



Salt Sample Statistical Study

March 2022

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ABSTRACT

Historically there has, at times, been large variability in measured uranium concentration amongst salt samples taken at the same time from the electrorefiner with relative standard deviations ranging from 0.59% up to a high of 114%. This has led to uncertainty in the actual uranium content of the salt. A series of surrogate experiments were performed with gadolinium to study the effects of Li_2O , Gd_2O_3 , and Gd metal additions to an LiCl-KCl-GdCl_3 salt on the gadolinium content of the salt. These experiments showed no increase in salt sample variability following the additions of the mentioned species, indicating that the carryover of these species from the oxide reduction process may not lead to the historic variability issues.

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ACRONYMS

ER	Electrorefiner
H ₂ O	Water (moisture)
HFEF	Hot Fuel Examination Facility
ICP-OES	Inductively Coupled Plasma – Optical Emission Spectroscopy
INL	Idaho National Laboratory
KCl	Potassium chloride
Li ₂ O	Lithium oxide
LiCl	Lithium chloride
LKE	Lithium chloride – potassium chloride eutectic
MFC	Materials and Fuels Complex
O ₂	Oxygen
OR	Oxide reduction
RCL	Radiochemistry Laboratory
RSD	Relative standard deviation
U	Uranium
UCl ₃	Uranium trichloride
UO ₂	Uranium dioxide
UOCl	Uranium oxychloride
ΔG	Gibbs free energy change

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Salt Sample Statistical Study

1. INTRODUCTION

Electrochemical processing of used nuclear fuel has been studied at the Idaho National Laboratory (INL) for several decades. Throughout this history, at times, high variability of uranium (U) concentration has been encountered. The following report summarizes some proposed explanations for this variability. A series of surrogate experiments were performed to test some of these possible explanations to better understand both the cause, and propose solutions to, this issue.

2. BACKGROUND

Equipment for recycling used LWR oxide fuel has been operated in the Hot Fuel Examination Facility (HFEF) argon hot cell, located at INL's Materials and Fuels Complex (MFC) for a little over five years. During this time several different campaigns with different fuels (total of greater than 25 kg heavy metal content) have been performed using a coupled oxide reduction (OR) module and an electrorefiner (ER). These oxide fuels were electrochemically reduced in a LiCl-Li₂O molten salt in the OR, followed by salt distillation prior to transfer into the ER containing primarily LiCl-KCl-UCl₃. Incomplete reduction in the OR can result in a mixture of U, UO₂, LiCl, Li, and Li₂O going into the OR salt distillation operation. Incomplete distillation can then lead to a mixture of U, UO₂, LiCl, Li₂O, and any other products of reactions that may have taken place during distillation being transferred into the ER salt bath.

Throughout the years of operations, ER salt samples have been taken at approximately 50 distinct times with multiple samples taken each time. At times a high variability of U concentration in the salt among samples taken at the same time has been encountered. Relative standard deviation (RSD) has ranged from 0.59% up to a high of 114% (as shown in Figure 1) leaving a large uncertainty in the actual uranium content of the salt.

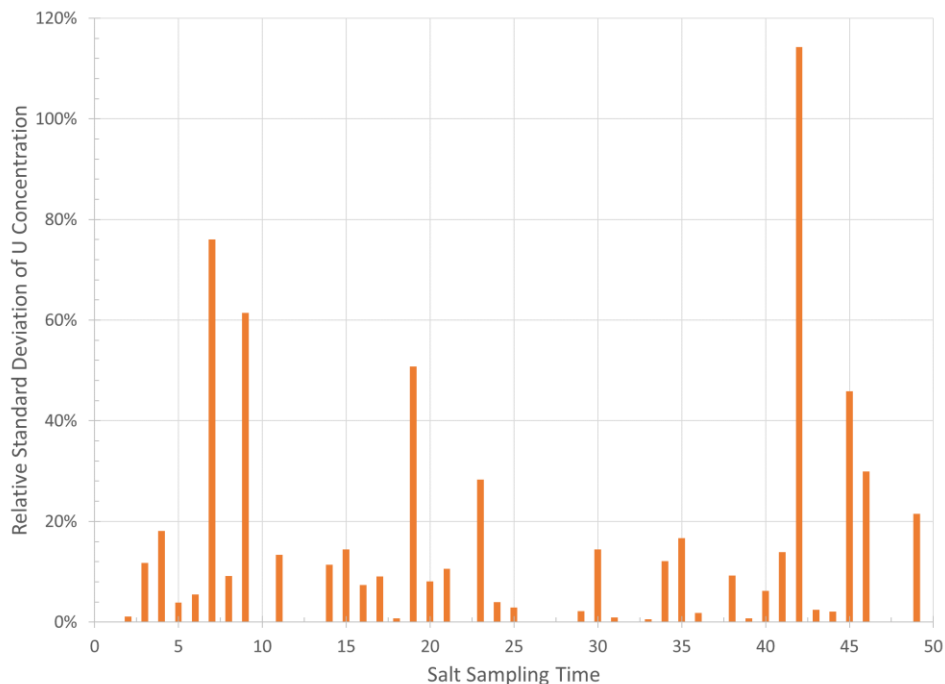
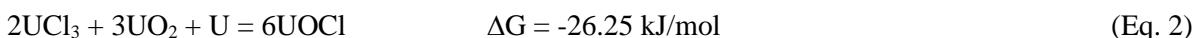


Figure 1. Relative standard deviation of Uranium concentration at different times throughout the history of ER operations in HFEF.

Note that for times without RSD values, samples were either acquired but not analyzed, or only one sample was analyzed.

3. POSSIBLE REACTIONS RESULTING IN INSOLUBLE PRODUCTS

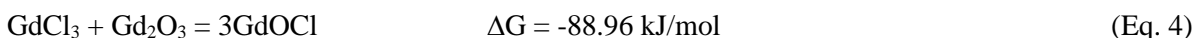
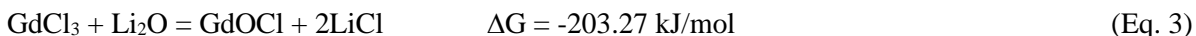
With the potential for carryover of components from the OR process discussed in Section 2, there exist some chemical reactions that can occur resulting in potentially insoluble reaction products. It has been proposed that one potential source of variability could be due to the presence of insoluble particles of uranium small enough to be slow to settle out of the salt. Some of these possible reactions were identified in a previous study performed at INL (Ref. 1) on oxychloride formation. The Gibbs free energy change (ΔG) for the reactions below were determined at the nominal ER operating temperature (500 °C) using HSC Chemistry 9 (Outotec).



In addition to the chemical reactions mentioned above, previous work at INL has brought up the possibility of some solubility of uranium metal in the ER salt in addition to formation of small, unsettled particles of uranium metal in the salt detached from the bulk fuel and/or collected purified uranium at the cathode during the electrochemical process. During this process the morphology of the uranium metal changes from a dense consolidated mass at the anode to a less-dense dendritic deposit at the cathode. This dendritic deposit has been known to detach from the cathode and, if large settle to the bottom, or if small remain suspended in the salt.

4. EXPERIMENTAL METHODS

To help determine the effects of these potential mechanisms on salt sampling a series of tests were performed with gadolinium as a surrogate for uranium. As with uranium, gadolinium exists as a trivalent species in the LiCl-KCl eutectic (LKE) salt at 500 °C. One difference, which leads to some expected different behavior involves the oxide form. Uranium is generally present as UO_2 , while gadolinium is Gd_2O_3 . The expected reactions that correspond to Equations 1 and 2 are shown below in Equation 3 and 4 with ΔG values calculated using HSC Chemistry 9 (Outotec).



All experiments were performed at 500 °C in an argon atmosphere glovebox in which the O_2 level remained below 20 ppm and the H_2O level remained below 1 ppm. Three separate LiCl-KCl-GdCl₃ salt baths were prepared in alumina (Al_2O_3) crucibles, and different combinations of gadolinium metal (Gd), gadolinium oxide (Gd_2O_3), and lithium oxide (Li_2O) were added to each. Following each addition, the molten salt was manually stirred using a stainless-steel rod and allowed to react/dissolve overnight. Following the overnight period, the salt was again manually stirred, allowed to settle for five minutes, and then a salt sample was taken by rapidly dipping a cold (room temperature) stainless-steel rod partially into the salt bath. An example of a salt sample taken using this method is shown in Figure 2. Five samples were taken from each salt mixture using this same technique.



Figure 2. Sample of LiCl-KCl-GdCl₃ salt taken using a cold stainless-steel rod.

The first LiCl-KCl-GdCl₃ mixture was prepared using 179.89 g of LiCl-KCl eutectic (Sigma Aldrich, 99.9%, ultra-dry) and 20.17 g (0.077 mol) GdCl₃ (Alfa-Aesar, 99.99%, ultra-dry). This should result in a Gd concentration of 6.01 wt%. Five samples (designated B1 – B5) were taken after allowing the mixture to equilibrate at 500 °C overnight. To this mixture, 1.15 g (0.038 mol) of Li_2O (Thermo Scientific, 99.5%) was added. According to Eq. 3, this amount of Li_2O should react with 1/2 of the GdCl₃ in the salt. This salt was mixed and allowed to equilibrate overnight. Following the overnight period, the salt was manually stirred and allowed to settle prior to five samples (L1 – L5) being taken using the cold stainless-steel dip rod. After sampling, a 2.84 g lump of Gd metal (Sigma Aldrich, 99.9%) was added to the salt, mixed, and allowed to equilibrate for close to five days (115 hours). Following this period, the salt was mixed, allowed to settle for five minutes, and five salt samples (LG1 – LG5) were taken. After sampling, the furnace was turned off and allowed to cool.

The second LiCl-KCl-GdCl₃ mixture was prepared using 179.95 g of LiCl-KCl eutectic (Sigma Aldrich, 99.9%, ultra-dry) and 20.14 g (0.076 mol) GdCl₃ (Alfa-Aesar, 99.99%, ultra-dry). This should result in a Gd concentration of 6.00 wt%. Five samples (designated B6 – B10) were taken after allowing the mixture to equilibrate at 500 °C overnight. To this mixture, 7.39 g (0.047 mol) Gd metal (Sigma Aldrich, 99.9%) was added. The salt was mixed and allowed to equilibrate for almost six days (138 hours). Following this period, the salt was manually stirred and allowed to settle prior to five samples (G1 – G5) being taken using the cold stainless-steel dip rod. After sampling, 9.14 g (0.025 mol) of Gd₂O₃ (Thermo Scientific, 99.9%) was added to the salt, mixed, and allowed to equilibrate overnight. This amount of Gd₂O₃ should, according to Eq. 4, react with 1/3 of the GdCl₃ in the salt. Following this period, the salt was mixed, allowed to settle for five minutes, and five salt samples (GO1 – GO5) were taken. To this, 0.74 g (0.025 mol) of Li₂O (Thermo Scientific, 99.5%) was added. According to Eq. 3, this amount of Li₂O should react with 1/3 of the initial amount of GdCl₃ in the salt bath, or 1/2 of the GdCl₃ remaining after expected reactions with Gd₂O₃. This salt was mixed and allowed to equilibrate overnight. Following the overnight period, the salt was manually stirred and allowed to settle prior to five salt samples (GOL1 – GOL5) being taken using the cold stainless-steel dip rod. After sampling, the furnace was turned off and allowed to cool.

The third LiCl-KCl-GdCl₃ mixture contained 160.08 g of LiCl-KCl eutectic (Sigma Aldrich, 99.9%, ultra-dry) and 17.93 g (0.068 mol) GdCl₃ (Alfa-Aesar, 99.99%, ultra-dry). This should result in a Gd concentration of 6.01 wt%. Five samples (B11 – B15) were taken after allowing the mixture to equilibrate at 500 °C for approximately two hours. To this mixture, 12.31 g (0.034 mol) of Gd₂O₃ (Thermo Scientific, 99.9%) was added to the salt, mixed, and allowed to equilibrate for close to four days (88 hours). This amount of Gd₂O₃ should, according to Eq. 4, react with 1/2 of the GdCl₃ in the salt. After this equilibration period, the salt was manually stirred and allowed to settle prior to five salt samples (O1 – O5) being taken using the stainless-steel dip rod. After sampling, the furnace was turned off and allowed to cool.

A summary of all the salt samples taken along with the corresponding salt mixtures and additions and expected compositions (based on added mass of materials) is shown in Table 1. The table also shows the expected compositions based on the added mass of materials and assuming the reactions shown in Eq. 3 and Eq. 4 go to completion. Any GdOCl formed along with any Gd present is assumed to be insoluble in the salt and settles to the bottom of the salt bath. These samples were all analyzed at the Radiochemistry Laboratory (RCL) at MFC using Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES) for Li, K, and Gd content.

Table 1. Salt samples taken along with corresponding salt mixtures.

Sample Set	Salt mixture	Expected Composition (wt%)		
		Li	K	Gd
1. B1 – B5	LiCl-KCl-GdCl ₃ (179.89 g LKE) (20.17 g GdCl ₃)	6.48	26.41	6.01
2. L1 – L5	LiCl-KCl-GdCl ₃ + Li ₂ O (+ 1.15 g Li ₂ O)	6.99	27.37	3.04
3. LG1 – LG5	LiCl-KCl-GdCl ₃ -Li ₂ O + Gd (+ 2.84 g Gd)	6.99	27.37	3.04

4. B6 – B10	LiCl-KCl-GdCl ₃ (179.95 g LKE) (20.14 g GdCl ₃)	6.48	26.41	6.00
5. G1 – G5	LiCl-KCl-GdCl ₃ + Gd (+ 7.39 g Gd)	6.48	26.41	6.00
6. GO1 – GO5	LiCl-KCl-GdCl ₃ -Gd + Gd ₂ O ₃ (+ 9.14 g Gd ₂ O ₃)	6.71	27.34	4.13
7. GOL1 – GOL5	LiCl-KCl-GdCl ₃ -Gd-Gd ₂ O ₃ + Li ₂ O (+ 0.74 g Li ₂ O)	7.06	28.00	2.09
8. B11 – B15	LiCl-KCl-GdCl ₃ (160.08 g LKE) (17.93 g GdCl ₃)	6.48	26.41	6.01
9. O1 – O5	LiCl-KCl-GdCl ₃ + Gd ₂ O ₃ (+ 12.31 g Gd ₂ O ₃)	6.83	27.84	3.10

5. EXPERIMENTAL RESULTS AND DISCUSSION

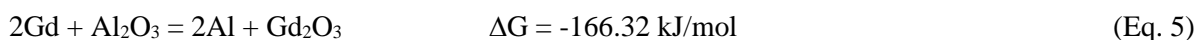
5.1 First Salt Mixture

The ICP-OES results for the samples from the first salt mixture along with results for the samples taken after the additions made are summarized in Table 2. All of the ICP-OES results can be seen in Appendix A.

The first set of salt samples (B1 – B5) were taken from the LiCl-KCl-GdCl₃ salt mixture. As mentioned in the previous section, based on the masses of constituents added, it was expected that the Gd concentration would be 6.01 wt%. The average Gd concentration of the five salt samples analyzed was determined to be 3.41 wt% with an RSD of 5.6%. The reason for this discrepancy remains to be determined. A potential reason may be that an overnight period at temperature was not long enough for the GdCl₃ to fully and homogeneously dissolve into the LiCl-KCl base salt.

After the addition of Li₂O to the mixture, it was expected that this Li₂O would react with the GdCl₃ according to Eq. 3 forming GdOCl and LiCl with the GdOCl expected to be mostly insoluble in the salt. As discussed in Section 4, the Gd concentration in the salt was expected to decrease by approximately half and the Li concentration was expected to increase slightly. The actual measured Gd concentration (L1 – L5) increased from 3.41 wt% to 3.92 wt% while the Li concentration may have decreased slightly, though within error it could be considered constant.

The addition of Gd metal to the mixture was not expected to react with any of the salt constituents. It is assumed that Gd metal has some solubility in GdCl₃, thus may have some limited solubility in LiCl-KCl-GdCl₃, which may slightly increase the Gd concentration in the salt. Gd metal is also likely to react with the Al₂O₃ crucible itself forming Gd₂O₃, which could then react with the GdCl₃ via Eq. 4, which would decrease the Gd concentration in the salt.



Results (LG1 – LG5) showed a slight decrease of Gd content in the salt, though within error it may not have changed. Throughout these samples there appears to be no addition to the base LiCl-KCl-GdCl₃

salt that greatly increased the variability as indicated by RSD, which actually showed lower variability than in the base salt.

Table 2. ICP-OES Results for samples taken from the first salt mixture.

Salt mixture	Sample	Composition (wt%)		
		Li	K	Gd
1. LiCl-KCl-GdCl ₃	B1	8.11	25.72	3.21
	B2	8.27	26.17	3.28
	B3	8.65	26.45	3.37
	B4	9.14	27.88	3.70
	B5	8.71	26.39	3.50
	Average \pm RSD	8.57 \pm 4.7%	26.52 \pm 3.1%	3.41 \pm 5.6%
2. LiCl-KCl-GdCl ₃ + Li ₂ O	L1	7.97	24.74	3.84
	L2	8.35	25.89	3.87
	L3	9.39	27.81	3.99
	L4	8.14	25.95	3.95
	L5	8.49	26.88	3.97
	Average \pm RSD	8.47 \pm 6.5%	26.25 \pm 4.4%	3.92 \pm 1.7%
3. LiCl-KCl-GdCl ₃ -Li ₂ O + Gd	LG1	8.11	25.08	3.65
	LG2	8.02	25.70	3.96
	LG3	8.57	26.29	3.74
	LG4	7.80	25.40	3.82
	LG5	8.27	26.96	4.06
	Average \pm RSD	8.15 \pm 3.5%	25.89 \pm 2.9%	3.85 \pm 4.3%

5.2 Second Salt Mixture

The ICP-OES results for the samples from the second salt mixture along with results for the samples taken after the additions made are summarized in Table 3. All of the ICP-OES results can be seen in Appendix A.

The first set of salt samples (B6 – B10) were taken from the LiCl-KCl-GdCl₃ salt mixture. As mentioned in the previous section, based on the masses of constituents added, it was expected that the Gd concentration would be 6.00 wt%. The average Gd concentration of the five salt samples analyzed was

determined to be 4.60 wt% with an RSD of 3.9%. As with the previous salt mixture, the reason for this discrepancy remains to be determined.

With the addition of Gd metal to the mixture, two situations were possible as discussed in Section 5.1, which could either increase or decrease the Gd concentration in the salt. Results (G1 – G5) showed a slight increase in the Gd concentration, which differed from the results following the addition of Gd to the previous salt mixture (Section 5.1, LG1 – LG5), showing no real change.

After the addition of Gd_2O_3 to the mixture, it was expected that it would react with the GdCl_3 according to Eq. 4 forming GdOCl . This GdOCl is expected to be insoluble in the salt and to settle to the bottom of the salt bath. As discussed in Section 4, the Gd concentration in the salt was expected to decrease by approximately 1/3. The actual measured Gd concentration (GO1 – GO5) decreased slightly from 5.46 wt% to 5.06 wt%, though not by the 1/3 that was predicted from Eq. 4.

The addition of Li_2O was expected to decrease the Gd concentration by reacting with GdCl_3 as predicted by Eq. 3 to form GdOCl and LiCl . The Li_2O was expected to react with approximately half of the GdCl_3 remaining in the salt, decreasing the Gd concentration by half while slightly increasing the Li concentration. The measured Gd concentration (GOL1 – GOL5) decreased by approximately 20% from 5.06 wt% to 4.06 wt%. The Li concentration did appear to increase slightly from 8.00 wt% to 8.23 wt%, though within errors, there may not have been any change.

The relative standard deviations of the three sets of samples taken after additions to the base salt (1.5%, 1.2%, and 1.9%) were all lower than the relative standard deviation of the base salt samples (3.9%). This data does not indicate any increase in variability due to these additions.

Table 3. ICP-OES Results for samples taken from the second salt mixture.

Salt mixture	Sample	Composition (wt%)		
		Li	K	Gd
4. LiCl-KCl-GdCl_3	B6	8.50	26.42	4.70
	B7	7.73	25.43	4.41
	B8	8.22	26.72	4.67
	B9	8.18	26.00	4.43
	B10	8.48	27.60	4.82
	Average \pm RSD	$8.22 \pm 3.8\%$	$26.43 \pm 3.1\%$	$4.60 \pm 3.9\%$
5. LiCl-KCl-GdCl_3 + Gd	G1	8.75	26.96	5.42
	G2	7.89	25.77	5.43
	G3	8.19	27.04	5.60
	G4	8.49	26.72	5.47
	G5	7.58	25.32	5.40
	Average \pm RSD	$8.18 \pm 5.7\%$	$26.36 \pm 2.9\%$	$5.46 \pm 1.5\%$
	GO1	7.84	25.83	5.10

6. LiCl-KCl-GdCl ₃ -Gd + Gd ₂ O ₃	GO2	7.62	26.12	5.06
	GO3	8.33	26.93	4.96
	GO4	7.83	26.55	5.06
	GO5	8.39	27.67	5.11
	Average ± RSD	8.00 ± 4.2%	26.62 ± 2.7%	5.06 ± 1.2%
7. LiCl-KCl-GdCl ₃ -Gd-Gd ₂ O ₃ + Li ₂ O	GOL1	8.46	26.96	4.00
	GOL2	8.43	27.59	4.15
	GOL3	7.92	26.68	4.00
	GOL4	7.82	25.89	4.01
	GOL5	8.51	27.84	4.13
	Average ± RSD	8.23 ± 4.0%	26.99 ± 2.9%	4.06 ± 1.9%

5.3 Third Salt Mixture

The ICP-OES results for the samples from the third salt mixture along with results for the samples taken after the addition made are summarized in Table 4. All of the ICP-OES results can be seen in Appendix A.

The first set of salt samples (B11 – B15) were taken from the LiCl-KCl-GdCl₃ salt mixture. As mentioned in the previous section, based on the masses of constituents added, it was expected that the Gd concentration would be 6.01 wt%. The average Gd concentration of the five salt samples analyzed was determined to be 3.69 wt% with an RSD of 4.8%. As with the previous salt mixture, the reason for this discrepancy remains to be determined.

After the addition of Gd₂O₃ to the mixture, it was expected that it would react with the GdCl₃ according to Eq. 4 forming GdOCl. This GdOCl is expected to be insoluble in the salt and to settle to the bottom of the salt bath. As discussed in Section 4, the Gd concentration in the salt was expected to decrease by approximately 1/2. The actual measured Gd concentration (O1 – O5) increased from 3.69 wt% to 4.51 wt%.

The relative standard deviation of the set of samples taken following the Gd₂O₃ addition was lower than that for the base LiCl-KCl-GdCl₃. This does not indicate that the formation of GdOCl via reaction with GdCl₃ leads to an increase in sample variability.

Table 4. ICP-OES Results for samples taken from the third salt mixture.

Salt mixture	Sample	Composition (wt%)		
		Li	K	Gd
8. LiCl-KCl-GdCl ₃	B11	7.76	26.91	3.83

	B12	8.73	27.59	3.50
	B13	8.79	27.85	3.63
	B14	8.14	26.45	3.59
	B15	8.58	28.12	3.93
	Average \pm RSD	8.40 \pm 5.3%	27.39 \pm 2.5%	3.69 \pm 4.8%
9. LiCl-KCl-GdCl ₃ + Gd ₂ O ₃	O1	7.62	25.91	4.73
	O2	8.23	26.78	4.38
	O3	8.11	26.57	4.46
	O4	8.56	27.36	4.48
	O5	8.03	26.99	4.52
	Average \pm RSD	8.11 \pm 4.2%	26.72 \pm 2.0%	4.51 \pm 2.9%

6. SUMMARY

Historically there has, at times, been large variability in measured uranium concentration amongst salt samples taken at the same time from the ER with relative standard deviations ranging from 0.59% up to a high of 114%. This has led to uncertainty in the actual uranium content of the salt. A series of surrogate experiments were performed with Gadolinium to study the effects of Li₂O, Gd₂O₃, and Gd metal additions to an LiCl-KCl-GdCl₃ salt on the Gd content of the salt.

These experiments showed no increase in salt sample variability following the additions of the mentioned species, indicating that the carryover of these species from the OR may not lead to the historical variability issues.

7. FUTURE WORK

Despite no evidence of increased Gd variability in the ER salt due to Li₂O and Gd₂O₃ carryover from the OR into the ER system, further similar work with uranium may be valuable and lead to a better understanding of the issues experienced previously in ER salt sampling. Additionally, exploring other potential reactions that can result in the formation of oxychlorides and/or oxides from the salt including oxygen and moisture contaminants in the argon atmosphere may be helpful in determining the source of the historical experience with Uranium concentration variability in the ER salt.

8. REFERENCES

1. D. Horvath, C. Heathman, and W. Phillips, "Oxychloride Formation in Molten Salts Used for Recycling Spent Nuclear Fuel – Literature Review and Thermodynamic Calculations," INL/LTC-21-63999, August 2021.

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Appendix A

ICP-OES Results

Hoover Salts	Li	K	Gd
Regression	0.99980	0.99950	0.99997
CCV mean %	105.6%	103.7%	97.3%
Detection Limit (ppm)	171.188	270.008	21.009
Margin of error	± 160	± 260	± 20
Sample ID	Li (mg/kg)	K (mg/kg)	Gd (mg/kg)
B1	81063.624	257242.042	32113.517
B2	82680.645	261671.022	32843.646
B3	86505.482	264497.108	33735.490
B4	91351.771	278813.643	36978.982
B5	87085.075	263881.996	35023.525
B6	85029.597	264187.396	46959.899
B7	77318.153	254306.212	44092.043
B8	82174.041	267199.858	46683.467
B9	81826.437	259979.799	44304.507
B10	84846.864	276048.944	48165.482
B11	77568.542	269120.203	38291.884
B12	87325.117	275946.089	35010.286
B13	87942.703	278536.365	36274.240
B14	81425.858	264485.707	35850.177
B15	85788.490	281229.399	39274.563
G1	87492.721	269573.501	54215.009
G2	78884.424	257726.747	54303.618
G3	81896.014	270434.504	56039.028
G4	84857.927	267183.254	54736.940
G5	75774.718	253180.320	53950.630
GO1	78402.544	258260.946	50974.423
GO2	76222.728	261184.166	50614.174
GO3	83292.752	269287.926	49568.141
GO4	78269.682	265491.118	50556.935
GO5	83896.195	276742.548	51115.472
GOL1	84552.190	269579.949	40036.547
GOL2	84347.020	275942.646	41456.735
GOL3	79211.389	266817.868	39961.147
GOL4	78162.375	258893.388	40069.811
GOL5	85086.508	278414.167	41338.445
L1	79682.267	247427.750	38350.821
L2	83504.755	258947.485	38709.730
L3	93861.693	278081.753	39923.686
L4	81376.704	259476.681	39536.088
L5	84917.133	268806.678	39714.295
LG1	81137.088	250803.906	36469.164
LG2	80176.328	256983.448	39555.546

LG3	85668.529	262914.575	37432.799
LG4	78010.572	253963.033	38213.610
LG5	82721.561	269627.895	40631.523
O1	76160.066	259082.707	47273.536
O2	82314.070	267840.985	43777.923
O3	81138.607	265708.348	44551.845
O4	85595.996	273649.118	44807.709
O5	80313.342	269921.305	45166.290
BLANK	84383.975	286232.696	0.000