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## REMOVAL OF URANIUM AND SALT FROM THE MOLTEN SALT REACTOR EXPERIMENT

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## ABSTRACT

In 1994, migration of  $^{233}\text{U}$  was discovered to have occurred at the Molten Salt Reactor Experiment (MSRE) at Oak Ridge National Laboratory (ORNL). This paper describes the actions now underway to remove uranium from the off-gas piping and the charcoal bed, to remove and stabilize the salts, and to convert the uranium to a stable oxide for long-term storage.

## I. INTRODUCTION

The Molten Salt Reactor Experiment was a fluid-fueled test reactor that operated successfully at ORNL between 1965 and 1969 as part of a program to develop a molten salt thermal breeder reactor. The  $\text{UF}_4$  fuel was dissolved in a mixture of  $\text{LiF}$ ,  $\text{BeF}_2$ , and  $\text{ZrF}_4$  salts, forming a clear liquid at reactor operating temperatures of about  $650^\circ\text{C}$ . As shown in Figure 1, the fuel salt circulated through the graphite-moderated reactor vessel to a circulating pump, and then through a heat exchanger before returning to the reactor vessel. A secondary  $\text{LiF}$ - $\text{BeF}_2$  coolant salt was pumped from the heat exchanger to an air-cooled radiator. A third batch of  $\text{LiF}$ - $\text{BeF}_2$  salt was used to flush the reactor loop before and after opening the system for maintenance. Two fuel salt drain tanks and one flush salt drain tank are located in a cell adjacent to the reactor cell, and a coolant salt drain tank is located on the opposite side of the reactor, under the radiator.

MSRE initially operated with  $^{235}\text{U}$  as fuel. In 1968, the original charge of uranium was stripped from the fuel carrier salt and was replaced with  $^{233}\text{U}$ . When reactor operations were terminated in 1969, the fuel salt was divided between the two fuel salt drain tanks, and the flush and coolant salts were drained into their respective tanks. All three salt batches were allowed to solidify, and remain in the MSRE facility. Because radiolysis of solid

salt releases fluorine, an annual procedure to warm the salt for two weeks was implemented to reverse the effects of radiolysis by recombination of the fluorine into the salt matrix.

In 1994,  $\text{UF}_6$  was unexpectedly found in a sample withdrawn from the off-gas system serving the drain tanks. Laboratory experiments now show that heating the solid salt in the presence of fluorine also oxidized  $\text{UF}_4$  to volatile  $\text{UF}_6$ . A total of 37.6 kg of uranium, containing 84%  $^{233}\text{U}$ , was originally discharged into the drain tanks as part of the total inventory of about 8000 kg of fuel and flush salt. This uranium also contains 160 ppm  $^{232}\text{U}$ , at present decay. Concentrations of some of the other key radionuclides present in the salt are shown in Table 1. When  $\text{UF}_6$  was discovered, it was estimated that about 4 kg was present as volatile  $\text{UF}_6$  in the off-gas system, with an unknown quantity possibly present as solid  $\text{UF}_6$  deposits. Further work has now shown that about half of the total uranium inventory was released from the fuel salt. Once the migration was identified, its path through the off-gas system was traced, and a solid deposit of about 2.6 kg of uranium was found loaded onto charcoal at the inlet of an adsorber bed used during reactor operations to trap noble gas fission products for decay.

The MSRE Remediation Project was formed to stabilize conditions at the facility, to remove  $\text{UF}_6$  from off-gas piping and tank headspace, to remove the uranium deposit from the charcoal bed, to remove uranium and salt from the drain tanks, to convert uranium to stable oxide for storage, and to package the salt for further storage and ultimate disposition. These actions are being performed under the Comprehensive Environmental Liability, Compensation and Recovery Act (CERCLA), and are also being tracked as part of the response to the Defense Nuclear Facilities Safety Board (DNFSB) Recommendation 94-1.

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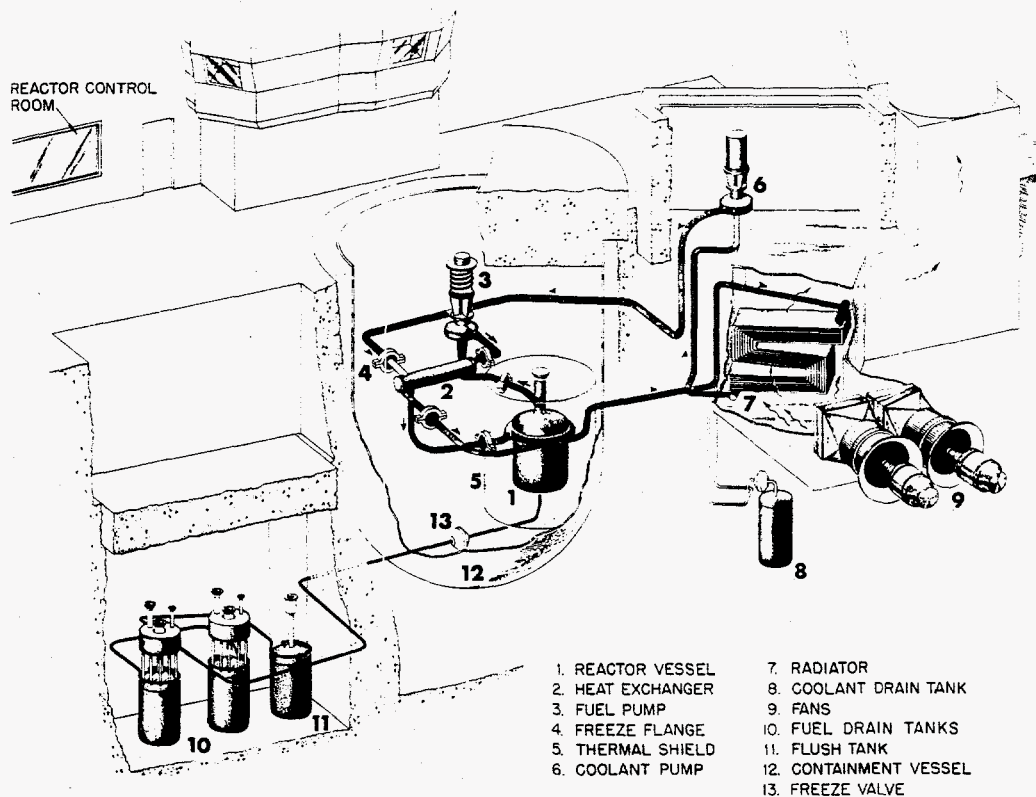


Figure 1. Arrangement of the principal components of MSRE.

Table 1. Activity concentrations of key radionuclides

Isotope	Concentration in fuel salt ( $\mu\text{Ci/g}$ )	Concentration in flush salt ( $\mu\text{Ci/g}$ )
$^{241}\text{Pu}$	44.7	0.90
Other Pu	12.2	0.25
$^{241}\text{Am}$	4.9	0.10
$^{90}\text{Sr}$	1410	26.5
$^{137}\text{Cs}$	1180	22.3
Salt mass	4650 kg	4265 kg

## II. WORK DESCRIPTION

### A. Interim Corrective Measures

When the migration of uranium from the salt stored in the drain tanks was discovered, kilogram quantities of fissile  $^{235}\text{U}$  had moved into piping and vessels not designed to maintain appropriate criticality safety margins. In particular, the uranium deposit in the charcoal

adsorber bed was located inside a 6 in. diameter pipe, which in turn was immersed in water. It was further recognized that, as the charcoal was exposed to fluorine at low temperatures, intermediate carbon-fluorine compounds were formed. These compounds could react exothermally to form  $\text{CF}_4$ .

Interim actions to improve safety margins included draining water from the charcoal bed cell and from other locations throughout the facility; isolating the charcoal bed from the drain tanks to prevent further accumulation of uranium or reaction with fluorine; and strengthening the top of the charcoal bed cell and filling it with  $\text{CO}_2$  so radionuclides could be retained even if the carbon-fluorine compounds did react. A criticality accident alarm system was installed in the facility, and the extent of uranium migration was established. Several pressure transducers were installed, showing that the off-gas system was at a positive pressure with respect to its surroundings. In addition, pressures varied at different locations (with no intervening valves), indicating some degree of plugging.

### B. Reactive Gas Removal

A trapping system was designed to safely remove  $\text{UF}_6$  and fluorine from the off-gas system. Sodium fluoride is

being used to chemisorb  $UF_6$  for interim storage and transfer to an oxide conversion facility, and alumina is being used to react fluorine in the gas stream. Process variables were tested in another facility before installation of the process in MSRE. It was found that a third trap, containing a zeolite molecular sieve, was needed to prevent corrosion by HF that formed as residual water was released from the alumina when reacting with fluorine.

The trapping system was installed on a portion of the off-gas piping located above the reactor cell. Once installed, it was found that non-volatile plugs had formed in the off-gas system, and only a small amount of uranium could be recovered. A bypass pipeline was constructed to allow the trapping system to be fed directly from the off-gas piping connected to the drain tank headspace, which contained the largest quantities of uranium. Another pipeline was connected to a portion of the off-gas system downstream of the original connection, allowing removal of gas from another significant volume ahead of the charcoal bed. With these pipelines, most of the off-gas piping was soon brought below atmospheric pressure, reducing the risk of a sudden release of radioactive material.

Operation of the bypass lines, however, demonstrated that plugs remained in the lines leading directly into the fuel salt drain tanks, and the drain tanks remained at a positive pressure. It was thought that the plugs consisted of  $UO_2F_2$ , radiolytically reduced  $UF_6$  (e.g.,  $UF_5$ ), or a combination of both. Equipment to inject  $ClF_3$  was installed so that these plugs might be converted back to volatile  $UF_6$ . Experience with  $ClF_3$  injections indicates that the plugs contain reduced  $UF_6$ , and the injections are increasing the porosity of the plugs. The gas flow rate from one of the fuel salt drain tanks soon increased, and in late December 1997, the porosity of the plug leading into the second fuel salt drain tank increased significantly. This brought both drain tanks to below atmospheric pressure.

Uranium is now being recovered from the drain tank headspaces at a much faster rate, and the drain tanks are at about 4 psia. As the evacuation proceeds (with continued  $ClF_3$  treatments), additional  $UF_6$  is being volatilized and the concentration of uranium in the gas stream has risen from about 10% by volume to over 60%. The original drain tank heaters were reactivated and a small amount of heat is now being applied to the drain tanks to aid in the sublimation of  $UF_6$  and to increase reaction rates of  $ClF_3$ . To date, 14 kg of  $UF_6$  have been recovered, and recovery of another 6 kg is anticipated.

As removal of uranium from the off-gas system is completed, additional heating and  $ClF_3$  treatments will be used to ensure that uranium is recovered from the entire

system and to reduce the possibility of releases as the tanks are entered in preparation for salt removal. A program of heating to just below melting in the presence of a fluorine cover gas is being considered to oxidize and trap additional uranium in the solid fuel salt prior to salt removal. Periodic operation of the trapping system is also planned to recover any additional radiolytic fluorine generated until the salts are removed and stabilized.

### C. Charcoal Bed Uranium Deposit Removal

When the  $UF_6$  and  $F_2$  from the drain tanks were trapped on the charcoal bed, the uranium was bound to the charcoal matrix and the fluorine reacted with the charcoal to form subfluorides of carbon,  $C_xF$  compounds. The form of the carbon-fluorine product is a function of temperature. At ambient temperatures, an equivalent to  $C_{2.6}F$  is formed. If heated, this can react exothermally to form  $CF_4$ . Although measures had been taken to reduce risk (including strengthening the cell and eliminating the most likely initiator by isolating the charcoal bed from fluorine in the rest of the off-gas system), the design of deposit removal equipment was being complicated by the need to guard against initiation of the reaction and to protect against energetic releases of radioactive material.

The laboratory testing program that was used to define the characteristics of the deposit was extended into a review of possible safe reactants. A proprietary process based on  $NH_3$  gas was developed to safely react the intermediate  $C_xF$  compounds to form stable reaction products. This process was successfully implemented in February and March of 1998. External temperature measurements confirmed the reaction of the  $C_xF$  compounds to stable material. With the carbon-fluorine compounds rendered inert, the  $CO_2$  charge has been removed from the charcoal bed cell, and preparations for physical removal of the uranium deposit are underway.

The charcoal bed containing the deposit (one of several in the charcoal bed cell) is constructed of 6 in. Schedule 40 stainless steel pipe configured in the shape of a "U" with each leg approximately 20 ft. long. Radiation and thermal profiling have been used to determine that the deposit of about 2.6 kg uranium is located in the upper 10 to 12 in. of the charcoal adsorber media. The uranium-laden charcoal media will be physically removed from the piping of the bed and will be packaged for interim storage and conversion of the uranium to a stable oxide. Major equipment items to be used are shown in Fig. 2.

A "cold tap" (terminology adopted when it was thought that the charcoal might be removed in its reactive state) will be installed on the outside of the charcoal bed just below the elevation of the uranium deposit. A tracked

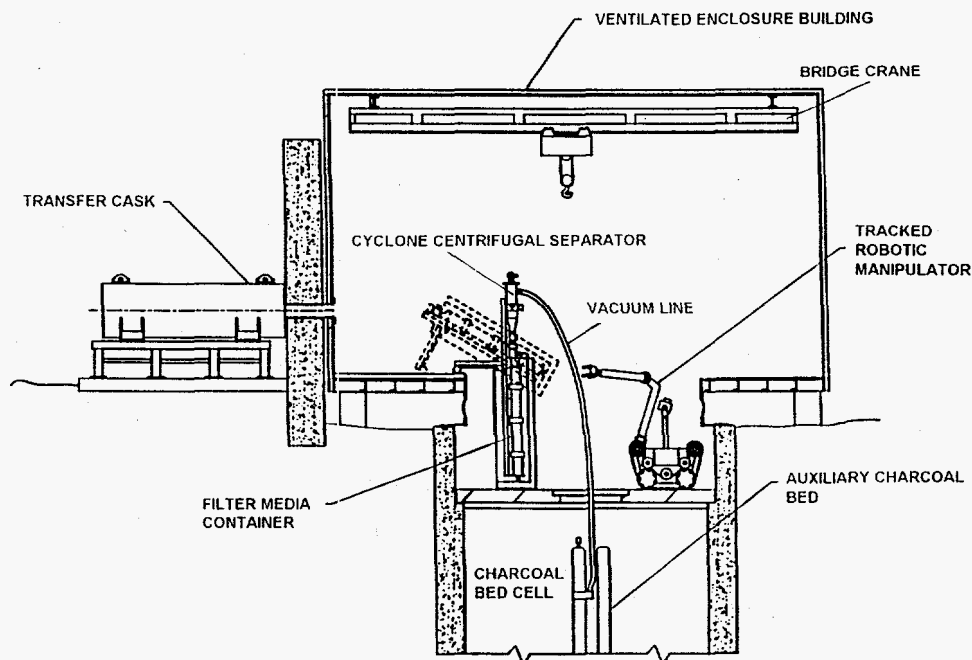


Figure 2. Arrangement of the charcoal bed uranium deposit removal equipment.

robotic manipulator will be used to install the tap on the highly radioactive charcoal bed. A vacuum system will then be used to pneumatically transfer the uranium-laden charcoal from the bed to a container located under a centrifugal separator. The affected charcoal will be removed in a single operation. Afterwards, the separator will be lifted away and the container will be sealed and rotated so that it can pass through a shield wall into a transfer cask. This cask will be used to move the container to an existing hot cell, where the charcoal will be repackaged into several smaller containers for interim storage and conversion to oxide.

The charcoal bed cell, located below grade just outside of the reactor building, will be covered with a ventilated and filtered enclosure to prevent the spread of radioactive material during the operation. A remotely-operated bridge crane will be installed inside the enclosure to assist the robotic manipulator during installation of the tap and operation of the removal equipment. The exhaust gas will be filtered, and its release will be controlled to minimize the dose associated with daughters of  $^{220}\text{Rn}$  (itself a daughter of  $^{232}\text{U}$ ).

A mockup of the charcoal bed cell has been constructed and the remotely-operated removal hardware is being tested. With completion of the ammonia treatment process, construction of the ventilated enclosure, shield

wall, and other facility enhancements is underway. The existing concrete plug over the cell will soon be removed, and the cell will be covered with a steel shield plate. This plate provides the openings that are needed to obtain access to the charcoal bed.

An initial access into the charcoal bed cell is planned for this summer to clear excess wiring and other debris from the above the charcoal beds, to conduct a more comprehensive radiation mapping of the deposit, and to physically probe the charcoal media to demonstrate that the vacuum system can remove the deposit. If necessary, other tools can be used to break up agglomerated material in the bed prior to vacuuming.

Installation of the remainder of the removal equipment is planned for the fall. Removal of the uranium deposit will be completed by February, 1999. After the deposit (and its associated radiation) has been removed, additional surveys of the other charcoal beds will be undertaken to determine whether further removal activities are appropriate.

#### D. Fuel Salt Disposition

Since reactor shutdown at the end of 1969, the salts have been kept in storage in their drain tanks awaiting definition of final disposition requirements. As with more

common reactor wastes, identification of a final ultimate disposition path remains elusive. Discovery of the uranium migration mechanism, along with the transuranic content of the salts, indicates that removal of the salts and processing into a more stable and transportable form is necessary.

A comprehensive review of potential disposition alternatives was performed as part of the preparation of a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) feasibility study and proposed plan. Radionuclide concentrations of a salt residue, with the fuel removed, match acceptance criteria for disposal of remote-handled transuranic waste at the Waste Isolation Pilot Plant (WIPP). However, obtaining permission to dispose of the salt residues in a facility designated for defense waste may prove difficult. The development of acceptance criteria for disposal at the Yucca Mountain site, undergoing characterization for the federal repository, is being monitored but a simple path leading to disposal in the repository has not yet been defined. Likewise, an existing solution for processing and disposal along with another, existing waste form has not been identified. Argonne National Laboratory has tested an electrorefining technology<sup>1</sup> that would produce a waste form similar to one being produced by processing EBR-II fuel. If this waste form is accepted by Yucca Mountain or another repository, further processing by electrorefining might be considered.

Thus, interim storage of a stabilized package is necessary at this time. A panel of the National Research Council was formed to review both disposition alternatives and the technologies needed to move forward, and concurs with the overall strategy.<sup>2</sup>

The plan calls for melting the salt, removing it from the drain tanks, separating the uranium from the salt (to prevent further generation of  $UF_6$  or a nuclear criticality), converting the uranium to oxide, and packaging the salt residue for interim storage at ORNL. By basing the package on WIPP acceptance criteria, the option of disposal at that facility is preserved, and the use of the transportation cask designed for remote-handled transuranic waste would allow transport to other sites as well. A CERCLA Record of Decision is being finalized to formally commit to this plan.

Investigative work has focused on understanding salt chemistry issues and on evaluating the remaining service life of existing equipment. Experiments have been performed on irradiated salt to gain an understanding of the  $UF_6$  formation mechanism and to establish the likely chemical state of the irradiated salt. Because  $F_2$  and  $UF_6$  have been released from the salt, the solid salt is left in a

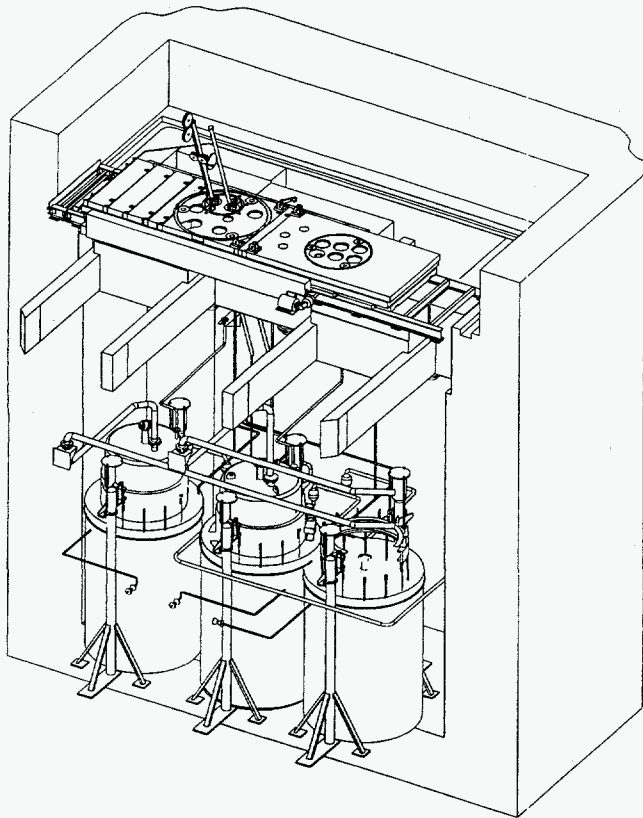
net reducing condition. Experiments have shown that simple melting of this salt would likely lead to further reduction of zirconium and uranium in the salt, causing diffusion of zirconium, and to a lesser extent uranium, into the Hastelloy tank walls. Incomplete melting of the radiolytically-reduced salt has also been observed. To ensure a complete melt, with all uranium in solution, a process of sparging a growing pool of molten salt with HF is being developed and tested.<sup>3</sup>

Before the salt can be melted, the drain tanks must be shown to remain fit for use with molten salt. This task requires unusual tooling, as the inspection must be done remotely, with the radioactive solid salt already present in the tanks. The basis for reuse of the tanks begins with the original tank inspections performed when the tanks were constructed. An evaluation of corrosion mechanisms has been performed, and reviews of process history and post-operation examinations indicate that corrosion should generally be modest.

Remote tooling is being developed to perform a visual inspection of the external (and later, internal) tank surfaces. Eddy current and ultrasonic examination tools will be used to verify remaining wall thickness. However, both tools are limited in their ability to identify localized effects, such as pitting. A remote-field electromagnetic technique has been modified to operate in a differential signal resolution mode. Scans of the tank wall can be taken remotely to identify both general corrosion and localized damage, with adequate spatial resolution. A mockup of the drain tank and its interfaces with the maintenance shield has been constructed and used to test tooling for implementation of these examination techniques, as well as for development of other tank and salt access tooling.

Initial surveys of the interior of the drain tank cell have been performed through an existing penetration in one of the shield blocks above the cell. The existing maintenance shield has now been installed over the drain tanks (Figure 3) to support examination and salt removal activities. Long-handled tools extend through ball-joint shield access ports, with cable drops from a slanted "fishing pole" to extend the reach of cameras, lights, and other tools. Access to the interior of one of the fuel salt drain tanks is planned for late 1998.

A conceptual design of the salt removal, processing, and packaging equipment has recently been completed. Salt will be melted while sparging with HF, and will then be transferred to a processing and packaging cell. The  $UF_4$  will be oxidized to  $UF_6$  and stripped from the salt using the same fluorination process as was used to strip the original  $^{235}U$  charge from the salt in 1968. The uranium



**Figure 3. Tooling for access to the fuel and flush salt drain tanks.**

will be sent to the oxide conversion facility, while the salt residue will be packaged with a getter to prevent the accumulation of further radiolytically-produced fluorine. Several trade studies addressing process, package, and equipment alternatives are now being conducted. Design and procurement packages are being defined, and procurement documents will be prepared in 1999. Removal of the flush salt is expected to begin late in the year 2000, and all three tanks are to be emptied by May 2002.

#### E. Uranium Conversion

Each of the three removal activities—removal and recovery of uranium from the off-gas system, removal of the uranium-laden charcoal from the charcoal bed, and removal of the stored fuel salt—produces a package suitable for on-site transportation and interim storage but not for long-term storage. Either  $UF_6$  or uranium-laden charcoal must be converted to a more stable oxide form. Furthermore, the high-energy gamma (2.6 MeV) radiation released from  $^{208}Tl$ , a daughter of  $^{232}U$ , requires that the conversion be performed in a hot cell.

Many industrial scale continuous and semi-continuous processes exist to convert uranium containing materials into an oxide. However, they are specific to a particular material and they are not suitable for processing relatively small batches of diverse materials. Also, they are not easily adaptable to hot cell operations and produce a significant volume of secondary liquid wastes.

A very simple proprietary process to convert all of the uranium laden materials is currently being tested. The process was selected according to the following criteria: (a) critically safe geometry; (b) minimization of uranium losses, secondary wastes, and contamination; (c) simplicity and adaptability to small-scale hot-cell operation; (d) no stirring, mixing, or open transfers between vessels; and (e) capability to treat all of the materials (i.e.,  $2NaF \cdot UF_6$  complex, uranium-laden charcoal, and possibly  $UF_6$  stripped directly from fuel salt and other miscellaneous materials). The process uses a uranium reclamation vessel connected in a closed-loop to the oxide conversion vessel. The only manipulations needed are the initial connection of the vessel containing the uranium and the disconnection at the end of the process to remove the uranium oxide from the second pot. The only waste generated after several batches is the spent HF-trapping cartridge (a solid waste). No tails or bottoms are produced.

Laboratory tests of the recovery and conversion at 1:10 scale were successfully completed. Initial testing of this process has yielded  $U_3O_8$  containing 290 ppm of residual fluoride. A full-scale prototype for the process has been assembled and testing is under way. Two full-scale tests of recovery and conversion of  $UF_6$  that was initially trapped on NaF have been successfully completed. The next test will be for uranium-laden charcoal.

Currently, one of the major uncertainties is the maximum acceptable fluoride content for the long-term storage of the converted oxide. There are two bases for a fluoride limitation: (1) pressure buildup due to radiolysis and (2) corrosion due to HF if  $H_2O$  is also present. Investigations to establish a fluoride limit for  $^{233}U_3O_8$  in the final package for long-term storage are being conducted. Experimental irradiations of (a) the converted oxide and (b) surrogate samples of known fluoride and water content are being used to simulate long term storage conditions. A variety of metal coupons (e.g., SS304, SS304L, SS316, SS316L, carbon steel, monel, aluminum) placed in contact with uranium oxide samples are being irradiated under very high radiation fields using a  $^{60}Co$  source.



### III. RESULTS AND CONCLUSIONS

The MSRE Remediation Project was created specifically to address risks to the public and workers that resulted from the migration of fissile and toxic materials. These risks were further exacerbated by the potential for a nuclear criticality, the potential for an energetic chemical reaction, and the potential for the discharge of airborne alpha radionuclides due to the positive pressure found in the system. The project objectives are to reduce risks as quickly as possible, and place all materials in a physical form and location appropriate for further interim storage and consistent with eventual ultimate disposition.

Today, many of the risks have been addressed. All significant quantities of fissile material are under appropriate criticality safety controls. No portion of the off-gas system is at a positive pressure with respect to its surroundings. The potential for an energetic chemical reaction in the charcoal bed has been eliminated. Almost half of the uranium inventory left after reactor operations has been removed, and nearly all significant sources of fissile and alpha-active material outside the drain tank cell, with the exception of the material in the charcoal bed and material left in non-volatile plugs in the piping, has been placed in a stable interim storage mode.

Progress is being made on the design and installation of equipment for removal of the uranium deposit in the charcoal bed, for conversion of recovered uranium into a stable oxide, and for removal and stabilization of the salts themselves. As these actions are completed, the MSRE facility will be prepared for normal decommissioning activities. With the arrival of Bechtel Jacobs Company, LLC, the new contractor for remediation activities on the Oak Ridge site, planning is being initiated for the transition from a Category 2 nuclear facility managed by a specialized workforce to a radiological facility being managed with typical surveillance and decommissioning actions.

All of the activities are being managed under the Federal Facilities Agreement for the Oak Ridge site, and with the present pace of work, all CERCLA and DNFSB milestones are being met.

The experience of bringing a facility idled for almost 30 years echoes similar experience at other sites across the DOE complex. Once the knowledge of the skilled workforce that originally operated the facility is lost, it is very expensive to bring new staff up to an appropriate skill level. Although in fact much of the equipment left at MSRE is in good operating condition, determining that 30 year old equipment can meet the stringent requirements for safely containing and processing hazardous nuclear

materials is expensive. The assistance of retirees that were familiar with past MSRE operations has proven invaluable to the present MSRE staff.

Although the MSRE Remediation Project is focused on its risk reduction and materials stabilization goals and is not a research project, several technological advances have been derived from the conduct of the project. Additional knowledge of the chemistry of fluoride salts has been gained, especially the areas of radiation damage to solid salts and reaction of solid salt with fluorine. The chemistry of carbon-fluorine systems at low temperatures and slow reaction rates has been explored, and knowledge is being gained on the radiochemistry of highly alpha-active solid  $UF_6$ . A novel flowsheet for the conversion of small batches of  $UF_6$  to  $U_3O_8$  in a highly radioactive, remote environment has been developed. Advances have been made in the technology of non-destructive examination of equipment, particularly the development of remote-field techniques that can be implemented remotely and yet efficiently.

Finally, this project has helped to draw attention to the potential benefits, as well as some of the difficulties, of fluoride molten salt technology for reactor and other processing systems. This technology may continue to serve in other applications, such as the use of a fluoride salt blanket in an accelerator-driven actinide transmutation system.

### ACKNOWLEDGMENTS

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