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ARTICLE

Rapid Analytical Methodology for Chloride Molten Salt Reactor Safeguards and Process Monitoring

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Technologies that enable near real-time isotopic analysis of advanced molten salt reactor (MSR) fuels are critically needed to safeguard these reactors, increase their operational efficiency, and enable their widespread deployment with confidence. We present a systematic approach to developing near real-time dissolution and chemical isolation of U, Pu, and major fission products from highly radioactive chloride molten salt samples. Chemical yields greater than 95% were observed for both uranium and the lanthanides. Interference reduction enabled the detection and quantification of key diagnostic isotopes (including ^{112}Ag, ^{147}Nd, and ^{153}Sm) that were previously undetectable in the original sample. The results from this initial scoping study lay the foundation for the development of future automated systems that can enable cost-efficient, near-real time chemical separation and analysis of extremely highly radioactive molten salt samples.

KEYWORDS: molten salt reactor, safeguards, rapid analysis, real-time

I. Introduction

Actinide and fission product isotopic analyses are key to safeguarding nuclear reactors and providing diagnostic evaluation of fuel performance (including fuel burnup). In solid fueled reactors, spatially resolved fissile material production and burnup are routinely assessed by the dissolution, chemical separation and analysis of fuel pins taken from various locations within the core. Due to their high radioactivity, these fuel pins are often allowed to cool for months to years prior to processing. This results in a significant decrease in the total radioactivity within the fuel as shorter-lived isotopes decay to their longer-lived progeny. It also allows for physical material accountancy protocols until final fissile material mass balance evaluations are performed via isotopic analyses following spent fuel reprocessing.

Molten Salt Reactors (MSRs) are a promising technology being pursued for closing the nuclear fuel cycle and ensuring long-term reliable sources of energy. Unlike traditional solid fueled reactors, however, the liquid nature of the MSR reactor core lends itself to online reprocessing for the extraction of fissile material isotopes (e.g., U-233, Pu-239, etc.)[1-4]. In order to ensure material accountancy and support the safeguarding of these reactors, new approaches for isotopic monitoring of highly radioactive molten salts must be developed.

Current state-of-the-art analytical chemistry techniques are not able to overcome the unique challenges associated with near real-time isotopic analyses of freshly irradiated nuclear fuels. For example, freshly irradiated nuclear samples contain a complex mixture of fission products and actinides. While many peak yield fission products (including ^{99}Mo, ^{95}Zr, ^{97}Zr, ^{132}Te, ^{140}Ba, and others) may be measured directly by gamma analyses of the irradiated sample, detection and/or quantification of low yield fission products and/or low gamma emitting isotopes (including ^{112}Ag, ^{147}Nd, and ^{153}Sm) by direct gamma spectrometry measurements is not possible due to the high radiation dose rates and high Compton background (resulting in high detection limits and detector dead times). To further complicate analyses, radioactive isotopes from one element are continuously decaying to produce isotopes of different elements, the result of which is the production of an overwhelming number of rapidly changing radiometric and mass spectrometric interferences. Finally, the high dose rates of these samples (often greater than 100 R/hr; 1 Sv/hr) pose a significant challenge to both personnel safety and equipment longevity during processing and analysis.

To remove radiometric interferences and enable the quantification of key actinide and fission product isotopes for material accountancy, samples must first be dissolved and chemically separated. In nuclear facilities around the world, these dissolutions and separations are frequently performed using open beaker dissolver vessels and chromatographic column separations. Because of the unique chemistry of each actinide/fission product element, techniques designed to purify one element are frequently incompatible with techniques employed to purify other target elements. To overcome this, traditional approaches employ sample evaporations and/or transpositions in between separation steps. Such techniques are necessitated due to the limited availability of fundamental chromatographic tools at present. The end result is a process that, while effective under certain circumstances, is time consuming, expensive, and relies heavily upon the skill of the technician in order to avoid sample losses and/or contamination events. At Idaho National Laboratory (INL) such a process can frequently take upward of a week to accomplish, with the risk for contamination present throughout the process.

In this work, we report preliminary steps to develop approaches for extremely rapid processing of highly radioactive molten salt samples, with the ultimate objective of

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developing systems and approaches that can enable near real-time isotopic analysis of freshly irradiated molten salt samples. Herein, we report initial studies evaluating the dissolution characteristics and simple chemical separation approaches for processing molten salt samples using aqueous based techniques. A systematic investigation of the optimal nitric acid and hydrochloric acid dissolution conditions for various chloride molten salt matrices is reported. Dissolutions are then directly coupled to a three-column extraction chromatography system for the extraction of U, Pu, Tc and the lanthanides. Preliminary demonstration of the approach is then performed using salt samples containing fresh fission products and actinides, with final analysis of the purified samples performed using gamma spectrometry. The foundation this work establishes for supporting subsequent development of near real-time isotopic analytical systems for molten salt reactors is further discussed.

II. Experimental Approach

Unless otherwise specified all reagents in this work were TraceMetals Grade or better. NaCl (≥ 99.95%), KCl (≥ 99.0%), LiCl (≥ 99.98%) were obtained from Sigma Aldrich. Inductively Coupled Plasma Mass Spectrometry (ICP-MS) elemental standards were obtained from High Purity Standards (ICP-MS A, B, and C).

Dissolution Optimizations

Most proposed chloride-based molten salt reactors employ NaCl, KCl, and/or LiCl as the primary salt in the fuel, either as individual salts or eutectic mixtures of multiple salts together.[5-10] As the combinations reported in the literature for mixed eutectic systems can vary, for this initial study we chose to study each salt system independently (thereby providing a foundation for applied, follow-on efforts).

To determine the solubility of each salt under different HNO_3 and HCl concentrations, ten to twenty-five milliliter solutions containing different HNO_3 and HCl acid concentrations were placed into individual 50 mL polypropylene centrifuge tubes. Small quantities of each salt were then added to each sample, following which samples were agitated by hand to facilitate dissolution. Once the solubility limit had been reached (as determined by visual inspection), the mass of total salt able to be dissolved was recorded and the solubility was determined via the total mass able to be dissolved divided by the milliliters of solution.

Column Separation Design

The multi-column separation approach employed in this work was designed based upon evaluation of published distribution coefficient data on several commercially available ion exchange and extraction chromatography resins.[12] For design purposes, we limited selection of the initial load solution matrix to either HCl or HNO_3 based solutions. In the case of HCl based separations, theoretical design was performed by simply comparing the distribution coefficient data for multiple resins at different concentrations to those obtained in pure HCl solutions.

For HNO_3 based dissolution conditions, the load solution matrix will contain a mixture of both Cl^- and NO_3^- anions. To simplify scoping evaluations, resin selection was downselected to only those that showed high similarities in sorption characteristics under both Cl^- and NO_3^- conditions. Ultimately, Eichrom TEVA resin was selected for Pu extraction, UTEVA for U extraction, and RE resin for the lanthanides. The solution conditions for Mo and Tc elution were selected based upon our best initial guesses informed by prior published work investigating similar separations on non-salt containing, complex fission product solutions.[11]

Simulated Uranium-Fission Product MSR Sample Production

For evaluation of a preliminary prototype flowsheet and system, fission products and uranium-237 were produced using the photofission process. 1.27 grams of depleted uranium metal were packaged and irradiated using the high-power S-band electron linear accelerator at Idaho State University's Idaho Accelerator Center. The irradiation lasted for 4 hours, with a 38 MeV end-point energy, 85 mA current, and 9.7 microsecond pulse width. Following irradiation, the sample was allowed to cool overnight, then shipped to Idaho National Laboratory for subsequent chemical processing.

Upon arrival at INL, the uranium-fission product sample was transferred to a Savillex PTFE vial. Nine milliliters of 9 M HNO_3 was added to the sample and the sample was heated on a hotplate until it was dissolved. Following dissolution, an aliquot of the solution was obtained and analyzed via gamma spectrometry. 400 µL aliquots of this sample were then transferred into individual 30 mL PTFE vials. Samples were evaporated to dryness and wet ashed three times with concentrated hydrochloric acid. Samples were then reconstituted in 400 µL of concentrated HCl and 20 mg of an appropriate salt (NaCl, KCl, LiCl). Residual solution was then evaporated using a heat lamp.

Prototype MSR Analytical System Evaluation

A simplified approach for extracting U, Pu, and several key fission products (Tc and the lanthanides as a group) was developed. A summary of this approach is given in **Fig. 1**.

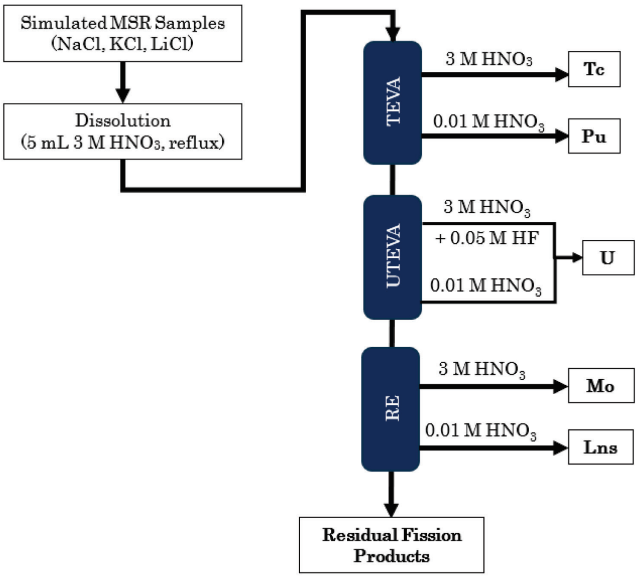

Fig. 1 Flowsheet for the chemical separation scheme applied in this work

Briefly, 5 mL of 3 M HNO_3 was added to each salt sample (NaCl, KCl and LiCl). Samples were then capped and refluxed for 30 minutes at 130 ^{0}C hotplate temperature (~80 ^{0}C solution temperature) to ensure complete dissolution. Following dissolution and analysis by gamma spectrometry, samples were pumped via peristaltic pumping through the prototype, semi-automated, three-column chemical separation system. A triple stack TEVA-UTEVA-RE column system was employed in this work. For the NaCl sample, three 10 mm diameter x 3 cm high columns were employed (~2.4 mL resin per column). For the LiCl and KCl samples, the UTEVA column was replaced with a 6.6 mm diameter x 10 cm high column (~3.4 mL resin).

Each dissolved sample was first pumped through all three columns in tandem. Following loading, the columns were rinsed using 10 mL of 3 M HNO_3. The valves were then turned to elute through each column in parallel, following which Tc was eluted from the TEVA column using 10 mL 3 M HNO_3, Pu was eluted from the TEVA column using 10 mL of 0.01 M HNO_3, U was eluted from the UTEVA column using 10 mL of 3 M HNO_3 + 0.05 M HF followed by 10 mL 0.01 M HNO_3, Mo was eluted from the RE column using 10 mL 3 M HNO_3, and the lanthanides were eluted from the RE column using 10 mL 0.01 M HNO_3.

Gamma Spectrometry Analyses

Radioactive samples were first analyzed using an IDM-200-V Ortec high-purity germanium (HPGe) gamma spectrometer. Detector efficiency curves were constructed through the use of a series of calibration sources (^{137}Cs, ^{60}Co, ^{152}Eu) at 10, 20, and 50 cm. Detailed Monte Carlo modeling was used to simulate the calibration sources within the uncertainty of the calibration source (1% at 1σ). Monte Carlo simulations were then performed to extrapolate the detector efficiency curves to the specific source geometries and distances employed for each sample analysis.

III. Results

Salt Dissolution Studies

Figure 2 shows the solubility of NaCl, LiCl, and KCl as a function of the concentration of HNO_3 and HCl. For all three salts, the solubility is observed to steadily decrease with increasing acid concentration. Similar solubilities are observed for both NaCl and KCl in both HNO_3 and HCl, with solubilities on the order of ~0.35 g/mL at 0.01 M and ~0.05-0.08 at 5 M. By 10 M, the solubility of NaCl and KCl decreases significantly, with only milligram levels or less able to be dissolved in 10 mL. For LiCl, significantly higher solubilities than those of NaCl and KCl are observed, with a solubility on the order of ~0.75 g/mL at 0.01 M, that decreases to ~0.4 g/mL by 10 M (for both HNO_3 and HCl).

To enable near-real time separation of highly radioactive molten salts, several important dissolution parameters must be met. First, the inert salt carrier, the actinide fuel, and the fission products must be able to be rapidly and completely dissolved. Second, the conditions selected for the dissolution must be compatible with downstream separation chemistry.

In the case of dissolving the inert salt, the data in Fig. 2 indicates that lower acid concentrations are more favorable for maximizing salt solubilities than higher concentrations. On the other hand, prior work by our group has shown that dissolution of the actinide fuel is much faster at higher concentrations of acid[11]. Therefore, a compromise between the solubility of each component may be essential in order to achieve the overall objective of complete sample dissolution in a rapid timescale.

Based upon solubility observations from this work, combined with prior chemical separation distribution coefficient data from our group[12], 3 M HNO_3 was selected for the dissolution matrix as it would facilitate both dissolution of the salt (at levels greater than ~80 mg/mL) and the fuel, as well as downstream chemical separations (which are currently optimized for a 3 M HNO_3 load solution as previously reported[11-13].

The dose rates measured for select chemically separated fractions are provided in **Table 1**, while a full summary of the gamma-emitting radionuclides measured via gamma-ray spectrometry is provided in **Table 2**. Chemical separation of the original sample enabled reduction in the dose rates for each of the separated fractions down to 19.5%, 0.4%, and 21.2%

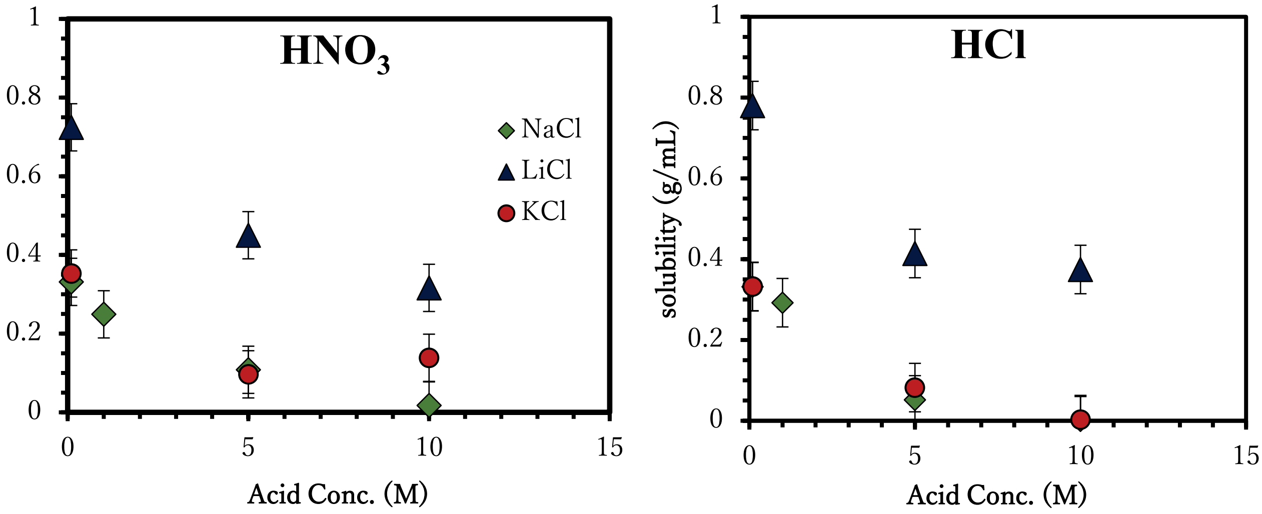

Fig. 2 Solubility data for NaCl, LiCl, and KCl as a function of concentration of (left) nitric acid and (right)

Table 1 Dose rates observed for chemically separation fractions of the NaCl sample

	U Fraction	Pu Fraction	FP Fraction	Tc Fraction	Mo Fraction	Ln Fraction
Contact Dose Rate (mR/hr)	417	9	1,180	29.7	50	453
% Total Dose	19.5%	0.4%	55.2%	1.4%	2.3%	21.2%

Table 2 Gamma-emitting radionuclides detected in various fractions for the LiCl, KCl, and NaCl molten salt simulant samples

Isotope	Salt Type	U Fraction	Pu Fraction	FP Fraction	Mo Fraction	Tc Fraction	Ln Fraction
^{91}Sr Activity	LiCl	< MDA	< MDA	< MDA*	< MDA	< MDA	< MDA
	KCl	< MDA	< MDA	< MDA*	< MDA	< MDA	< MDA
	NaCl	< MDA	< MDA	98.44 ± 9.52%	< MDA	1.56 ± 0.87%	< MDA
^{95}Zr Activity	LiCl	6.97 ± 0.86%	0.75 ± 0.13%	80.93 ± 5.21%	3.40 ± 0.56%	4.26 ± 0.60%	3.68 ± 0.65%
	KCl	6.69 ± 0.46%	0.74 ± 0.10%	82.36 ± 5.13%	3.43 ± 0.41%	3.85 ± 0.36%	2.93 ± 0.21%
	NaCl	4.81 ± 1.10%	0.72 ± 0.21%	84.99 ± 5.65%	3.19 ± 0.42%	2.90 ± 0.94%	3.39 ± 1.53%
^{99}Mo Activity	LiCl	2.14 ± 0.60%	0.24 ± 0.15%	7.48 ± 1.28%	82.07 ± 6.14%	1.54 ± 0.61%	6.54 ± 1.07%
	KCl	2.89 ± 0.34%	0.15 ± 0.07%	16.71 ± 1.32%	74.86 ± 5.37%	2.10 ± 0.29%	3.29 ± 0.27%
	NaCl	0.64 ± 0.23%	0.25 ± 0.07%	54.71 ± 3.94%	41.70 ± 3.13%	1.25 ± 0.31%	1.44 ± 0.50%
^{132}Te Activity	LiCl	2.02 ± 0.26%	< MDA	95.14 ± 9.47%	1.52 ± 0.20%	1.32 ± 0.17%	< MDA
	KCl	2.10 ± 0.21%	< MDA	94.67 ± 9.40%	1.45 ± 0.16%	1.77 ± 0.18%	< MDA
	NaCl	0.36 ± 0.07%	< MDA	98.25 ± 9.78%	0.56 ± 0.09%	0.83 ± 0.11%	< MDA
^{132}I Activity	LiCl	16.08 ± 5.92%	3.66 ± 4.54%	53.73 ± 221.78%	2.91 ± 8.83%	2.78 ± 3.16%	20.85 ± 48.55%
	KCl	59.54 ± 22.55%	0.99 ± 2.29%	0.03 ± 0.10%	2.24 ± 1.92%	1.23 ± 1.61%	35.98 ± 67.54%
	NaCl	45.08 ± 5.89%	11.16 ± 2.18%	< MDA	< MDA	11.60 ± 5.34%	32.17 ± 39.43%
^{140}Ba Activity	LiCl	2.40 ± 0.57%	0.23 ± 0.09%	93.42 ± 6.06%	1.72 ± 0.42%	1.41 ± 0.33%	0.82 ± 0.43%
	KCl	2.55 ± 0.26%	0.15 ± 0.04%	93.12 ± 5.92%	1.57 ± 0.29%	1.93 ± 0.22%	0.68 ± 0.09%
	NaCl	0.49 ± 0.37%	0.22 ± 0.10%	96.55 ± 6.37%	0.81 ± 0.25%	0.88 ± 0.46%	1.05 ± 0.76%
^{143}Ce Activity	LiCl	< MDA	< MDA	< MDA	< MDA	< MDA	100.00 ± 6.67%
	KCl	3.70 ± 0.51%	< MDA	< MDA	< MDA	1.63 ± 0.28%	94.68 ± 5.99%
	NaCl	0.42 ± 0.10%	0.11 ± 0.02%	< MDA	< MDA	0.83 ± 0.13%	98.63 ± 6.27%
^{147}Nd Activity	LiCl	< MDA	< MDA	< MDA	< MDA	2.47 ± 1.26%	97.53 ± 16.95%
	KCl	2.35 ± 0.46%	< MDA	< MDA	< MDA	3.82 ± 1.01%	93.83 ± 16.05%
	NaCl	< MDA	< MDA	< MDA	< MDA	< MDA	100.00 ± 17.92%
^{237}U Activity	LiCl	94.46 ± 6.26%	0.23 ± 0.02%	< MDA	< MDA	5.32 ± 0.35%	< MDA
	KCl	93.33 ± 5.78%	0.23 ± 0.02%	< MDA	0.02 ± 0.01%	6.25 ± 0.42%	0.17 ± 0.01%
	NaCl	80.01 ± 3.90%	0.06 ± 0.01%	< MDA	< MDA	1.71 ± 0.12%	18.22 ± 1.21%
^{239}Np Activity	LiCl	< MDA	10.63 ± 3.41%	< MDA	< MDA	< MDA	89.37 ± 17.91%
	KCl	4.60 ± 2.07%	21.61 ± 2.27%	< MDA	< MDA	< MDA	73.79 ± 5.51%
	NaCl	< MDA	1.61 ± 0.63%	11.58 ± 5.41%	< MDA	< MDA	86.81 ± 8.84%

**Analyses performed several days following the fission event; ^{91}Sr in the KCl and LiCl samples had decayed away and thus was not detectable*

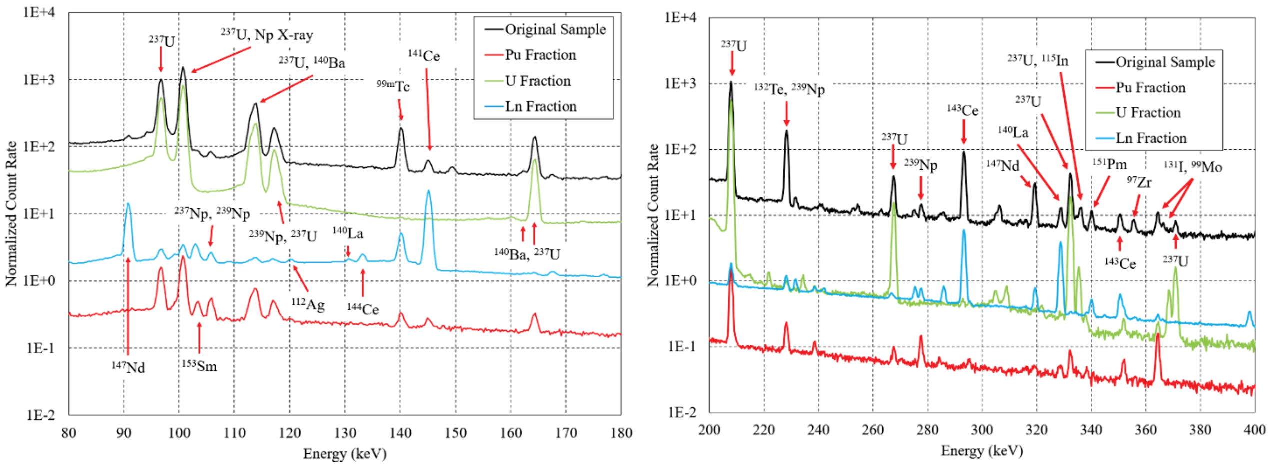

Fig. 3 Gamma ray spectrometry comparison of the original sample versus the Pu, U, and the Ln fractions at (left) 80- 180 keV and (right) 200-400 keV

of the original dose rates for U, Pu and the lanthanides (respectively), with the majority of the dose for the uranium and lanthanide fractions resulting from ^{237}U and lanthanide isotopes (rather than interferences). The value in this dose reduction can be best illustrated through the plutonium fraction (where the only gamma emitting radionuclides were contaminants, as opposed to the other fractions). Chemical separation of this fraction enabled reduction in the beta/gamma dose rate from 2.1 R/hr down to 9 mR/hr. As standard silicon surface barrier detectors used for alpha spectrometry are highly sensitive to beta and gamma radiation, this three-order of magnitude reduction is a significant step forward for enabling the coupling of a prototype separation system with alpha spectrometry detection. This dose rate reduction would similarly benefit other techniques including mass spectrometry by decreasing the dose to personnel and potentially increasing the lifetime of equipment components.

Further value for rapid chemical can be seen in the separated lanthanide fraction, shown in **Fig. 3**, by comparing the Ln and original sample curves in blue and black, respectively. Chemical yields for the lanthanides for the NaCl, KCl, and LiCl separations were all greater than 95%, indicating high suitability for the RE Resin column for future application to lanthanide purification from highly radioactive fission product samples (as anticipated). The chemical purity for the lanthanide fraction was also observed to be very high, with the majority of contamination resulting from iodine and neptunium isotopes. The high recovery and purification from other gamma emitting radionuclides enabled the detection of multiple low-fission yield isotopes such as ^{147}Nd (using the 91 keV photopeak) and ^{153}Sm (103.2 keV), which were not able to be detected in the initial sample due to the high Compton background. The detection of these isotopes is a significant achievement as these isotopes can be employed as diagnostic indicators of the fuel burnup and therefore the performance and operational history. Furthermore, as many of these isotopes are relatively short-lived (e.g., with half-lives less than 15 days), the data from most of these isotopes is not currently able to be exploited since they will have decayed by the time samples can be shipped to a standard analytical laboratory for analysis. Another example of the value of chemical separations for lanthanide characterization is ^{147}Nd; while this isotope can be detected in the unseparated sample, its quantification is complicated by the overlapping photopeak from ^{133}I (20.8 hour half-life), which has a much larger relative yield at early examination times. At analysis times approximately less than 3 days following the fission event, these high yields can prevent quantification of the ^{147}Nd even with multiplet fitting for the photopeaks. On the other hand, chemical separations employed in this work enabled minimization of iodine interferences and thus significant improvement in ^{147}Nd quantification.

The removal of Tc may in some cases be desired as ^{99m}Tc can contribute appreciably to the beta/gamma dose for samples up to approximately 2 weeks following sample collection. Prior work by our team has shown that if care is not taken to isolate ^{99m}Tc at the beginning of the separation, it may end up contaminating multiple different fractions, making it challenging to obtain high purity separated samples. For this reason, the initial test separation evaluated in this work employed a 3 M HNO_3 load and rinse solution, followed attempted ^{99m}Tc elution using 0.01 M HNO_3. While distribution coefficient values for Tc show that it is only weakly bound to the column at 3 M HNO_3, preliminary work by our group using uranium-fission product samples without salts, using different column lengths and elution volumes, has shown that Tc can still be retained using TEVA resin under these conditions given sufficient column length for the TEVA column. On the other hand, during all three salt samples in this work, instead of being retained on the TEVA column, Tc passed directly through and was collected in the column raffinate (the "FP" fraction). Thus, for future iterations, should Tc recovery be desired, a longer column or lower acid concentration is recommended.

IV. Conclusion

This works reports initial scoping evaluations of rapid chemical separations approaches for irradiated NaCl, KCl, and LiCl samples containing short-lived uranium and fission product isotopes. The solubilities and dissolution speeds for uranium bearing NaCl, KCl, and LiCl salts was evaluated and optimized. In the case of uranium, the use of a longer column for the KCl sample and adequate rinsing enabled high recovery of U (> 93%) with very high chemical purity from the fission products (excepting radioiodines). In the case of the plutonium fractions, very high dose reduction was observed, with the final purified Pu fraction representing 0.4% of the initial sample beta/gamma activity. While plutonium analyses for plutonium recovery is ongoing and will be reported in a future publication, the initial results in this work show significant promise for rapidly decreasing the radiometric impurities in the plutonium fraction, thereby improving the ability to enable follow-on plutonium analyses by alpha spectrometry and similar methods. The value to rapid, high-purity chemical separations is further illustrated with the lanthanide fraction, where isolation of the lanthanides in high yield (greater than 95%) and purity enabled the detection and quantification of several potential diagnostic isotopes (including ^{112}Ag, ^{147}Nd, and ^{153}Sm) which were previously undetectable in the initial sample due to high Compton background interferences. Finally, the use of the prototype, semi-automated chemical separation system employed in this work resulted in significant reduction in personnel dose during handling, with the dose rate reduction estimated by our team (based on the dose taken by personnel during similar processes employed on aged uranium-fission product samples) estimated to be more than 100 fold decrease in dose versus gravimetric separations. Future work building upon this initial study will seek to further improve chemical recoveries and purities, purify and quantify additional actinide isotopes including Th, Am, Np and Pu, and evaluate other molten salt reactor matrices including fluoride-based salts as well as U-Th based systems. These studies will enable the design and construction of automated system(s) that can then be employed for near-real time chemical separation and analysis of extremely highly radioactive molten salt samples from molten salt reactors. Coupling of these systems to high-precision analytical tools can then be employed to quantitatively explore the measurement precision that can be attained when employing this methodology (along with its associated implications on uncertainties in the total fissile material within the reactor at any given time). This and subsequent follow-on studies can then be leveraged to enable automated system development for rapidly processing freshly irradiated molten salt samples with dose rates greater than 100 R/hr (1 Sv/hr), thereby enabling future near real-time analyses of molten salt samples from advanced molten salt reactors.

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