.

#### **CURRENT STATUS**

Currently, the SPF is in a layup condition awaiting future sodium to be processed. A minimum amount of liquid is left in the sodium tanks, and approximately 1 m<sup>3</sup> (270 gallons) of 50 wt% sodium hydroxide is stored in the caustic cooling tank to charge the reaction vessel for future restart. All the systems are in a shutdown condition, with heaters de-energized and breakers open, a nitrogen blanket on the tanks, and service water lines drained. Since the SPF is still considered an operational facility, all facility and equipment surveillance requirements per the applicable SPF or EBR-II Permit are still in effect. These surveillance requirements include daily inspections of the SPF Process Area and Transfer Line, the SPF Barrel Holding Room, and the SPF Container Storage Area. Weekly inspections are required on the Temporary Accumulation Areas Container Storage Area.

#### **CONCLUSION**

Processing of the 330 m<sup>3</sup> (87,000 gallons) of EBR-II primary sodium, 50 m<sup>3</sup> (13,000 gallons) of EBR-II secondary sodium, and 290 m<sup>3</sup> (77,000 gallons) of Fermi-1 primary sodium was successfully completed in March 2001. Although many problems were encountered throughout the SPF operating period, they were evaluated in a timely manner and corrective actions were completed safely without incident. The modifications resulting from the various problems have improved the process and facility so that future sodium processing facilities may benefit from these experiences. Completing sodium processing ahead of schedule and within budget was due to the teamwork and dedication of engineering, operations, and maintenance personnel.

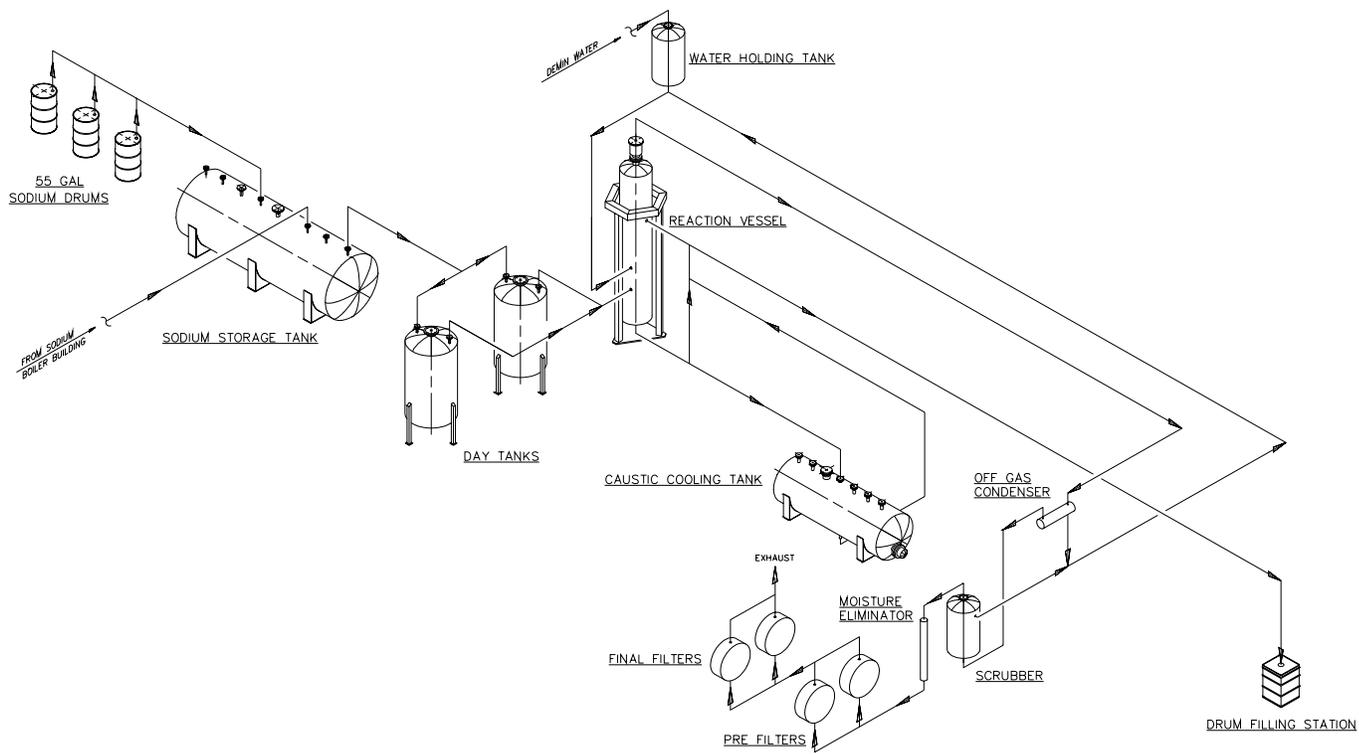


Figure 1. Sodium Process Facility Flow Diagram



Figure 2. Transfer Line