### Technical Approach for the Development of a Near Tank Cesium Removal Process - 9143

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### ABSTRACT

Parsons has been selected for development of two Advanced Remediation Technology (ART) projects. One of these projects is the Near Tank Cesium Removal (NTCR) project. The NTCR system uses the same basic ion exchange approach for Cs removal that has been used for decades in the nuclear industry. The essential difference in this approach is the development of a modular, mobile design concept based on a simplified process employing an advanced resin media and the use of cool nitric acid for elution and heated nitric acid for resin digestion. Under these conditions, the NTCR process shows significant improvements over the baseline ion exchange technology. These improvements will allow DOE to deploy a NTCR, free up tank space and accelerate closure of SSTs prior to Waste Treatment Plant Pretreatment Facility startup (WTP PTF).

Current estimates indicate that the Hanford tank farm system will run out of available storage space prior to startup of the WTP PTF currently scheduled for 2019. The lack of tank space will constrain the near-term goal of retrieving waste from single-shell tanks prior to full operation of the WTP. A deployment of an NTCR system will allow LAW processing to begin as soon as supplemental treatment (e.g. Bulk Vitrification) or the WTP LAW Vitrification Facility becomes available. The NTCR system is a self contained modular, transportable system that requires only limited process chemicals and separates the HLW into two process streams. Once the cesium is removed, the low activity waste stream can be vitrified. The high activity stream would be stored in the DST system until vitrified by the WTP High Level Waste (HLW) Facility. This technology can be sized to feed the WTP LAW melters at the nominal operating capacity (30 MT glass per day. Alternatively, it could be sized to feed a supplemental treatment system such the Bulk Vitrification process. The NTCR system is based on an eluteable ion exchange system using the Spherical Resorcinol Formaldehyde (SRF) Resin. The system employs a simplified flowsheet and equipment design that enables rapid deployment. The development of the NTCR system is consistent with accepted guidelines for engineering project development. Top-level project requirements, which were incorporated into the preliminary system design, are described. These requirements were developed in Phase I with appropriate design documentation, including system descriptions and process flow diagrams. Then a project risk review was performed based on the system design. Potential programmatic and technical risks to the project are identified and discussed. This risk review provides the basis for identifying the objectives and developing the testing scope to address them for the Phase II testing program. Finally, the proposed pilot-scale demonstration unit for Phase II is described.

## INTRODUCTION

The high-level waste (HLW) stored in the tanks at the Hanford Site consists of a mixture of sludge, salt cake, and supernatant liquids. The insoluble sludge fraction consists of metal oxides and hydroxides and contains many of the radionuclides. The salt cake, generated by extensive evaporation of aqueous solutions, consists primarily of dried sodium salts. The supernatant liquids consist of concentrated (5 to 15 M) aqueous solutions of sodium and potassium salts. The bulk of the water-soluble radionuclides, such as 137Cs and 99Tc, are contained in the salt cake and supernate.

The Hanford site is developing separations technologies to pretreat these wastes with the objective of reducing the overall cost of disposal while satisfying regulatory and environmental considerations. This is generally accomplished by separating the wastes into a low-activity waste (LAW) and a high-activity waste (HAW) fraction. The HAW fraction contains most of the radioactive and hazardous components that are incorporated into a relatively high-quality waste form such as borosilicate glass. Most of this waste will be disposed of at the planned HLW repository at Yucca Mt, NV. The production and disposal of the HAW form is relatively expensive, so there is an economic incentive to minimize the quantity of this waste form. The LAW fraction contains smaller amounts of radioactive and hazardous components and the majority of the bulk constituents of the waste. The waste forms for this fraction are typically less expensive to produce and are typically less robust than the HAW forms. Most of these wastes will be disposed of onsite in near-surface disposal facilities. The radionuclides in the aqueous fraction that are most often the target of separations processes include 137Cs, 99Tc, 90Sr, and transuranic components.

The primary mission of the U.S. Department of Energy's (DOE) River Protection Project (RPP) is to retrieve and treat the Hanford tank waste and close the tank farms. The farms are categorized into those containing tanks with single shells (SSTs) and those with double shells (DSTs). The SSTs are the oldest tanks, and some are known to have leaked radioactive material into the groundwater as a result of shell corrosion and breaching. No DSTs are known to have leaked. An interim objective of the DOE-RPP has been to pump supernate from the SSTs into DSTs to avoid further contamination of the groundwater. Therefore, most of the SSTs now contain only saltcake and sludge. The SST retrieval process involves dissolving the saltcake, mobilizing the sludge, and transferring this waste to one of the 28 DSTs. Available DST space is one of the key factors limiting the RPP mission goal of SST retrieval and closure. Once pumped to the DST tank farms, the tank waste can be provided to the Waste Treatment and immobilization Plant (WTP) for treatment.

The WTP consists of pretreatment, LAW vitrification, and HAW vitrification facilities. Waste from the DSTs is transferred via pipeline to the WTP pretreatment facility where it is separated into the LAW and HAW fractions. The HAW fraction is staged for immobilization at the HAW vitrification facility. Likewise, the LAW fraction is staged for immobilization at the LAW vitrification facility.

The focus of this paper is on the development of a near-tank cesium removal (NTCR) system that could be deployed in the near term to separate cesium from the aqueous phase of the retrieved tank wastes. The NTCR is a self-contained system that requires only limited cold chemical feeds and separates the HLW into two process streams. This separation is achieved using a regenerable ion exchange material, resorcinol formaldehyde (RF). Once the cesium is removed, the treated effluent is suitable for disposal after conversion to the disposal waste form. The HAW stream containing the cesium would be stored until vitrified as HLW. This technology is a compact system capable of treating a significant quantity of waste. The system would be sized to match the nominal operating capacity of the LAW treatment process. This paper will summarize the steps taken in developing the NTCR process. The structure of this project was developed consistent with accepted guidelines for engineering project development. Specifically, the requirements for the project were first identified in Phase I. These requirements were then incorporated into the preliminary system design by developing the appropriate design documentation, including system descriptions and process flow diagrams (PFDs). Based on the system design developed later, a project risk review was performed, and the potential programmatic and technical risks to the project were identified and discussed later. From this risk review, the objectives for the Phase II testing program were identified. The objectives and the testing scope to address them are also described. A description of the pilot-scale test rig proposed for Phase II is included.

# HANFORD WASTE TREATMENT REQUIREMENTS

The WTP facilities will be large, complex plants design to process any and all Hanford tank wastes. These facilities have significant budget and schedule requirements; startup of the WTP facilities is currently planned in 2019. The limited available DST space at this time will likely limit near-term progress on the retrieval of the wastes from the SSTs into the DSTs. Current estimates indicate that the tank farms will run out of available storage space before start-up of the Pretreatment Facility (PF). A recent study at Hanford explored the possibility of start-up of the LAW vitrification facility as early as 2013 [1], well ahead of the startup of the pretreatment and HLW vitrification facilities. This concept was referred to as "Start LAW First," and this terminology is adopted in this paper. The benefits of Start LAW First cited in this study include freeing up DST space, allowing SST retrieval to proceed, and demonstrating progress on tank treatment, independent of the schedule for pretreatment facility completion. The LAW vitrification pretreatment requirements include the separation of solids (filtration) and 137Cs. The study further identified cesium ion exchange as the preferred technology for cesium removal. Since the pretreatment facility will not be started until 2019, an alternative concept such as the NTCR is needed to remove cesium from the feed to the LAW vitrification facility.

The bulk vitrification system (BVS) concept is currently under development for demonstration. If this demonstration is successful, additional processing lines would be added for processing approximately 3 years later. The feed for this process requires a low solids and 137Cs content. The current approach for providing feed that meets the cesium concentration requirements is to target SSTs containing salt cake with a relatively low 137Cs concentration. Before dissolving the salt cake, an in-tank selective dissolution process will be employed to remove salt brine containing much of the 137Cs. A concept such as the NTCR also has the potential to provide decontaminated feed to the BVS with flexibility to treat LAW containing higher concentrations of 137Cs that may not be achievable with selective dissolution. The Hanford tank farms contain an inventory of 43-million curies of 137Cs. Management and separation of the cesium is dictated by specific limits placed on cesium as a result of its dose contribution and the maximum quantities of 137Cs allowable for immobilized LAW disposal in near-surface facilities at Hanford. In general, many of the SST wastes contain a smaller inventory of 137Cs and hence a lower concentration of cesium because these wastes have been subjected to previous cesium separation processes.

The Na:Cs mole ratio is a key parameter in defining ion exchange behavior. A higher ratio implies a lower cesium concentration since the feed to an ion exchange process is generally fixed in the range of 5 to 7 M Na. Lower cesium concentrations (higher Na:Cs ratio) are easier to process because the effective capacity of the resin is greater, and the required decontamination factors are less. The Best Basis Inventory (BBI) does not report total cesium, so the total cesium inventory was determined from the 137Cs quantities by assuming that the 137Cs is 20% of the total cesium in the SSTs (A, B, BX, BY, C, S, SX, T, TX, TY, U tank farms) and 25% of the total cesium in the DSTs (AN, AP, AW, AY, AZ, SY tank farms). For example, it was reported that 137Cs was 25% of the total cesium in samples from tanks AW-101 and AN-107 (both DSTs) [2]. It is assumed that there is a smaller fraction of 137Cs in the SST wastes because these wastes are generally older than the wastes in the DSTs. The Na:Cs molar ratio is typically higher in the SSTs because some of the cesium has been separated from these wastes in previous processes.

The Na:K mole ratio is also a key parameter for characterizing the performance of the cesium IX process. Potassium competes with cesium for the ion exchange sites and reduces the effective capacity of the resin. Lower Na:K mole ratios imply a higher potassium concentration at a constant sodium concentration in the feed to the process. The waste stored in the DSTs typically has higher inventories of potassium than that in the SSTs. The lower cesium and potassium inventories in the SSTs indicate that the cesium would be separated into a relatively smaller HAW volume than from the wastes in the DSTs.

The option of starting the LAW vitrification facility in advance of the pretreatment facilities and the HLW vitrification facility was evaluated [1]. The document presents treatment options for LAW to allow the LAW vitrification facility to process waste before completing construction and start-up of the pretreatment and the HLW facilities of the WTP. For the LAW vitrification facility to accept supernate, the waste must be pretreated to remove 137Cs to the designated limits for acceptance to the WTP LAW vitrification facility. The WTP LAW vitrification facility requires 137Cs in the LAW to be below 1.85E-04 Ci 137Cs/L

at 7 M Na. The average flow rate requirement for processing supernatant was estimated as ~20 liters per minute (~5.4 gpm) at 70% operating efficiency. With a sodium concentration of 5.5 to 6 M Na, this would provide 950 to 1040 metric tons of sodium per year. This is a sufficient quantity to supply two LAW melters capable of producing a total of 30 MTG/day with an average waste sodium oxide loading of ~18 wt% and an operating efficiency of 70 percent. The Start LAW First study focused on the pretreatment of wastes in the AP and AN tank farms. The AP tank farm was targeted since it is close to the waste transfer lines going to the WTP, and these tanks contain minimal sludge, so they can be relatively easily emptied to receive waste from the SSTs. As an alternative to processing wastes from the DSTs, waste could also be obtained from the other tank farms once sufficient space is made available in DSTs.

An alternative to immobilizing SST and DST waste in the WTP is to process the waste using the BVS. The BVS is designed to immobilize LAW as Class C low-level waste, as defined in 10 CFR 61.55, Waste Classification. Similar to the WTP, the proposed BVS process requires separation of cesium from the tank wastes to produce LAW before vitrification. The 137Cs content of the LAW feed to the BVS is 1.23E-3 Ci/mole of sodium. The nominal sodium concentration in the feed is 5 M, so the corresponding maximum 137Cs concentration is 6.2E-03 Ci/L of 5 M sodium solution. The average feed rate to the BVS is 1.13 gpm. A full-scale deployment with additional processing lines would have a higher feed-rate requirement. The required average 137Cs decontamination factors required for feed to the BVS range from 23 in some of the DST wastes to as low as 2 in some SST wastes. However, decontamination requirements for most of the SST LAW is notably between 5 and 23, which would indicate a preference for a selective separation process such as ion exchange to avoid generating large volumes of HAW.

The following section contains a general discussion of the operational requirements that an NTCR system will have to satisfy. It is intended to provide background information to support adopting the technology deployment. It is assumed that the NTCR would meet the current design requirements for the tank-farm contractor.

### **Interface Control Process**

To verify that the NTCR process equipment integrates into the tank farm in a seamless manner, interface control documents (ICDs) will be developed to define the agreed-upon actions to be taken by the deployment installation contractor and the site contractor. Interface control diagrams will also be prepared to define the hardware interfaces such as utilities and waste transfer lines. Programmatic ICDs will be developed for matters such as facility siting and permitting.

## **Nuclear Safety**

The NTCR process will be a new nuclear facility/activity at Hanford and will be required to meet applicable regulatory requirements. Nuclear safety requirements are captured in 10 CFR 830 Subpart B, 10 CFR 835, and the associated DOE Directives that constitute the "safe-harbor" compliance methodologies (e.g. DOE-STD-1027, DOE-420.1B, and DOE-STD-3009). Any new requirements from a recent DOE-EM Policy Memorandum on Integrating Safety into Design will be addressed for deployment. The high inhalation dose material (strontium and actinides), which is primarily in the solid phase, is filtered out upstream of the NTCR facility. The primary radionuclide in the NTCR system inventory is Cs, and the Hazard Category (HC)-2 threshold from DOE STD 1027 is relatively high (89,000 curies) for this radionuclide. However, due to the eluate's high Cs concentration and expected quantities, the NTCR facility is expected to be a HC-2 facility.

As a HC-2 DOE nuclear facility, DOE directives will require a thorough safety analysis. The principle hazards associated with the NTCR facility include direct radiation exposure, radiolytic hydrogen/flammable gas explosions, waste spills, and chemical hazards due to the high caustic in the waste feed and the nitric acid and caustic used in the process. A vessel-vent purge system, feed limitations on organics, and robust primary (i.e. vessels and piping) and secondary (i.e. dikes, facility structure, and filtered ventilation) confinement boundaries are probable choices for control systems. Sufficient shielding will also need to be incorporated to meet 10 CFR 835 radiation protection requirements. Additionally, recent DOE-EM policy for new HC-2 facilities (DOE-EM Memorandum, Interim Guidance on Safety Integration into Early Phases of Nuclear Facility Design, July 18, 2006) may dictate that more rigorous design criteria for seismic, confinement ventilation, and fire protection controls be implemented. If the

safety analyses (performed consistent with the DOE-EM Memorandum) determine that the unmitigated doses from the NTCR facility do not exceed 100 rem at 100 meters for some or all of the accident scenarios considered, then certain prescribed design criteria may be relaxed (i.e. PC-2 vs. PC-3 or no safety-related fire protection system) thereby making the facility more economical to deploy.

### Permitting

One of the more attractive attributes of the NTCR process is ease of integration and operation onsite under existing environmental and regulatory controls. No new site permits are expected since the NTCR system will be operated as part of the SST or DST system under the existing RCRA permit. A notice of construction (NOC) for radioactive air emissions may be necessary if the air emissions resulted in any significant offsite consequence or contributed a significant fraction of the site emissions. The NTCR process does not introduce any new safety hazards or require new regulatory permits or waste-designation changes. Compliance with existing site air and water permits is not expected to be problematic for the following reasons: 1) no new chemicals not already in use in the tank farms are introduced in the NTCR process, 2) the emissions associated with routine operation of the NTCR will be minimal (similar calculations performed by Parsons for other waste-processing activities indicate that expected emissions would result in a dose of << 0.1 mrem/yr), and 3) air emissions can be mitigated via standard industrial techniques (e.g., high-efficiency particulate air filtration). Some radioactive air emissions are anticipated. Calculations are required to show that the offsite dose consequences are insignificant (<0.1 mRem/year as is expected). If the offsite dose consequence is insignificant, a NOC should not be required per Washington Administrative Code 246-247. In addition, none of the thresholds in 40 CFR 61, Subpart H (National Emissions Standards for Hazardous Air Pollutants NESHAP-Rad) should be exceeded, and no special radioactive air emission monitoring or regulated emission controls should be required. Nonradioactive air emissions are anticipated. None of the gases from resin decomposition (CO2, N2, and NO2) that have been observed in tests to date are criteria pollutants. The emissions limit for a new source review of nitrogen oxides indicated in WAC 173-400-110 is expected to be well below the 2 tons/year limit. Additional applicable Washington standards are WAC 173-400 (new source review) and WAC 246-247 (air radionuclide design standards).

In accordance with a U.S. Nuclear Regulatory Commission letter1, removal of entrained solids and Cs137 from supernatant and saltcake wastes is required in order to meet the waste incidental to reprocessing determination for these wastes. However, after removal of entrained solids and Cs137 from the supernate and saltcake, wastes are considered to be LAW and will be immobilized in glass and disposed at the Hanford Site Integrated Disposal Facility (IDF). The concentrated Cs stream is considered HLW and will be stored until it can be vitrified in the WTP HLW facility for disposal at the national repository.

# **Environmental Protection Safety and Regulatory Requirements**

The NTCR will be designed to protect the public and environment in accordance with the requirements of WAC 173-303 (Dangerous Waste Regulations, Washington Administrative Code as amended). A permit application for the Research, Development and Demonstration (RDD) of the NTCR process for the demonstration unit will be required.

#### Waste Management

The process requires all liquid wastes to be routed either to the effluent treatment facility, in the LAW stream, or returned to the tank farms. The NTCR operating facility will comply with federal environmental regulations (40 CRF -260, -261, -264, and -265) and Washington WAC 173-303. The NTCR will be designed to disposition secondary radioactive liquid effluent waste streams generated at the NTCR in accordance with HNF-3172, Liquid Waste Processing Facilities Waste Acceptance Criteria, as necessary. The NTCR process will be designed for ease of decontamination during operation and for ease of facility deactivation at the end of system life in accordance with DOE Order 430.1A and 10 CFR 835.1002(d), Design and Control.

<sup>&</sup>lt;sup>1</sup>Letter, C. J. Paperiello, Office of Nuclear Material Safety and Safeguards, U.S. Nuclear Regulatory Commission, to J. Kinzer, U.S. Department of Energy, Richland Operations Office, Richland, Washington, Classification of Hanford Low-Activity Tank Waste Fraction, dated June 9, 1997

### **Occupational Radiological Protection**

The NTCR design will be compliant with the requirements of 10 CFR 835, "Occupational Radiation Protection," and will incorporate as low as reasonably achievable (ALARA) concepts for minimizing radiation exposure through physical design features (e.g., confinement, ventilation, and shielding).

### **Occupational Safety and Health**

The NTCR will incorporate design features that comply with applicable subparts of 29 CFR 1910, "Occupational Safety and Health Standards," for continuous occupancy as well as the new DOE Worker Safety Rule 10 CFR 851.

# **GENERAL OVERVIEW**

Figure 1 is a block flow diagram of the NTCR. The NTCR system stages filtered LAW at a nominal sodium concentration of 6 M received from the 200E or 200W tank farms. The LAW is processed through ion exchange columns to sufficiently separate cesium such that the 137Cs concentration makes it acceptable for immobilization in either the WTP or BVS. Treated LAW is staged for subsequent routing to either the WTP or BVS. LAW processing is terminated when the ion exchange resin is sufficiently loaded that the product 137Cs concentration becomes unacceptably high. The ion exchange columns are then flushed to displace LAW and eluted with nitric acid to remove cesium from the resin. The cesium-rich eluate is routed for DST storage. The ion exchange beds are regenerated with a caustic solution to prepare them to process further batches of LAW. These reagents are prepared and staged in the NTCR. The spent ion exchange resin is periodically digested with nitric acid, and the columns are replenished with fresh resin. The digestion process generates off-gas, which is scrubbed before being routed to the facility off-gas system. The baseline NTCR produces no routine secondary wastes because all solutions except the Cs-rich eluate are routed to the LAW vitrification facility or to a BVS.

### **Process Description and Technology Basis**

NTCR technology is based on the results of extensive testing over several years at the Pacific Northwest National Laboratory and Savannah River National Laboratory. Two organic ion exchange resins were considered for employment in the NTCR. SuperLig 644 (SL644) has been manufactured by IBC Technologies, Inc., American Fork, UT, in volumes up to 100 gallons. It is currently the baseline technology for the WTP but was criticized by an independent engineering and flowsheet review team (EFRT) for its chemical instability based on work by others [3]. The EFRT recommended its replacement, which confirmed the view of Bechtel National, Inc. (BNI), who had already embarked on a program to develop an alternative. Spherical resorcinol formaldehyde (SRF) was down-selected early in this program and subsequently tested for its chemical and hydraulic performance. The SRF has been manufactured by Microbeads (Skedsmokorset, Norway) and Boulder Scientific (Boulder, CO) in batches up to 100 gallons. The technology for manufacturing the SRF is owned by Microbeads and involves the production of spherical particles of RF on spherical polystyrene seeds. The granular form of RF was originally developed at SRS for cesium separation from alkaline tank waste. Granular RF (GRF) was also extensively tested for cesium separation from Hanford tank waste and is the reserve resin [4].

The SRF was tested in two columns with actual LAW from Tank AP-101 ([Na]/[K] = 7,  $[Na]/[Cs] = 1.15 \times 105$ ) for which a decontamination factor (DF) of 914 was required [5]. The 137Cs concentration in the actual AP-101 LAW effluents from the lead and lag columns exceeded the target after processing 25 and 90 bed volumes (BVs), respectively, at a flow rate of 2.89 BV/h. However, it was estimated that 115 BVs could have been processed while maintaining the overall required DF [5].

Most of the cesium was eluted into the first 6 BVs of eluant (0.5 M nitric acid). Additional tests on the SRF for separating cesium from actual AN-102 LAW ([Na]/[K] = 185,  $[Na]/[Cs] = 8.34 \times 104$ ) indicated a DF of 1060 was required [6]. This test used the same material as that used for processing the AP-101 LAW, and the nearly instantaneous breakthrough from the lead column was attributed to residual cesium on the resin from that test. However, the DF measured for the lag column effluent remained above target throughout the test, which processed 202 BVs of LAW. The overall DF measured on the composite effluent was  $1.11 \times 10E5$ . The superior performance of SRF processing AN-102 LAW compared to AP-101 is due to the lower concentration of potassium, which is the major competitor to cesium for the ion

exchange sites. The majority of the cesium was again eluted in the first 6 BVs of eluant. Others also analyzed the SRF from the actual AP-101 and AN-102 testing after it had processed 30 BVs of 0.5 M nitric acid [7]. This analysis showed that the transuranic (TRU) concentration on the resin of 2 nCi/g was much lower than the 100 nCi/g threshold for TRU waste classification. The residual 137Cs concentration of 3.2 Ci/m3 categorizes the resin between the Category 1 and 3 limits defined in the Hanford Site Solid Waste Acceptance Criteria requiring that the spent resin be loaded into high-integrity containers for disposal.

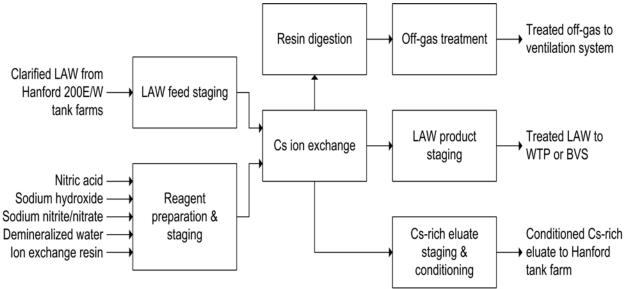


Figure 1: NTCR Block Flow Diagram

A parametric elution testing on GRF and SRF, processing simulated AN-105 LAW was performed [8]. The volume in which the peak cesium concentration in the eluate occurred decreased with increasing acid concentration. For example, the peak was attained after processing 5 BVs of 0.5 M nitric acid and after 3 BVs using 1.5 M nitric acid. A higher temperature (45oC compared to 25oC) was found better in minimizing the final residual cesium concentration on the resin. The WTP design basis for spent resin disposal (60 Ci 137Cs/g resin) was attained after processing 5 BVs of 0.5 M nitric acid for the SRF. Researchers performed hydraulic testing of the SRF during LAW processing and regeneration [10]. Bed voidage in LAW was 41% following fluidized regeneration and 38% after down-flow regeneration. Downflow regenerated SRF beds could not be fluidized with LAW because they behaved as plugs adhering to the column walls. The latter phenomenon occurred because the bed undergoes constrained expansion during down-flow regeneration. Therefore, fluidized regeneration was conducted to allow the resin to freely expand. Introduction of the LAW in an up-flow manner was desirable because otherwise it preferentially flows beneath the resident sodium hydroxide at high velocity on account of its higher density. The resulting high turbulence was observed to cause significant disturbance to the resin bed, leading to a non-level surface that could cause premature cesium breakthrough [8].

Ion exchange resins with similar structures to RF have been reported to react and dissolve in nitric acid. For example, it was reported that a sulfonated phenol formaldehyde resin (Amberlite IR-1) underwent "rapid, energetic reaction" with 2 M nitric acid [11]. Others considered phenol-formaldehyde resins to be the least resistant to oxidation and reaction with nitric acid [12]. With regard to RF, it was shown that the RF structure is activated toward oxidation from oxidizing reagents such as nitric acid [13]. Additionally, it was reported the presence of nitrogen in various derivatized RF resins after exposing them to 0.8 M nitric acid for 48 hours [14]. The nitrated product is expected to be subject to thermal decomposition catalyzed by acidic or basic reagents based on the work with evolution of nitrogen dioxide gas and oxidized carbon compounds [15]. More recently, nearly complete destruction of SRF in 3M nitric acid at 55<sup>o</sup>C while studying RF oxidation and energetics from a process safety perspective [16] was observed. Battelle demonstrated the principle of SRF digestion in tests conducted as part of this project by digesting the SRF in nitric acid, which destroyed 80% of the organic material as measured by total organic carbon (TOC)

analysis. One gram of dry SRF was dissolved in 200 mL of 3 M nitric acid at approximately 90oC within 3½ hours. The TOC concentration in the nitric acid reduced from a theoretical maximum of 3300 mg/L (derived if all the resin instantaneously dissolved upon contact with the nitric acid) to approximately 500 mg/L after 34 hours. The reduction in TOC is assumed to be caused by oxidation of the organic compounds to form carbon dioxide and volatile carbon compounds. Nitrogen dioxide gas was also observed, as expected, presumably evolving as the nitrated organic compounds thermally decomposed.

The foregoing work provides the technical foundation for employing SRF in the NTCR to separate cesium from LAW and to digest it in nitric acid. SRF has been shown to provide selectivity for cesium over other competing metals (notably potassium) while processing LAW containing the highest potassium concentration in the Hanford tank-farm complex. Sufficient data have been generated for the SRF that a preliminary design can be established.

### **Preliminary Process Design Concept**

Major process equipment in the NTCR system consists of tanks, packed columns, and pumps. Clarified LAW with a sodium concentration of nominally 6 M is received into a 70 m3 tank. This tank is sized to hold one 24-hour day's supply of LAW and the effluents from the LAW flush and regeneration operations of a single cycle. A positive displacement pump pumps the LAW through two ion exchange columns, each containing 790 L of spherical RF ion exchange resin. The ion exchange beds are nominally 1 m tall and 1 m diameter. The beds are sized to provide a residence time of 20 minutes at a nominal flow rate of 40 L/minute (10.4 gpm), consistent with the tests performed to date with this resin. The nominal flow rate of 40 L/minute allows the NTCR system to feed two LAW melters producing 30 MTG/day at 70% total operating efficiency (TOE). The NTCR system is assumed to have a TOE of 60% comprising an assumed 70% for general maintenance and another 78% to account for the time the system is offline undergoing elution and regeneration. Treated LAW is received into one of two tanks with capacities of 810 m3 each. The contents of a tank are sampled and analyzed to confirm that the composition is consistent with vitrification acceptance criteria. The size of the tanks is sufficient for the NTCR system to continue operating for 2 weeks while samples are analyzed, and the treated LAW is pumped to the vitrification facility using a centrifugal pump.

Eluant (0.5 M nitric acid) is pumped through the system to a tank, which is sized to hold 21 m3 of the effluent from eluting and regenerating both columns. A centrifugal pump is used to transfer the neutralized eluate to storage. Phase II testing will provide the data necessary to define the sizes of equipment required for resin digestion. For conceptual purposes, the digester acid tank has an assumed volume of 4000 L, or 5 BVs. A positive displacement pump recirculates the acid between a tank and the ion exchange columns while a centrifugal pump transfers the effluent for disposal. The off-gas scrubber is envisioned as a column packed with metal Raschig rings, and a positive displacement pump recirculates scrub acid between it and a tank. A reagent preparation sub-system provides the feeds for the LAW flush, elution, and regeneration operations. PFDs for the reagent preparation sub-system were not prepared, but it is nominally sized to provide sufficient reagents for one month of operation. PFDs for the facility off-gas system were also not prepared, but this sub-system is expected to be based on current proven technology. The NTCR preliminary design concept provides the foundation for the detailed design effort in Phase II. The design will be refined as Phase II testing is completed and the results are incorporated to provide a design with minimized risk.

#### **Preliminary Process Control and Monitoring Strategy**

LAW processing is ostensibly terminated when the product 137Cs concentration becomes unacceptably high. Typically, gamma monitors would be used to measure the concentration of 137Ba, which is the meta-stable daughter product of 137Cs. However, the 137Ba released from the resin after decay of separated 137Cs needs to be accounted for to avoid over-estimating the 137Cs concentration in the effluent. This could be accomplished by providing a sufficient lag between two monitors to allow the released 137Ba to decay sufficiently that a statistically accurate measure of the meta-stable 137Ba can be made based on a differential measurement. Such a technique has been described [17]. There are two points at which the 137Cs concentration could be measured. One advantage of measuring the concentration in the LAW exiting IXC-1 is that only that column need be eluted because there would be insignificant cesium on the resin in IXC-2. However, there probably would be insufficient space to allow two monitors to be

positioned with sufficient lag. In this case, a conservative control strategy would necessarily be adopted, and the resin would be under-utilized. Alternatively, the 137Cs concentration could be measured in the LAW exiting IXC-2. This option would utilize a greater portion of the resin capacity, although both columns would require elution. In addition, the time lag afforded by the guard column would allow for meta-stable 137Ba measurement by the difference between monitors on the exits from the lead and guard columns. However, the statistical accuracy of the technique would require consideration. Notwithstanding the meta-stable 137Ba monitoring issues, these two options are exemplified by considering the experimental results from testing actual AP-101 LAW previously reported [5]. If LAW processing were terminated when sufficient breakthrough had occurred on the lead column (25 BVs), then 13 BVs of neutralized eluate (7 BVs of acidic eluate and 6 BVs of caustic regeneration effluent) would be generated, leading to a cesium concentration factor of 2. Alternatively, if the process were operated using the concentration in the guard column effluent so that 90 BVs of LAW was processed, giving 13 BVs of neutralized eluate, then a concentration factor of nearly 7 is attained.

Another option would be to operate the NTCR system based on the 137Cs concentration in the effluent tank, i.e., the composite waste. This option would probably require that a bench-scale pre-test be performed to estimate the volume of LAW that could be processed. The NTCR system would then be operated based on the estimate and using gamma monitors in the column exit line to warn of premature breakthrough. A concentration factor of nearly 9 would be achieved based on previous results if this operational mode were adopted for processing AP-101 LAW [5]. The relative cesium concentration factors achieved in the different modes will be dependent on the breakthrough profile for the LAW being processed. For example, LAW with high relative potassium concentrations (e.g., AP-101 LAW) tend to have a broad breakthrough profile that leads to a significant improvement in the concentration factor when the operation is based on the lag, rather than the lead, column effluent concentration. However, the potassium concentration in AP-101 LAW is the highest in any tank waste. For example, the average sodium-to-potassium molar ratio throughout the tank-farm complex is approximately 85, compared to 7 in AP-101. Therefore, there may be little difference between the concentration factors achieved for the majority of wastes for the different operational modes. In this case, an operation based on the lead column would be desirable to minimize chemical degradation of the guard column resin. However, the monitoring technique limitations need to also be considered in optimizing the NTCR control strategy in Phase II.

#### **Facility Description**

The process module (i.e., containment enclosure) will be designed for mobility so that it can be moved between DST tank farms if required. An above-grade structure similar to the Waste Transfer Enclosure used for C Farm retrieval will be used for cesium removal. This structure provides both radiation shielding and containment with ventilation, radiation monitoring, and leak detection. This deployment will have a small footprint. The NTCR facility will include engineering features that minimize equipment maintenance and maximize reliability. For example, carousel-type operation of the ion exchange columns was discounted to minimize the number of valves. All of the components that contact radioactive waste will be contained within the structure, including the clarified LAW feed receipt, eluate, and LAW effluent tanks, LAW feed pump and the ion exchange columns. Also located in the structure is the resin digestion system digester and scrub acid tanks, scrubber pump, and off-gas scrubber. As much of the equipment as possible, for ease of maintenance, will be located outside of the vault, including the feed displacement, regeneration and eluant tanks, the eluant chiller, and the regeneration pump. All of the process equipment contained in the above-grade concrete or steel containment structures or modules will be designed for ease of decontamination and eventual decommissioning. Items that are expected to require maintenance or repair (pumps, heat exchangers, mixers, etc.) will be designed as small skid units to be easily installed or removed from the containment structure/module. Piping spools will use PUREX-style remote connections to allow removal, replacement, or re-routing as required. All of the tanks and piping will be made with 300 series stainless steel, suitable for the intended chemicals and content. The LAW feed, eluate, LAW effluent and digester tanks will be double walled with leak detection in the annulus. A cold-chemical equipment pad will be installed near the process module for makeup of the cold-chemical process solutions. Equipment to be located on this pad includes the feed displacement, eluant displacement, eluant displacement, resindestruction solutions, chemical-transfer pumps, and the eluant heat exchanger. Secondary containment will be provided for the process chemical equipment. The cold chemical feed tanks can be replenished by tanker trucks on a periodic basis. A control trailer will be installed (similar to that used for the C Farm

retrievals) near the process module to receive and process data, monitor operational parameters, and send control signals via a human-machine interface computer console(s).

# **Hazards Considerations**

The deployed NTCR system interfaces with several existing site service systems (e.g., power) and uses approved chemicals (e.g., NaOH) such that no safety hazards will be introduced resulting in a modification of environmental safety and health requirements by the M&O contractor. Table 2 identifies the primary unique hazards associated with the proposed NTCR system and provides the mitigation strategy as well as the examples of the mitigation used in previous site activities.

Hazard Characteristic	Specific NTCR Hazard	Mitigation	Prior Use of Mitigation Strategy
Radiological	Operations and maintenance staff exposed to radiation.	Remote maintenance equipment. Process equipment installed inside shielded containment enclosure.	Waste Transfer Enclosure designed and fabricated by Parsons for C-103/105 retrieval.
Chemical	Operations staff exposed to NOx gas during resin digestion.	Chemically separate the NOx from the digestion off-gas.	Standard industrial practice for removing NOx gas.
	Operations and maintenance staff exposed to corrosive reagents.	Remote maintenance equipment. Process equipment installed inside containment enclosures.	Waste transfer enclosure designed and fabricated by Parsons for C- 103/105 retrieval.
Explosion	Release of radioactive and chemical material following explosion of nitrated benzene-based resin.	Use a resin such that the nitrated product is not explosive.	WTP basis is King et al. (2006) which shows that nitrated resorcinol formaldehyde is not explosive.
Deflagration	Release of radioactive and chemical material following ignition of radiolytically produced hydrogen gas. Exposure of facility staff to corrosive and radioactive material.	Active mixing with an agitator system to verify hydrogen gas release. Ability to provide nitrogen pad to vessel overhead in the event of extended loss of power. Emergency elution	Used in DWPF and WTP.
Criticality	A small quantity of uranium and plutonium may separate on the resin. Facility staff exposed to criticality incident.	The quantities of uranium and plutonium separated onto the resin are bounded by those found in current process streams. Use a resin with low affinity for fissile elements. Filter feed to remove particulate uranium and plutonium	WTP limits the process volume and inventory.

 Table 2. NTCR Hazards Considerations

## Integration with Existing Facilities at Hanford

The NTCR can be sited at several possible locations within the 200 Area East or 200 Area West tank farms to supply either the WTP LAW vitrification facility or a bulk vitrification facility. Proposed locations in the 200 East and 200 West Areas are discussed in this section.

## 200 East Area Siting

A siting description of a cesium ion exchange facility is provided in detail in the Start LAW First document [1]. The specified site is near the AP Tank Farm and allows relatively easy access to the AP Tank Farm and the waste transfer lines to and from the WTP. A new transfer pipeline would be needed to provide filtered feed to the NTCR. Feed filtration would be provided by a Spintek Filter installed in tank risers in DST 241-AP-104. This design concept is similar to the "Spintek" filtration system designed and

demonstrated jointly by Oak Ridge National Laboratory and the SRS [18]. A short section of the existing LAW transfer pipeline (SN-701) currently connecting the tank farms to the WTP pretreatment facility will be removed. This line will be reconnected to the NTCR to return the neutralized cesium eluate to the DST system (e.g., AP or AZ Tank Farm). The other portion of the pipeline will be connected to the NTCR to route cesium decontaminated effluent to a new double-contained receiver tank or an existing DST before transfer to the WTP LAW facility. All other secondary liquid wastes will be combined with the decontaminated supernate for vitrification.

### 200 West Area Siting

The most attractive location for the NTCR in the west area is to co-locate it with the demonstration bulk vitrification facility near the S Tank Farm. This location would allow feed to be obtained from one of the DSTs in the SY Tank Farm or to obtain feed directly from one of the SSTs, such as the S Tank Farm tanks. Waste transfers to and from the NTCR can be made via an above-ground flexible waste-transfer hose in hose (similar to the Riverbend hose at the Hanford site). The cesium decontaminated effluent would be routed to the bulk vitrification feed tanks. The cesium-rich eluate would be returned to a different SY farm tank for cross-site transfer to the 200 East area. The Cs-enriched neutralized effluent will dictate the need for radiation shielding of the waste-transfer lines. A flexible waste-transfer hose could still be used to connect the NTCR waste-processing enclosure to an appropriate valve box or pump pit, provided that temporary above-ground concrete shield blocks are used. As an alternative, the flexible hose could be buried at least 4 feet if interference-free zones are available between the two connection points. The cesium ion exchange facility will require filtered supernate feed. The DBVF accepts supernate with solids removed by hydrocyclone, but not filtered. Because the hydrocyclone will not remove particle sizes as low as is achieved with filtration, additional provisions for supernate filtration in the 200 West Area will be required. The additional provisions for filtration may include processing with the Spintek filtration system, proposed for use in the 200 East Area, or a cross-flow filtration system. General interfaces are described in the following sections.

### **Eluate Storage**

The cesium rich eluate streams will be returned to the DSTs for storage until the HLW vitrification facility is available. The cesium-rich eluate stream composition will be adjusted with a portion of the LAW displacement or regeneration solutions so that the composition complies with all tank farm waste-chemistry control limits specified in RPP-10726 and in SD-WM-OCD-015.

#### Secondary Liquid Wastes

In the baseline approach, some of the feed displacement and regeneration solutions will be used to neutralize the eluant and the resin destruction solution. The neutralized resin destruction liquor and any remaining displacement and regeneration solutions will be blended with the cesium decontaminated solution to be routed to the vitrification facilities.

Alternatives for the disposal of the spent ion exchange solution remaining after resin destruction include sending the solution to the ETF or returning the solution to the tank farms (subject to limitations).

#### Solid Wastes

The baseline disposition pathway for the radioactively contaminated solid waste is to package it for disposal at the Hanford Central Waste Complex (CWC). This includes baseline disposition for air pollution control scrubber and filter components as well as personnel protective equipment. Analytical Sample Analysis: As indicated in the "Start LAW First" study, the WTP analytical laboratory would be used for sample analysis for a system located in the 200 East area. Analytical support for an NTCR system deployed in the 200 West area could be provided by the 222-S laboratory.

#### **Other Interfaces**

The other infrastructure required to support either unit is limited to electrical power (approximately 30 kW from a 480 V AC source) and flush water. The cold-chemical feed tanks can be replenished by tanker trucks on a periodic basis. The monitoring and control functions would be performed either in a portable control trailer or in any other convenient facility with the appropriate local area network tie-ins (e.g., 242-A Evaporator Control Room or the AY/AZ Control Room).

### **Decontamination and Decommissioning**

Decommissioning is defined as the safe removal of the nuclear facility from service and the reduction of the residual radioactivity to a level that permits release of the property for restricted or unrestricted use. Decommissioning of the NTCR process will occur at the conclusion of the project. The baseline disposition path for the decommissioned facility piping, equipment, concrete, and steel is the CWC.

## **RISK CATEGORIES**

In general, the level of risk associated with an activity is determined by multiplying the probability of occurrence by the consequence of an event. The probability of occurrence is stated as the likelihood that an event or a condition may adversely affect the project. The consequence of an identified issue is stated in terms of the level of impact on the design and deployment of the process. The risk matrix was developed and provides a visual reference to assess risk and prompt corrective/preventative actions when risk becomes elevated. In this case, the level of risk is classified as either low, medium, or high. The x-axis (probability) exhibits three distinct ranges of probability that the identified issue will cause an undesirable event. Similarly, the y-axis exhibits three distinct levels of consequence that may be observed should the event occur. Project staff used the risk matrix by first identifying an issue of concern. Second, a probability range is selected after assessing the likelihood of an event. Third, the most likely and worst consequence, prompting the project to take action. Varying degrees of action are necessary based on the assessed risk level. For example, when low-risk issues are identified, the project may require few mitigating actions to address the issues posing a risk. In contrast, when high-risk issues are identified, significant mitigating actions must be taken, or an undesirable event will occur.

# NTCR PILOT PLANT (NTCR-PP) DESCRIPTION AND FUNCTIONALITY

The NTCR-PP provides a platform to aid in identifying and demonstrating optimum NTCR deployed plant (NTCR-DP) process operations and equipment design. As such, the NTCR-PP provides a platform in which a single complete ion exchange cycle and one resin digestion operation can be performed in one test. The first operation in an ion exchange cycle consists of processing simulated LAW. Subsequent operations include LAW displacement using a dilute caustic solution, resin elution to remove the cesium into a dilute nitric acid solution, and regeneration to prepare the resin to process another batch of simulated LAW. The NTCR-PP also provides a platform to perform resin digestion tests. Warm nitric acid is re-circulated through the ion exchange column to dissolve the resin. The nitric acid solution is then held at temperature to digest the dissolved resin to produce an off-gas consisting of carbon dioxide and nitrogen dioxide and water. The gaseous effluent from this operation is scrubbed with dilute nitric acid to remove nitrogen dioxide gas before discharge through the facility off-gas system.

## **General Requirements**

The NTCR-PP is designed for operational flexibility to provide the capability to test a variety of operational scenarios. It is not limited to only testing and demonstrating the baseline NTCR-DP operations concept described in the Parsons Team proposal and paper. However, the NTCR-PP equipment is specified for operation with organic ion exchange resins and caustic and acidic aqueous process solutions. The NTCR-PP provides for the completion of a single complete ion exchange cycle in equipment scaled to reproduce the expected full-scale operational characteristics while minimizing operating costs. For example, the ion exchange columns in the NTCR-PP subsystem are full-height to provide the same pressure drop expected in the full-scale system, but only 33-cm in diameter (compared to 1 m in full-scale) to minimize the costs associated with procuring the simulated LAW and reagents and subsequent waste disposal. Simulated LAW processing is prototypic in that the material is processed through two ion exchange columns in series.

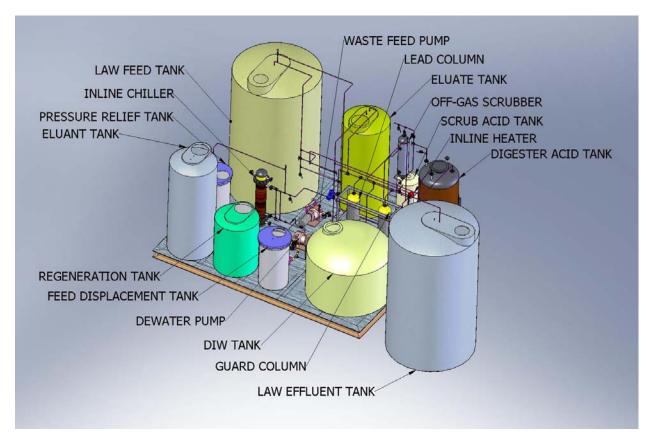
The lead and guard columns can be eluted and regenerated in series together. Only the lead column can be independently eluted and regenerated, as baselined for the NTCR-DP. Elution and regeneration can be performed either in an up-flow or down-flow manner for testing flexibility. Switching between up-flow and down-flow can provide for more efficient displacement of reagents from the system. For example, performing up-flow elution makes sure that cesium-rich (and most radioactive in the NTCR-DP) fluid only

contaminates the equipment and lines used to down-flow simulated LAW feed. Sufficient pumping capacity is available to provide for slight bed fluidization in up-flow (5% bed expansion). Fluidized regeneration may prove advantageous in minimizing bed compression and wall stresses, which would otherwise be manifested as the resin underwent constrained expansion in down-flow regeneration. In addition, up-flow regeneration with a relatively heavy reagent confirms the most efficient displacement of the eluant because the former is always beneath. The NTCR-PP provides for testing resin dissolution in the digester—ion exchange column hybrid at near full-scale to maximize confidence in the design. Therefore, the NTCR-PP uses a column of full-height (to reproduce the expected full-scale pressure drop) and third-diameter to avoid compromising design details in a smaller scale prototype.

The NTCR-PP general arrangement is graphically illustrated in Figure 3. Alkaline and acidic reagents are separately grouped. In addition, equipment and reagents required for the ion exchange and resin digestion functions of the NTCR-PP are also separately grouped for operational ease.

#### **Nominal NTCR-PP Operation**

The ion exchange columns will be assembled to enable the ion exchange resin slurry to be directly added through the column top manually by means of a small pump. Resin will also be extracted through the column top by means of a vacuum-assisted transfer. A complete ion exchange cycle consists of four operations: LAW processing, LAW displacement, elution, and regeneration. Simulated LAW is fed from the feed tank by means of a positive displacement pump, past a pressure relief valve, and then down-flow through the columns (IXC-1, IXC-2) in series before discharge into the effluent tank. The effluent from the bottom of the first column is fed to the top of the second column. The nominal flow rate of simulated LAW is 45 L/hour (or 3 BV/h, as baselined for the NTCR-DP), but the pump has capacity for up to 12 BV/h. The effluents from both ion exchange columns are sampled independently. In the NTCR-DP, LAW processing continues until cesium is detected in the effluent at the maximum acceptable concentration. However, processing simulated LAW can continue to any desired end-point in the NTCR-PP to satisfy specific test objectives. In the NTCR-DP, the processed LAW would be routed to either the WTP or the bulk vitrification facility for immobilization. However, in the NTCR-PP, processed simulated LAW may be either dispositioned as hazardous waste or recycled after supplementing it with separated metals (e.g., cesium and potassium) for subsequent tests.



## Figure 3: NTCR-PP General Arrangement

The NTCR-PP will process a simplified simulated LAW containing sodium, potassium, aluminum, and cesium metals and hydroxide, nitrate, and nitrite anions at concentrations representative of those expected in the LAW feed to the NTCR-DP. The simplified simulated LAW replicates the physical (density and viscosity) and chemical characteristics important to demonstrating NTCR operation. Other metals and anions at lower concentrations in LAW are important in affecting the chemical performance of the resin, but these effects are better explored at the bench-scale to minimize cost. The inclusion of aluminum in the simplified simulated LAW is important because it will precipitate as the solution pH is reduced. The precipitation of aluminum is an important phenomenon to consider in optimizing NTCR operation as both basic and acidic process solutions are processed.

The simulated LAW feed tank should be empty before proceeding to resin elution and regeneration as it serves as the effluent tank for the feed displacement and regeneration operations. The lead column or both the lead and guard columns can be eluted and regenerated either down-flow or up-flow, depending on the tested operational scenario. Feed displacement, elution, and regeneration are performed by pumping reagents from the feed tanks. Sampling valves enable the elution and regeneration effluents and feeds to be sampled. The raw reagents (concentrated solutions or solid material) are directly added to the feed tanks manually by means of a small pump and hose. The desired volume of de-mineralized water is then added and the tank contents are mixed to produce the required process solution.

The first operation displaces the simulated LAW from the system by pumping a lower density feed displacement solution (either a neutral or caustic salt solution, baselined 0.1 M sodium hydroxide in the NTCR-DP) from the tank at a nominal flow rate of 45 L/hour (3 BV/hour) down-flow through the columns (as baselined for the NTCR-DP). Down-flow is preferred because a reagent with a density lower than the simulated LAW makes sure of efficient plug-flow and, therefore, most efficient LAW displacement.

The resin is eluted with nominally 0.5 M nitric acid at a flow rate of 23 L/hour (1.5 BV/hour) up through the columns (as baselined for NTCR-DP) from the tank. For the NTCR-DP, up-flow is preferred because it minimizes contamination of the equipment and piping from the columns when operated in a down-flow manner. Contamination of subsequently treated LAW is thereby averted. The eluant can be chilled to no less than  $10^{\circ}$ C by routing it through the chiller loop. Chilling the eluant may prove beneficial in increasing resin life, but may also reduce elution efficiency, and the option will be tested given the trade-off. The eluate is directed into the eluate tank where it will be neutralized with the subsequent regeneration effluent and sentenced for disposal.

Finally, the resin is regenerated with a caustic salt solution at a flow rate of 45 L/hour (3 BV/hour) up through the columns (as baselined for the NTCR-DP). The NTCR-DP baseline calls for the regeneration solution to be a caustic salt solution sufficiently concentrated that its density is greater than that of the simulated LAW. Therefore, a solution of 3 M sodium nitrate and 2 M sodium hydroxide is baselined for the NTCR-DP. A heavy solution is used to minimize the turbulence in the column headspace manifested as the simulated LAW is subsequently introduced down-flow (i.e., the LAW displaces the regeneration solution from the system in plug-flow). Fluidized regeneration may be advantageous in avoiding bed stresses and agglomeration that may otherwise occur as the resin expands. Nevertheless, up-flow regeneration is preferred for the NTCR-DP to again minimize contamination of the equipment and piping from the columns when operated in a down-flow manner. The initial volume of effluent is directed to the eluate tank where it will neutralize the eluate. In the NTCR-DP, neutralization of the acidic eluate is necessary for it to be transferred for storage in a mild steel DST at the Hanford site tank farm. The remaining regeneration effluent is routed to the LAW feed tank from where it can be slated for waste disposal. The system can be rinsed with de-mineralized water from tanks at any point in the elution and regeneration sequence.

In the NTCR-DP, the resin must be periodically replaced as its performance degrades as a result of chemical oxidation and radiation. The spent resin is removed from the column by dissolving it in warm nitric acid; 3 - 5 M nitric acid at 70oC is baselined for the NTCR-DP after elution. The NTCR-PP allows for the resin to be dissolved from only the lead column. The equipment and process required for dissolving the resin from the guard column are identical and so are not replicated in the NTCR-PP for simplification. The digestion acid (nominally 3 M nitric acid, as baselined for the NTCR-DP) is prepared by adding the desired volume of demineralized water to the tank and then adding concentrated stock by means of a portable pump and hose. Higher concentrations of acid are unlikely to be used since initial testing indicates that the dissolution process becomes difficult to control. The tank contents are then mixed and heated to the desired temperature up to  $90^{0}$ C.

Concurrent with the preparations for the digester circuit, the scrub acid is prepared. As for the digestion acid, the scrub acid is prepared from concentrated nitric acid and hydrogen peroxide stocks, which are added to the tank manually by means of a portable pump and hose. A solution with the desired concentration is made up by adding a metered quantity of de-mineralized water and mixing with the agitator. The scrub acid solution is pumped to the top of the scrubber, contacts the off-gas, and is returned to the tank. The scrub acid product can be sampled. The scrub acid will eventually be slated for disposal.

At this point, the dissolution acid is re-circulated through the exchange column at the desired flow rate (nominally 262 L/h, or 3 BV/h as baselined for the NTCR-DP) using a pump. The feed and product digester acid can be sampled as needed. Any off-gas is entrained into the recirculating dissolution acid and passed to a tank where it is released for scrubbing. Once the resin is completely dissolved, the solution is held in a tank at up to  $90^{0}$ C to convert the dissolved organic compounds to carbon dioxide and nitrous oxide gases and water. The dissolution acid is slated for disposal once the organics concentration has reduced to the value desired to satisfy test objectives.

The off-gas is fed to the scrubber base and flows under its own pressure to the facility off-gas system. Nitrogen dioxide is absorbed into the scrub acid. The hydrogen peroxide dissolved in the scrub acid oxidizes any nitrous oxide to the more readily absorbed nitrogen dioxide. The off-gas can also be diverted around the scrubber if the nitrogen dioxide concentration is sufficiently low. Valves can be used to sample the off-gas before and after scrubbing, respectively.

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