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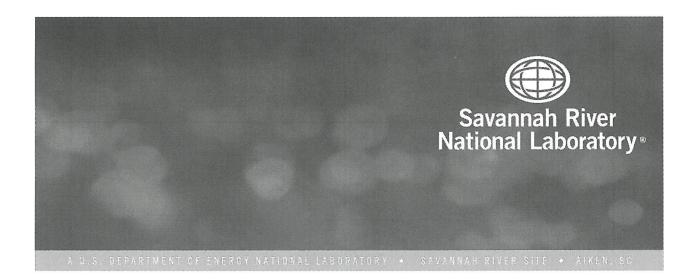
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# Demonstration of the Blending of the H-Canyon 2nd U Cycle Product with Natural U to Produce a HALEU Solution

T. S. Rudisill
W. E. Daniel
January 2021
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# Demonstration of the Blending of the H-Canyon 2nd U Cycle Product with Natural U to Produce a HALEU Solution

T. S. Rudisill W. E. Daniel

January 2021



# REVIEWS AND APPROVALS

Tracy S. Rudisill

Digitally signed by Tracy S. Rudisill Date: 2021.01.28 09:57:20 -05'00'

T. S. Rudisill, Actinide Materials and Separations Science and Technology

WILLIAM DANIEL (Affiliate) Digitally signed by WILLIAM DANIEL (Affiliate) Date: 2021.01.28 10:03:17 -05'00'

W. E. Daniel, Actinide Materials and Separations Science and Technology

TECHNICAL REVIEW:

Philip M. Almond

Digitally signed by Philip M. Almond Date: 2021.01.28 11:17:50 -05'00'

P. M. Almond, Nuclear Materials Systems Technology, Reviewed per E7 2.60

APPROVAL:

JONATHAN DUFFEY (Affiliate)

Digitally signed by JONATHAN DUFFEY (Affiliate)

Date: 2021.01.28 12:25:06 -05'00'

J. M. Duffey, Manager

Actinide Materials and Separations Science and Technology

Kristine Zeigler

Digitally signed by Kristine Zeigler

Date: 2021.01.28 13:27:19 -05'00'

K. E. Zeigler, Director

Actinide Materials and Separations Science and Technology

William F. Bates

Digitally signed by William F. Bates Date: 2021.01.28 14:52:14 -05'00'

W. F. Bates, Manager Nuclear Materials Management

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# **EXECUTIVE SUMMARY**

Continued growth in the demand for high assay low enriched uranium (HALEU) is projected to support the conversion of US high performance research reactors from high to low enrichment and the development and deployment of a new generation of advanced reactors. Current options to meet the growing US need for HALEU include (1) blend-down of excess highly enriched uranium (HEU) from the nuclear weapons program, (2) blend-down of HEU metal from other stockpiles, or (3) recycle of irradiated HEU recovered from spent nuclear fuel (SNF). The recycle of HEU from SNF has been performed at the Savannah River Site (SRS) H-Canyon facility for nearly two decades to produce 4.95 wt % U-235 for sale and subsequent use in fuel for Tennessee Valley Authority (TVA) reactors. The SRS H-Canyon facilities could also be used to produce HALEU

The initial high

concentration of U-235 in the fresh fuel results in the production of a significant amount of U-236 by neutron capture.

The HALEU produced in H-Canyon will not exceed the U-236 concentration limit in the ASTM C1462 standard for U metal enriched to more than 15% and less than 20% U-235, which applies to advanced reactors.

To validate that HALEU can be produced using the H-Canyon facilities, the Savannah River National Laboratory (SRNL) performed a demonstration of HALEU blending using the product solution from the 2<sup>nd</sup> U Cycle of solvent extraction. The objective of the demonstration was to show that requirements in the ASTM C1462 standard for U metal enriched between 15 and 20% could be met. Samples of uranyl nitrate solutions were obtained from tanks in the H-Canyon Outside Facilities used to store the 2<sup>nd</sup> U Cycle Product and the natural uranium (NU) which was used to blend-down the U-235 concentration in reprocessed MTR and other fuels to meet requirements for sale to the TVA. The two solutions were characterized for impurities and subsequently blended in proportions which targeted a solution containing 19.50 to

19.75 wt % U-235 with impurity concentrations which met the ASTM C1462 standard. The HALEU was blended based on the mass of the solution, U isotopic concentrations, and the measured specific gravity. Triplicate samples of the HALEU solution were analyzed to demonstrate that the U isotopic and impurity concentrations met the limitations in the C1462 standard.

A HALEU solution was successfully prepared using the 2<sup>nd</sup> U Cycle Product and NU obtained from H-Canyon targeting an approximate 30 g/L U solution containing nominally 19.5 wt % U-235. The composition of the solution met all isotopic and impurity requirements of the ASTM C1462 standard except for U-232. The measured U-232 concentration in the HALEU solution slightly exceeded the criterion (0.00229 µg/g U versus ≤0.002 µg/g U) in the ASTM C1462 standard by about 14%; however, with the uncertainty associated with the measured value, the concentration at a significant confidence limit (e.g., 95%) is indistinguishable from the U-232 criterion in the standard. The measured U-234, U-235, and U-236 concentrations in the solution were 0.3, 19.4, and 1.3 wt % with the balance being U-238. The U-236 concentration was well below the 4 wt % limit in the ASTM C1462 standard. These results demonstrate that the purified U in the H-Canyon Outside Facilities could be used to produce 2 to 2.4 metric tons of HALEU (if blended to 19.75 wt % U-235) which meets the requirements in the ASTM C1462 standard with the qualification noted for U-232.

The impurity concentrations measured in the HALEU solution were all less than the maximum values permitted by the ASTM C1462 standard. The transuranic activity in the solution which includes Np, Pu, Am, Cm, and Cf isotopes was 155 Bq/g U which is less than the criterion (250 Bq/g U) in the C1462 standard. The isotopes contributing to the total were Np-237 and Pu-238. All other isotopes were below the minimum detection of analysis (MDA). The total beta activity in the HALEU solution was minimal with

Sr-90 contributing 31 Bq/g U. The Tc-99 activity was less than the MDA. The gamma-emitting fission and activation products in the HALEU solution were also low as all isotopes detected were below the MDA. All trace metal concentrations were below limits in the ASTM standard based on analyses performed by the SRNL F/H Analytical Characterization Laboratory using an analysis method in which the U was initially removed to improve the accuracy.

The HALEU solution was also analyzed for a number of anionic species and total inorganic and organic carbon; although, the ASTM C1462 standard does not have limits for anionic impurities and the limit for carbon in the standard applies to carbon in the U metal. Both inorganic and organic carbon concentrations in the solution were below the MDA; however, the MDA values for both forms of carbon sum to a value which would exceed the limit in the standard. Most of the carbon present in the HALEU solution should be destroyed in the thermal process used to produce a U oxide and will not be present in the U metal. The concentrations of all anions except for nitrate were below the MDA, but the detection limits were greater than 3000  $\mu$ g/g U. The high detection limits can be attributed to the presence of a high concentration of U which required a significant dilution of the samples prior to analysis. The presence of anionic species in the HALEU is not expected to be a problem.

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# LIST OF ABBREVIATIONS

AD Analytical Development

F/H Lab F/H Analytical Characterization Laboratory

ICPES Inductively-coupled Plasma Emission Spectroscopy

ICPMS Inductively-coupled Plasma Mass Spectroscopy

GPHA Gamma Pulse Height Analysis

HALEU high assay low enriched uranium

HEU highly enriched uranium

HFIR High Flux Isotope Reactor

IC-Anion Ion Chromatography – Anions

LEU low enriched uranium

LIMS Laboratory Information Management System

MDA minimum detection of analysis

MTR material test reactor

MURR University of Missouri Research Reactor Center

N/A not applicable

NMC&A Nuclear Material Control and Accountability

NU natural uranium

PIPS passivated, implanted, planar silicon

SNF spent nuclear fuel

SRNL Savannah River National Laboratory

SRS Savannah River Site

TBR To be reported

TIC/TOC Total Inorganic Carbon/Total Organic Carbon

TRU transuranic

TVA Tennessee Valley Authority

US United States

#### 1.0 Introduction

The National Nuclear Security Administration's Office of Material Management and Minimization U.S. High Performance Research Reactor project is developing a low enriched uranium (LEU) fuel as a candidate replacement for the highly enriched uranium (HEU) fuels currently being used in the U.S. high performance reactors. The new high assay LEU (HALEU) fuel design is based on a monolithic U-Mo alloy with a U-235 enrichment of 19.75 wt %. <sup>1</sup> There is also a need for HALEU enriched to levels between 5 and 20 wt % to support the deployment of small modular and other advanced reactors. <sup>2</sup> The United States currently has the capability to enrich natural U (NU) that contains 0.71 wt % U-235 to concentrations approaching 5 wt % to support the fabrication of fuel for commercial nuclear reactors for the generation of electricity. <sup>3</sup> However, to address the growing need for HALEU the United States can (1) blend-down excess HEU from the nuclear weapons program, (2) blend-down HEU metal from other stockpiles, or (3) recycle irradiated HEU recovered from spent nuclear fuel (SNF).

The recycle of HEU from SNF has been performed at the Savannah River Site (SRS) H-Canyon facility for many years to produce a product solution which can be fabricated into fuels for commercial reactors.<sup>4</sup> Highly enriched U was recovered from material test reactor (MTR) fuels by dissolving the fuels and purifying the uranyl nitrate solution using the 1<sup>st</sup> and 2<sup>nd</sup> U Cycles of solvent extraction. The purified U solution was subsequently blended with NU to produce a final product solution with a U-235 concentration of nominally 4.95 wt % for sale and subsequent use in fuel for Tennessee Valley Authority (TVA) reactors.

The SRS H-Canyon facilities could also be used to produce HALEU. An approach was recently prepared outlining the steps necessary to transition from the production of 4.95 wt % LEU to a 19.75 wt % HALEU product. <sup>5</sup> An estimate was also prepared to convert previously separated HEU solution stored in the H-Canyon Outside Facilities to HALEU. <sup>6</sup> The purified U represents 2 to 2.4 metric tons of HALEU if blended to 19.75 wt % U-235. The complexity of a transition to HALEU production would be influenced by the specification selected which the HALEU must meet. There are two specifications currently in use for U enriched to nominally 19.75 wt % (i.e., HALEU). An ASTM International standard specification for U metal enriched to more than 15 wt % and less than 20 wt % is available for nuclear grade U metal that has either been processed through an enrichment plant, or has been produced by the blending of HEU with other sources of U to obtain blended U of any U-235 concentration between 15 and 20 wt %. <sup>7</sup>

**Table 1-1. Specifications for HALEU Metal** 

Element/Isotope	Units	ASTM C1462-00 Specification
U (metal)	wt %	≥ 99.85%
U-232	μg/g U	≤ 0.002
U-234	μg/g U	≤ 10000
U-235	wt %	15 < U-235 < 20
U-236	μg/g U	≤ 40000
TRU alpha activity <sup>1</sup>	Bq/g U	≤ 250
Total Beta <sup>2</sup>	Bq/g U	_
Activation Products <sup>3</sup>	Bq/g U	_
Fission Products <sup>4</sup>	Bq/g U	≤ 600.0
Al	μg/g U	≤ 150
As	μg/g U	
Be	μg/g U	≤ 10

Element/Isotope	Units	ASTM C1462-00 Specification
В	μg/g U	≤ 1
Ca	μg/g U	≤ 100
C	μg/g U	≤ 800
Cr	μg/g U	≤ 50
Со	μg/g U	≤ 10
Cu	μg/g U	≤ 50
Fe	μg/g U	≤ 250
Pb	μg/g U	≤ 10
Li	μg/g U	≤ 10
Mg	μg/g U	≤50
Mn	μg/g U	≤50
Mo	μg/g U	≤ 100
Ni	μg/g U	≤ 100
P	μg/g U	≤ 100
Si	μg/g U	≤ 250
Na	μg/g U	≤ 25
Sn	μg/g U	≤ 100
W	μg/g U	≤ 100
V	μg/g U	≤30
Zr	μg/g U	≤ 250
Total Impurities	μg/g U	≤ 1500
Equivalent B Content <sup>5</sup>	μg/g U	≤4

The initial high concentration of U-235 in the fresh fuel results in the production of a significant amount of U-236 by neutron capture. Table 1-2 shows the final isotopic concentrations for a typical High Flux Isotope Reactor (HFIR) core and typical assemblies

HFIR		MU	IRR
Isotope	Concentration	Isotope	Concentration
_	(wt %)	_	(wt %)
U-234	1.14	U-235	87.23
U-235	85.55	U-236	4.33
U-236	6.57	U-234+U-238	8.44
U-238	6.75	_	_

Table 1-2. U Isotopic Concentrations from Fuels Discharged from the HFIR and MURR

The concentration of U-236 in the HEU purified by the H-Canyon process can be easily diluted below the requirement in the ASTM C-1462 standard (≤40000 µg/g U) using NU.

The amount of

U required for dilution would depend on the enrichment and burnup of the MTR fuel and the U-235 enrichment of the supplemental HEU.

To validate that HALEU can be produced based on the strategy outlined above for the H-Canyon facility, the Savannah River National Laboratory (SRNL) performed a demonstration of HALEU production using the product solution from the 2<sup>nd</sup> U Cycle of solvent extraction. A sample of uranyl nitrate solution from the B3-1 storage tank was transferred to SRNL for use in the demonstration. The solution from the B3-1 tank best represents solution that has been processed through the 2<sup>nd</sup> U Cycle in the recent past. Natural U which was previously used to blend-down the U-235 concentration in reprocessed MTR and other fuels to meet the requirements for the TVA specification was also obtained from H-Canyon for use in the demonstration. The two solutions were characterized for impurities and subsequently blended in proportions which targeted a solution containing nominally 19.75% U-235 with impurity concentrations which met the ASTM C1462 standard for U metal enriched to more than 15 wt % and less than 20 wt % U-235 (Table 1-1). The characterization, blending, and analysis of the HALEU solution are described in the following sections of the report.

# 2.0 Experimental Procedure

### 2.1 Characterization of H-Canyon Solutions

Samples of the 2<sup>nd</sup> U Cycle product and NU previously used to blend-down the U-235 concentration in reprocessed MTR fuels for sale and subsequent use in fuel for TVA reactors were obtained from the H-Canyon facility. The solutions were shipped from H-Canyon to SRNL in 8 oz (237 mL) glass bottles (Figure 2-1). Once the solutions were received at SRNL, the multiple bottles were composited into high density polyethylene bottles to allow representative sampling of each solution. Multiple samples were taken to characterize the composition and impurities concentrations in the uranium solutions. The analyses performed are described below and the applicable analytes are identified.

 Inductively-coupled Plasma Emission Spectroscopy (ICPES) – Multi-element analysis method for metal impurities.



Uranyl Nitrate Solution from H-Canyon 2<sup>nd</sup> U Cycle



**NU Solution** 

Figure 2-1. H-Canyon U Solutions

- Inductively-coupled Plasma Mass Spectroscopy (ICPMS) Multi-element analysis method for metal impurities and U isotopic concentrations.
- Gamma Pulse Height Analysis (GPHA) Aliquots of the sample were analyzed by coaxial high purity germanium gamma-ray spectrophotometers to measure the gamma-emitting radionuclides present.
- Thenoyltrifluoroacetone (TTA) Extraction/Alpha Pulse Height Analysis (APHA) Aliquots of the sample were initially spiked with a <sup>236</sup>Pu tracer. The Pu was extracted from the matrix using TTA following a series of oxidation-state adjustments. The TTA extracts were mounted on stainless steel counting plates and counted for Pu-238 and Pu-239/240 using passivated, implanted, planar silicon (PIPS) alpha spectrometers. The separation efficiency was based on the <sup>236</sup>Pu recovery.
- Am/Cm/Cf Isotopes The Am, Cm and Cf isotopes were extracted from the sample matrix using a CMPO/TBP-based solid phase extractant and purified further with an HDEHP-based solid phase extractant. The Am-242, 242m, 243, Cm-243, 245, 247, and Cf-249, 251 concentrations were measured using low energy photon/X-ray, thin-windowed, semi-planar high purity germanium spectrometers. The Cm-242, 244 concentrations were measured using PIPS alpha spectrometers. The efficiency of the separations was determined from the Eu-154 measured by GPHA.

- U-232 The U isotopes were extracted from the sample matrix using Eichrom Technologies' UTEVA® resin (diamyl amylphosphonate absorbed on a polymer bead). The extracted U was mounted on stainless steel counting plates and counted. The U-232 to U-234 mass ratio was determined from the APHA. The U-232 concentration was subsequently calculated from the concentration of U-234 measured by ICPMS.
- Np-237 A known amount of Am-243 is initially added to a sample which is evaporated to dryness, reconstituted in HNO<sub>3</sub>, reduced, and extracted using Eichrom Technologies' TEVA® resin (Aliquat® 336 quaternary amine absorbed on a polymer bead). The extraction recovers the Np-237 and the Np-239 daughter of Am-243 (which is the chemical tracer). The Np-239 tracer is quantified by GPHA (and the Np-237 as well if it is above the GPHA detection limit). The sample is then analyzed by ICPMS to measure the Np-237 mass.
- Sr-90 Aliquots of the sample were initially spiked with natural Sr carrier. The Sr isotopes were extracted from the matrix using a crown ether-based solid phase extractant. The Sr-90 concentrations were measured by liquid scintillation counting. Elemental Sr carrier yields were measured for each aliquot by neutron activation analysis and were used to correct the Sr-90 value for any Sr losses incurred during the radiochemical separations.
- Tc-99 A Tc-99m tracer was generated in the SRNL neutron activation analysis facility by activating Mo-99 to produce Tc-99m by neutron irradiation. The Tc-99m was extracted from the Mo-99 target material with methyl isobutyl ketone. The Tc-99m tracer was added to aliquots of the samples. The Tc species were extracted from the sample using a solid extractant based on Aliquat-336™. The Tc-99 concentrations were measured by liquid scintillation counting. The Tc-99m yields were measured for each aliquot with a NaI well gamma spectrometer and were used to correct the Tc-99 values for any Tc losses incurred during the radiochemical separations.
- Ion Chromatography Anions (IC-Anions) The concentration of a suite of anions including fluoride, formate, chloride, nitrite, nitrate, phosphate, sulfate, oxalate, and bromide are measured in the sample using ion chromatography. The anions are separated into discrete bands in the column packed with anion exchange resin and the integrated concentration is measured using an electrical conductivity detector.
- Total Inorganic Carbon/Total Organic Carbon (TIC/TOC) The total carbon concentration in a sample is measured using a LECO carbon analyzer. The TOC concentration is measured by treating the sample with H<sub>3</sub>PO<sub>4</sub> to release the inorganic carbon and analyzing the sample using the carbon analyzer. The TIC carbon concentration is determined as the difference in the total carbon and the TOC concentrations.
- Free acid The free acid concentration of the solutions was measured by titrating a sample with base.

A summary of the analysis methods used to quantify the U and impurity concentrations is provided in Table 2-1.

Table 2-1. Summary of Analysis Methods to Quantify U and Impurity Concentrations

Element/Isotope	Analytical Method	Element/Isotope	Analytical Method
U-total	ICPES/ICPMS	Gd	ICPMS
U-232	U-232	Fe	ICPMS

Element/Isotope	Analytical Method	Element/Isotope	Analytical Method
U-234	ICPMS	Pb	ICPMS
U-235	ICPMS	Li	ICPMS
U-236	ICPMS	Mg	ICPES
TRU alpha activity	Pu TTA/APHA, Am/Cm/Cf	Mn	ICPMS
Total Beta	<sup>90</sup> Sr, <sup>99</sup> Tc	Mo	ICPMS
Activation Products	GPHA	Ni	ICPMS
Fission Products	GPHA	Nb	ICPMS
Al	ICPMS	N	N/A
As	ICPMS	P	ICPMS
Be	ICPMS	K	ICPMS
В	ICPMS	Sm	ICPMS
Cd	ICPMS	Si	ICPMS
Ca	ICPMS	Ag	ICPMS
С	TIC/TOC	Na	ICPMS
Cr	ICPMS	Sn	ICPMS
Со	ICPMS	W	ICPMS
Cu	ICPMS	V	ICPMS
Dy	ICPMS	Zn	ICPMS
Eu	ICPMS	Zr	ICPMS

## 2.2 HALEU Solution Blending

The blending of the 2<sup>nd</sup> U Cycle product and NU to produce the HALEU solution was based on calculations performed using U mass and the isotopic concentrations of U. The U isotopic concentrations were measured during the characterization of the 2<sup>nd</sup> U Cycle Product and the NU solutions. To estimate the mass of U in the NU solution required to blend-down the U-235 concentration in the 2<sup>nd</sup> U Cycle Product to 19.75% on a per gram of U basis, the mass of U in the NU solution was increased until the U-235 concentration from the two sources summed to the desired concentration. This process required the calculation of the mass of each U isotope in the two solutions which were then added together to calculate the U-235 concentration of the blended material. The calculation of the mass of NU required to obtain a blended product containing 19.75% U-235 is illustrated in the following two tables. The U isotopic concentration measured for the 2<sup>nd</sup>U Cycle Product and NU are provided in Table 2-2. These values were used in an iterative calculation to determine the mass of NU required to blend-down one gram of U in the 2<sup>nd</sup> U Cycle Product to achieve a U-235 concentration of 19.75%. The solution to the iterative calculation is shown in Table 2-3.

Table 2-2. U Isotopic Concentration for 2<sup>nd</sup> U Cycle Product and NU Solutions

2 <sup>nd</sup> U Cycl	2 <sup>nd</sup> U Cycle Product		U
Isotope	Concentration	Isotope	Concentration
_	(wt %)	_	(wt %)
U-234	0.81	U-234	0.00
U-235	65.09	U-235	0.70
U-236	4.53	U-236	0.00
U-238	29.57	U-238	99.30

Cycle	Product to Achie	ve a U-235 Conce	entration of 19.75	%
2 <sup>nd</sup> U Cycle (g)	1.0000	Isotope	Mass	Concentration
NU(g)	2.3798	_	(g)	(wt %)
Total U (g)	3.3798	U-234	0.0082	0.2426

Table 2-3. Calculation of the Mass of NU Required to Blend-down One Gram of U in the 2<sup>nd</sup> U

2 <sup>nd</sup> U Cycle (g)	1.0000	Isotope	Mass	Concentration
NU(g)	2.3798	_	(g)	(wt %)
Total U (g)	3.3798	U-234	0.0082	0.2426
_	_	U-235	0.6675	19.7500
_	_	U-236	0.0453	1.3409
_	_	U-237	2.6588	78.6665
_	_	U-total	3.3798	100.0000

To prepare the HALEU solution, the scale of the blending process was set at one and one-half grams of the 2<sup>nd</sup> U Cycle Product. The U concentration of the 2<sup>nd</sup> U Cycle product solution is nominally 10 grams per liter which provided about 150 mL of the H-Canyon product solution. With the addition of the NU solution, the total volume was more than sufficient to allow sampling for solution characterization. The amount of U in the NU solution required for blending was subsequently calculated from the ratio of the mass of U in the NU solution to the mass of the U in the 2<sup>nd</sup> U Cycle Product solution (Table 2-3). The amounts of the two solutions required to prepare the HALEU solution were based on mass. Initially, the required volumes of solution were determined from measured concentrations. Specific gravities of the solution were determined by measuring the mass of a known volume of solution multiple times. The specific gravities were then used to calculate the mass of each solution required to provide the desired amount of U. The blending was completed by combining the weighed amounts of the two solutions.

# 2.3 Characterization of HALEU Solution

The HALEU solution was characterized in the same manner as the 2<sup>nd</sup> U Cycle Product to demonstrate that the blended product met the criteria of the ASTM C1462 standard (Table 1-1).<sup>7</sup> The analysis methods used to characterize the solution for the U isotopic concentrations and the trace metal and radioactive impurities are discussed in Section 2.1 and summarized in Table 2-1.

### 2.4 Quality Assurance

Requirements for performing reviews of technical reports and the extent of the review are established in manual E7 2.60. A design verification report will be generated with a unique identification and transmitted to SRS Document Control, using form OSR 19-196 or equivalent. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Signature of the E7 technical reviewer on the final document signifies completion of the design verification review. Record keeping of the review on form WSRC-IM-2002-00011 and retention in an ELN is considered equivalent to OSR 19-196.

#### 3.0 Results and Discussion

### **3.1** Characterization of 2<sup>nd</sup> U Cycle Product

The 2<sup>nd</sup> U Cycle Product solution was characterized based on the requirements for U isotopic and impurity concentrations in the specifications for HALEU metal given in Table 1-1. The U isotopic concentrations for the 2<sup>nd</sup> U Cycle Product were calculated using the concentrations measured for the U-234, U-235, U-236, and U-238 isotopes by ICPMS provided in Table A-8 (Appendix A). The U isotopic composition of the solution is given in Table 3-1. The one sigma uncertainties in the concentrations were calculated using the standard deviation of the average of the three analyses and propagation of errors techniques. The isotopic concentrations were subsequently used as the basis for blending the HALEU solution. The measured concentrations of the U isotopes were also used as the basis for calculating the total concentration of U in the solution. The measured U concentration was  $9.72 \pm 0.06$  g/L.

Isotope	Concentration	Uncertainty
	(wt %)	(wt %)
U-234	0.81	0.01
U-235	65.09	0.68
U-236	4.53	0.04
U-238	29.57	0.38

Table 3-1. U Isotopic Composition of 2<sup>nd</sup> U Cycle Product

The impurity concentrations measured in the 2<sup>nd</sup> U Cycle product are summarized in Table 3-2 through Table 3-11 for the isotopic and elemental concentrations controlled by the specifications for HALEU metal. The data presented in the tables are the averages of the concentrations measured in the triplicate samples which were analyzed by the radiochemical and elemental analyses used to characterize the solution. The one sigma uncertainty in the concentrations was calculated using the standard deviation of the average of the three analyses and propagation of errors techniques. The objective of the HALEU blending demonstration was to meet the requirements of the ASTM C1462 standard for U metal enriched to more than 15% and less than 20% U-235, which is applicable to advanced reactors. To illustrate the purity of the 2<sup>nd</sup> U Cycle Product, the applicable criteria from the C1462 standard is also provided in the tables to allow a comparison of the results from the solution characterization.

The U-232 concentration measured in the  $2^{nd}$  U Cycle Product solution was calculated using the data provided in Table A-1. A U-232 half-life of 24.1 years was used to calculate a specific activity of 22.4 Ci/g. The U-232 concentration (in units of  $\mu$ g/g U) was subsequently calculated using the average U-232 concentration, the specific activity, and the concentration of the H-Canyon U product solution (9.72 ± 0.06 g/L). The results of the calculation are summarized in Table 3-2.

Table 3-2. U-232 Analyses for the 2<sup>nd</sup> U Cycle Product Solution

U-232	Uncertainty	<b>ASTM C1462</b>
(μg/g U)	(μg/g U)	(μg/g U)
0.0037	0.0020	≤0.0020

The Np-237 concentration measured in the  $2^{nd}$  U Cycle Product solution was calculated using the data provided in Table A-2. The concentration (in units of Bq/g U) was calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The results of the calculation are summarized in Table 3-3. The ASTM C1462 criterion given in the table is for the transuranic alpha activity which would include the sum of the Np, Pu, Am, Cm, and Cf activities in the  $2^{nd}$  U Cycle Product solution.

Table 3-3. Np-237 Analysis for the 2<sup>nd</sup> U Cycle Product Solution

Np-237	Uncertainty	ASTM C1462 TRU Activity
(Bq/g U)	(Bq/g U)	(Bq/g U)
13.9	0.8	≤250

The Pu isotopic concentrations measured in the  $2^{nd}$  U Cycle Product solution were calculated using the data provided in Table A-3. The concentrations of the Pu-239/240 isotopes were below the minimum detection of the analysis (MDA). The value reported is the average detection limit of the analysis for the three samples. The concentration of Pu-238 and the Pu-239/240 average detection limit (in units of Bq/g U) were calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The results of the calculation are summarized in Table 3-4. The ASTM C1462 criterion given in the table is for the transuranic

(TRU) alpha activity which would include the sum of the Np, Pu, Am, Cm, and Cf activities in the 2<sup>nd</sup> U Cycle Product solution.

Table 3-4. Pu Isotopic	Analyses for the 2 <sup>nd</sup> U	<b>Cycle Product Solution</b>
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Isotope	Concentration	Uncertainty	ASTM C1462 TRU Activity
_	(Bq/g U)	(Bq/g U)	(Bq/g U)
Pu-238	204	63	≤250
Pu-239/240	<4.12	MDA	_

The Am, Cm, and Cf isotopic concentrations measured in the  $2^{nd}$  U Cycle Product solution were calculated using the data provided in Table A-4. The concentrations of the isotopes were all below the MDA or upper limit values for the analyses. The values reported in Table 3-5 are the average detection/upper limits of the analysis for the three samples. The concentrations (in units of Bq/g U) were calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The ASTM C1462 criterion given in the table is for the TRU alpha activity which would include the sum of the Np, Pu, Am, Cm, and Cf activities in the  $2^{nd}$  U Cycle Product solution.

Table 3-5. Am, Cm, and Cf Isotopic Analyses for the 2<sup>nd</sup> U Cycle Product Solution

Isotope	Concentration	Uncertainty	ASTM C1462 TRU Activity
_	(Bq/g U)	(Bq/g U)	(Bq/g U)
Am-241	<21.7	MDA	≤250
Am-243	<7.6	MDA	_
Am-242m	<7.3	MDA	_
Cm-242	<6.0	MDA	_
Cm-243	<40.0	MDA	-
Cm-244	<3.5	MDA	_
Cm-245	<32.9	MDA	_
Cm-247	<40.0	MDA	_
Cf-249	<43.4	MDA	_
Cf-251	<40.4	MDA	_

The Sr-90 concentration measured in the  $2^{nd}$  U Cycle Product solution was calculated using the data provided in Table A-5. The concentration of the isotope was below the MDA. The value reported is the average detection limit of the analysis for the three samples. The concentration (in units of Bq/g U) was calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The results of the calculation are summarized in Table 3-6. The ASTM C1462 standard does not have a criterion for total beta activity which would include Sr-90.

Table 3-6. Sr-90 Analysis for the 2<sup>nd</sup> U Cycle Product Solution

Sr-90	Uncertainty
(Bq/g U)	(Bq/g U)
<107	MDA

The Tc-99 concentration measured in the 2<sup>nd</sup> U Cycle Product solution was calculated using the data provided in Table A-6. The concentration of the isotope was below the MDA. The value reported is the

average detection limit of the analysis for the three samples. The concentration (in units of Bq/g U) was calculated using the concentration of the H-Canyon U product solution ( $9.72 \pm 0.06$  g/L). The results of the calculation are summarized in Table 3-7. The ASTM C1462 standard does not have a criterion for total beta activity which would include Tc-99.

Table 3-7. Tc-99 Analysis for the 2<sup>nd</sup> U Cycle Product Solution

Tc-99	Uncertainty
(Bq/g U)	(Bq/g U)
<3.19	MDA

The concentrations of gamma-emitting isotopes measured in the  $2^{nd}$  U Cycle Product solution were calculated using the data provided in Table A-7. The concentrations of a number of the isotopes were below the MDA. The values reported are the average detection limit of the analysis for the three samples. The concentration of the isotopes which were quantified and the average detection limit for the isotopes with concentrations below the MDA (in units of Bq/g U) were calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The results of the calculations are summarized in Table 3-8. The ASTM C1462 criterion given in the table is for the total gamma activity. Although the limit is given as 600 Bq/g U, the standard notes that the limit may be as high as 6000 Bq/g U depending upon the requirements of the fuel fabricator. Gamma-emitting isotopes are not identified by the C1462 standard.

Table 3-8. Gamma-emitting Isotope Analyses for the 2<sup>nd</sup> U Cycle Product Solution

Isotope	Concentration	Uncertainty	ASTM C1462 Activity Limit
_	(Bq/g U)	(Bq/g U)	(Bq/g U)
<sup>58</sup> Co	<18.8	MDA	≤600 (6000)
<sup>60</sup> Co	<20.2	MDA	_
<sup>95</sup> Nb	<21.3	MDA	_
<sup>95</sup> Zr	<33.3	MDA	_
<sup>103</sup> Ru	<18.2	MDA	_
<sup>106</sup> Ru	<141	MDA	_
<sup>125</sup> Sb	<56.2	MDA	_
<sup>134</sup> Cs	<19.5	MDA	- ,
<sup>137</sup> Cs	<21.9	MDA	_
<sup>144</sup> Ce	<572	MDA	_
<sup>208</sup> Tl	382	8	_
<sup>212</sup> Bi	1209	23	_
<sup>212</sup> Pb	1168	87	_
<sup>224</sup> Ra	1063	49	-
<sup>234m</sup> Pa	4417	674	_
<sup>237</sup> Np/ <sup>233</sup> Pa	121	10	_
<sup>241</sup> Am	<582	MDA	_

The concentrations of trace metals measured in the 2<sup>nd</sup> U Cycle Product solution were calculated using the data provided in Table A-9. The concentration of a number of the elements were below the MDA. The values reported are the average detection limit of the analysis for the three samples. The concentration of

the elements which were quantified and the average detection limit for the elements with concentrations below the MDA (in units of  $\mu g/g$  U) were calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The results of the calculations are summarized in Table 3-9. The maximum concentrations allowed by the ASTM C1462 standard are provided in the table for most elements. For elements in the table for which a maximum concentration is not provided in the standard

The concentration of B given in Table 3-9 is likely biased high due to the use of glass sample vials and the subsequent leaching of B from the glass by the acidic solution.

Table 3-9. Trace Metal Analyses for the 2<sup>nd</sup> U Cycle Product Solution

Element	Concentration	Uncertainty	ASTM C1462 Limits
_	(μg/g U)	(μg/g U)	(μg/g U)
Li	0.621	0.05	≤10
Be	< 0.051	MDA	≤10
В	413	118	≤1
Na	< 0.051	MDA	≤25
Mg	19.4	11	≤50
Al	83.3	20	≤150
Si	804	94	≤250
P	278	6	≤100
Ca	65	4	≤100
V	0.070	0.025	≤30
Cr	9.22	0.14	≤50
Mn	2.14	0.51	≤50
Fe	283	7	≤250
Со	0.110	0.008	≤10
Ni	13.6	0.8	≤100
Cu	7.67	0.63	≤50
Zr	0.256	0.128	≤250
Mo	0.516	0.050	≤100
Sn	0.150	0.043	≤100
W	0.639	0.097	≤100
Pb	0.377	0.009	≤10

The concentrations of TIC/TOC measured in the  $2^{nd}$  U Cycle Product solution were calculated using the data provided in Table A-10. The concentration of both forms of carbon were below the MDA. The values reported are the average detection limit of the analysis for the three samples. The concentrations (in units of  $\mu$ g/g U) were calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The results of the calculation are summarized in Table 3-10. Although the MDA is much higher than the limit for total carbon in the ASTM C1462 standard, these values are for trace TIC/TOC in the U solution which should mostly be destroyed in the thermal process used to produce a U oxide. The limit given in the ASTM C1462 standard is also for trace carbon in U metal.

Carbon	Concentration	Uncertainty	ASTM C1462 Limit
_	(μg/g U)	(μg/g U)	(μg/g U)
Inorganic	<2057	MDA	≤800
Organic	<2057	MDA	_
Total	<4114	MDA	_

Table 3-10. Carbon Analyses for the 2<sup>nd</sup> U Cycle Product Solution

The concentrations of anions measured in the  $2^{nd}$  U Cycle Product solution were calculated using the data provided in Table A-11. The concentrations of all anions except for the nitrate associated with the U and trace metals and HNO<sub>3</sub> were below the MDA. The values reported for the anions with concentrations below the MDA are the average detection limit of the analysis for the three samples. The concentrations of the anions with reported values below the MDA (in units of  $\mu g/g$  U) were calculated using the concentration of the H-Canyon U product solution (9.72  $\pm$  0.06 g/L). The results of the calculation are summarized in Table 3-11. Neither the ASTM C1462 standard

has criteria for anions; although, anionic impurities would fall under the criterion for total impurities which is  $\leq 1500~\mu g/g~U$  for the ASTM standard unantities of some of the anionic impurities (e.g., formate, nitrite and oxalate) will be mostly destroyed or volatilized in the thermal process used to produce U oxide.

Anion	Concentration	Uncertainty
_	(μg/g U)	(μg/g U)
Fluoride	<103	MDA
Formate	<103	MDA
Chloride	<103	MDA
Nitrite	<103	MDA
Phosphate	<103	MDA
Sulfate	<103	MDA
Oxalate	<103	MDA
Bromide	<514	MDA

Table 3-11. Anion Analyses for the 2<sup>nd</sup> U Cycle Product Solution

The HNO<sub>3</sub> concentration of the  $2^{nd}$  U Cycle Product solution was measured as part of the characterization. The analyses for triplicate samples are provided in Table A-12. The average HNO<sub>3</sub> concentration for the H-Canyon product solution was  $0.611 \pm 0.014$  M.

## 3.2 Characterization of NU

The NU solution was analyzed for U isotopic concentrations and for impurity concentrations based on the requirements in the specifications for HALEU metal given in Table 1-1. In addition to the U analysis,

analyses were performed for Pu isotopes (to check for cross-contamination), trace metals, TIC/TOC, anions, and free acid. The U isotopic concentrations for the NU solution were calculated using the concentrations measured for the U-234, U-235, U-236, and U-238 isotopes by ICPMS provided in Table B-2 (Appendix B). The U isotopic composition of the solution is given in Table 3-12. The one sigma uncertainties in the concentrations were calculated using the standard deviation of the average of the three analyses and propagation of errors techniques. The isotopic concentrations were subsequently used for blending the HALEU solution. The measured concentrations of the U isotopes were also used as the basis for calculating the total concentration of U in the solution. The measured U concentration was  $496.8 \pm 0.8 \text{ g/L}$ .

Isotope	Concentration	Uncertainty
_	(wt %)	(wt %)
U-234	0.00	0.00
U-235	0.70	0.01
U-236	0.00	0.00
U-238	99.30	0.23

Table 3-12. U Isotopic Composition of NU

The impurity concentrations measured in the NU solution are summarized in Table 3-13 through Table 3-16 for the isotopic and elemental concentrations controlled by the specifications for HALEU metal. The data presented in the tables are the averages of the concentrations measured in the triplicate samples which were analyzed by the radiochemical and elemental analyses used to characterize the solution. The one sigma uncertainty in the concentrations was calculated using the standard deviation of the average of the three analyses and propagation of errors techniques. The objective of the HALEU blending demonstration was to meet the requirements of the ASTM C1462 standard for U metal enriched to more than 15% and less than 20% U-235, which is applicable to advanced reactors. To illustrate the purity of the NU solution, the applicable criteria from the C1462 standard are also provided in the tables to allow a comparison of the results from the solutioncharacterization.

The Pu isotopic concentrations measured in the NU solution were calculated using the data provided in Table B-1. The concentrations of the Pu-238 and Pu-239/240 isotopes were below the MDA. There was no evidence of TRU alpha contamination in the NU storage tank. The values reported are the average detection limits of the analyses for the three samples. The concentrations of the average detection limits (in units of Bq/g U) were calculated using the concentration of the NU solution (496.8  $\pm$  0.8 g/L). The results of the calculation are summarized in Table 3-13. The ASTM C1462 criterion given in the table is for the transuranic (TRU) alpha activity which would include the sum of the activities from all TRU elements in the solution.

Isotope	Concentration	Uncertainty	ASTM C1462 TRU Activity
_	(Bq/g U)	(Bq/g U)	(Bq/g U)
Pu-238	< 0.105	MDA	≤250
Pu-239/240	< 0.113	MDA	_

Table 3-13. Pu Isotopic Analyses for the NU Solution

The trace metal concentrations in the NU solution were analyzed by two ICPMS methods. The data in Table B-2 were analyzed by diluting the high concentration of U in the samples prior to analysis. Data reported in Table B-3 were analyzed after removal of the U from the samples using Eichrom Technologies' UTEVA® resin. For both analysis methods, the concentration of a number of the elements were below the MDA. The MDA's following U removal are much lower when compared to the analysis of the same element following dilution of the high U concentration. The MDA values reported are the average detection limit of the

analysis for the three samples. The concentration of the elements which were quantified and the average detection limit for the elements with concentrations below the MDA (in units of  $\mu g/g$  U) were calculated using the concentration of the NU solution (496.8  $\pm$  0.8 g/L). The results of the calculation for both analysis methods are summarized in Table 3-14. The maximum concentrations allowed by the ASTM C1462 standard are provided in the table for most elements.

the ASTM C1462 standard

have a criterion for total impurities. The

maximum concentrations are ≤1500 µg/g U for the ASTM standard

The concentration of B given in Table 3-14 (which is already a low value based on the high U concentration) is likely biased high due to the use of glass sample vials and the subsequent leaching of B from the glass by the acidic solution.

Table 3-14. Trace Metal Analyses for the NU Solution

Element	U Dilution IC	PMS Method	U Removal IC	CPMS Method	ASTM C1462
_	Conc	Uncert	Conc	Uncert	Limits
_	(μg/g U)	(μg/g U)	(μg/g U)	(μg/g U)	(μg/g U)
Li	_	-	< 0.004	MDA	≤10
Be	_	-	< 0.004	MDA	≤10
В	_	_	0.020	0.001	≤1
Na	_	_	0.545	0.021	≤25
Mg	_	_	0.029	0.002	≤50
Al	_	_	0.149	0.009	≤150
Si	_	-	0.133	0.009	≤250
P	_	-	0.332	0.020	≤100
Ca	_	_	< 0.322	MDA	≤100
V	_	_	0.0010	0.0001	≤30
Cr	_	_	0.171	0.001	≤50
Mn	_	_	0.0128	0.0004	≤50
Fe	_	_	0.843	0.026	≤250
Со	< 0.201	MDA	0.0016	0.0001	≤10
Ni	_	_	0.143	0.002	≤100
Cu	-	_	< 0.014	MDA	≤50
Zr	< 0.201	MDA	0.0045	0.0001	≤250
Mo	1.01	0.08	< 0.0245	0.0001	≤100
Sn	< 0.201	MDA	< 0.002	MDA	≤100
W	0.45	0.05	0.0070	0.0009	≤100
Pb	< 0.201	MDA	< 0.002	MDA	≤10

The concentrations of TIC/TOC measured in the NU solution were calculated using the data provided in Table B-4. The concentration of both forms of carbon were below the MDA. The values reported are the average detection limit of the analysis for the three samples. The concentrations (in units of  $\mu$ g/g U) were calculated using the concentration of the NU solution (496.8 ± 0.08 g/L). The results of the calculations are summarized in Table 3 15. Although the MDA is lower than the limit for total carbon in the ASTM C1462 standard, the trace amounts of TIC/TOC in the U solution should be mostly destroyed in the thermal process used to produce a U oxide. The limit given in the ASTM C1462 standard is also for trace carbon in U metal.

Carbon	Concentration	Uncertainty	ASTM C1462 Limit
_	(μg/g U)	(μg/g U)	(μg/g U)
Inorganic	<40	MDA	≤800
Organic	<40	MDA	_
Total	<81	MDA	_

The concentrations of anions measured in the NU solution were calculated using the data provided in Table

Table 3-15. Carbon Analyses for the NU Solution

B-5. The concentrations of all anions except for the nitrate associated with the U and trace metals and HNO<sub>3</sub> were below the MDA. The values reported for the anions with concentrations below the MDA are the average detection limit of the analysis for the three samples. The concentrations of the anions with reported values below the MDA (in units of  $\mu g/g$  U) were calculated using the concentrations of the NU solution (496.8 ± 0.08 g/L). The results of the calculation are summarized in Table 3-16. Neither the ASTM C1462 standard although, anionic impurities would fall under the criterion for total impurities which is  $\leq$ 1500  $\mu g/g$  U for the ASTM standard and in Table 3-16. The trace quantities of some of the anionic impurities (e.g., formate, nitrite and oxalate) will be mostly destroyed or volatilized in the thermal process used to produce U oxide.

Anion	Concentration	Uncertainty	ASTM C1462 Limit
_	(μg/g U)	(μg/g U)	(µg/g U)
Fluoride	<2.01	MDA	_
Formate	<2.01	MDA	_
Chloride	<2.01	MDA	<u> </u>
Nitrite	<2.01	MDA	_
Phosphate	<2.01	MDA	_
Sulfate	<2.01	MDA	_
Oxalate	<2.01	MDA	_
Bromide	<10.1	MDA	_

Table 3-16. Anion Analyses for the NU Solution

The HNO<sub>3</sub> concentration of the NU solution was measured as part of the characterization. The analyses for triplicate samples are provided in Table B-6. The average HNO<sub>3</sub> concentration for the H-Canyon product solution was  $0.321 \pm 0.034$  M.

#### 3.3 HALEU Blend for Advanced Reactors

To perform the HALEU blending demonstration, target masses for total U were selected to provide sufficient volume for the large number of analyses required to characterize the solution, but also to stay below the Nuclear Material Control and Accountability (NMC&A) reportable mass limit for enriched U

when preparing the samples. The target U total masses for the HALEU blend are shown in Table 3-17 along with the U-235 masses based on the isotopic concentrations measured for the 2<sup>nd</sup> U Cycle Product (Table 3-1) and NU (Table 3-12) solutions. Using the measured total U concentration for each solution (Table 3-17), the target volumes for the two solutions were calculated. Based on measured densities for each solution (Table 3-17), the target volumes were converted into target masses.

Target Target <sup>235</sup>U H-Canyon Measured **Target** Measured **Target Solution** Utotal Mass Mass  $\mathbf{U}_{\mathrm{total}}$ Volume **Density** Sol'n Mass (g/L)(mL) (g/mL)(g) (g) **(g)** 2<sup>nd</sup> U Cycle 1.5000 0.97632 9.72 154.28 1.0316 159.1514 NU 496.78 7.19 11.9912 3.5697 0.02495 1.6688 **Totals** 5.0697 1.00126 161.47 171.1426

Table 3-17. Target Masses for HALEU Blend 1

When performing the blend, the target masses could not be achieved exactly due to limitations in transferring the solution into the weighing vessel. The actual masses of each solution combined are shown in Table 3-18. Based on the calculations from the actual masses the target blend should be 19.74 wt % U-235.

Solution	Actual Mass	Actual Volume	Actual U <sub>total</sub> Mass	Actual <sup>235</sup> U Mass
_	(g)	(mL)	(g)	(g)
2 <sup>nd</sup> U Cycle	159.1546	154.29	1.5000	0.9763
NU	12.0041	7.19	3.5736	0.0250
Totals	171.1587	161.48	5.0736	1.0013

Table 3-18. Actual Masses for HALEU Blend 1

The U isotopic concentrations of the blended solution were measured prior to characterization. The U-235 concentration was only 19.00 wt % which was lower than expected. Therefore, the addition of an aliquot of the 2<sup>nd</sup> U Cycle Product was planned to increase the U-235 concentration to 19.75 wt %. For the new HALEU blend (Blend 1A), an estimated mass of 8.4456 g of the 2<sup>nd</sup> U Cycle Product solution was needed to adjust the U-235 concentration to 19.75 wt % (Table 3-19). When performing the blend, the target masses could not be achieved exactly due to limitations in transferring the solutions into the weighing vessel. The actual masses for the HALEU Blend 1A are shown in Table 3-20.

Table 3-19. Target Masses for HALEU Blend 1A

Solution	U <sub>total</sub> Mass	<sup>235</sup> U Mass	Measured U <sub>total</sub>	Volume	Measured Density	Target Sol'n Mass
_	(g)	(g)	(g/L)	(mL)	(g/mL)	(g)
Blend 1	4.8419	0.9202	30.6433	158.01	1.0436	164.8971
2 <sup>nd</sup> U Cycle*	0.0796	0.0518	9.72	8.19	1.0316	8.4456
Totals	4.9215	0.9720	_	166.1945	_	173.3427

<sup>\*</sup>Target values

Table 3-20. Actual Masses for HALEU Blend	Table 5-Zu. Acu	uai wiasses	IOF II	AL		siena	IA
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Solution	Actual Mass	Actual Volume	Actual U <sub>total</sub> Mass	Actual <sup>235</sup> U Mass
_	(g)	(mL)	(g)	(g)
Blend 1	164.8971	158.01	4.8419	0.9202
2 <sup>nd</sup> U Cycle	8.4410	8.18	0.0796	0.0518
Totals	173.3381	166.19	4.9214	0.9720

The U isotopic concentrations of the blended solution were again measured prior to characterization. The U-235 concentration was now 20.79 wt % which was higher than expected. Having undershot the U-235 concentration in Blend 1 and subsequently exceeded the target U-235 concentration in Blend 1A, a U-235 concentration of 19.5 wt % was selected for a second modified HALEU blend (Blend 1B) based on the addition of an aliquot of the NU solution. The U concentration of the NU solution was measured by both ICPES (501.3 g/L U) and ICPMS (496.8 g/L U) providing a range of concentrations. Two calculations were performed to estimate the amount of NU that was needed to dilute the U-235 concentration in Blend 1B to 19.5 wt % (Table 3-21 and Table 3-22). The amount of NU ranged from 1.1076 to 1.1178 g which was used as the target range for Blend 1B. The actual amount of NU added to Blend 1A was 1.1142 g (Table 3-23) which gives a predicted U-235 concentration of 19.5 wt %.

Table 3-21. Target Masses for HALEU Blend 1B based on ICPES Analysis of NU Solution

Solution	U <sub>total</sub> Mass	<sup>235</sup> U Mass	Measured U <sub>total</sub>	Volume	Measured Density	Target Sol'n Mass
_	(g)	(g)	(g/L)	(mL)	(g/mL)	(g)
Blend 1A	4.8606	1.0104	30.7979	157.82	1.0540	166.3462
NU*	0.3328	2.33E-03	501.33	0.66	1.6688	1.1076
Totals	5.1934	1.0127	_	158.48	-	167.4538

<sup>\*</sup>Target values

Table 3-22. Target Masses for HALEU Blend 1B based on ICPMS Analysis of NU Solution

Solution	U-total Mass	<sup>235</sup> U Mass	Measured U <sub>total</sub>	Volume	Measured Density	Target Sol'n Mass
_	(g)	(g)	(g/L)	(mL)	(g/mL)	(g)
Blend 1A	4.8606	1.0104	30.7979	157.82	1.0540	166.3462
NU*	0.3328	2.33E-03	496.78	0.67	1.6688	1.1178
Totals	5.1934	1.0127	_	158.49	_	167.4640

<sup>\*</sup>Target values

Table 3-23. HALEU Blend 1B Actual Masses

Solution	Actual Mass	Actual Volume	Actual U <sub>total</sub> Mass	Actual <sup>235</sup> U Mass
_	(g)	(mL)	(g)	(g)
Blend 1A	166.3462	157.82	4.8606	1.0104
NU	1.1142	0.67	0.3317	2.32E-3
Totals	167.4604	158.49	5.1923	1.0127

No attempt was made to measure the U isotopic concentrations of the HALEU Blend 1B prior to performing a complete characterization of the solution for the impurity concentrations which are specified in the ASTM C1462 standard due to time constraints. As part of the solution characterization, the U isotopic concentrations were measured by SRNL Analytical Development (AD) and the SRNL F/H Analytical Characterization Laboratory (F/II Lab). The U-235 concentration measured by the AD ICPMS method was 20.0 wt %; however, the U-235 isotopic concentration measured by the F/H Lab ICPMS method was 19.36 wt %. Both values are based on the average of the results from the analysis of three samples. The F/H Lab ICPMS method is approved for NMC&A isotopic measurements<sup>11</sup> and is considered a more accurate measurement and best represents the reported U-235 concentration. A complete characterization of the HALEU solution is provided in the following section.

# 3.4 Characterization of HALEU Solution

The final HALEU solution (Blend 1B) was characterized based on the requirements for U isotopic and impurity concentrations in the specifications for HALEU metal given in Table 1-1. A comparison of the analytical results to the ASTM C1462 standard for U metal enriched between 15 and 20%7 is the primary focus of this section;

The U isotopic concentrations for the solution from Table C-8 (Appendix C) are summarized in Table 3-24. The one sigma uncertainties in the concentrations were calculated from the triplicate analyses performed. The target U-235 concentration for the final HALEU blend (Blend 1B) was 19.5 wt % to provide assurance that the upper limit (i.e., 20 wt %) for the ASTM C1462 standard was not exceeded. Adjustments to the U-235 concentration of the HALEU blend could be made if desired. A more accurate measurement of the isotopic concentrations of the 2<sup>nd</sup> U Cycle Product and NU solution using the F/H Lab certified method would likely facilitate obtaining a more accurate blending of the U-235 concentration in the HALEU solution. The measured concentrations of the U isotopes (Table C-9) were used as the basis for calculating the total concentration of U in the solution. The measured U concentration was 30.8 ± 0.1 g/L.

Concentration Uncertainty **Isotope** (wt %) (wt %) U-234 0.278 0.001 U-235 19.364 0.005 U-236 1.284 0.002 **U-238** 79.075 0.006

Table 3-24. U Isotopic Composition of the HALEU Solution

The impurity concentrations measured in the HALEU solution are summarized in Table 3-25 through Table 3-34 for the isotopic and elemental concentrations controlled by the specifications for HALEU metal. The data presented in the tables are the averages of the concentrations measured in the triplicate samples which were analyzed by the radiochemical and elemental analyses used to characterize the solution. The one sigma uncertainty in the concentrations was calculated using the standard deviation of the average of the three analyses and propagation of errors techniques. The objective of the HALEU blending demonstration was to meet the requirements of the ASTM C1462 standard for U metal enriched to more than 15% and less than 20% U-235, which is applicable to advanced reactors. To illustrate the purity of the 2<sup>nd</sup> U Cycle Product, the applicable criteria from the C1462 standard is also provided in the tables to allow a comparison of the results from the solution characterization.

The U-232 concentration measured in the  $2^{nd}$  U Cycle Product solution was calculated using the data provided in Table C-1. A U-232 half-life of 24.1 years was used to calculate a specific activity of 22.4 Ci/g. The U-232 concentration (in units of  $\mu g/g$  U) was subsequently calculated using the average U-232

concentration, the specific activity, and the concentration of U in the HALEU solution (30.8  $\pm$  0.1 g/L). The results of the calculation are summarized in Table 3-25.

Table 3-25. U-232 Analyses for the HALEU Solution

U-232	Uncertainty	ASTM C1462
(μg/g U)	(μg/g U)	(μg/g U)
0.00229	0.00026	≤0.0020

The measured concentration of U-232 in the HALEU solution slightly exceeded (14%) the criterion in the ASTM C1462 standard. However, with the uncertainty associated with the measured value, the concentration at a significant confidence limit (e.g., 95%) is indistinguishable from the U-232 criterion in the standard. There is also an inconsistency with the U-232 concentration measured in the 2<sup>nd</sup> U Cycle Product compared to the value measured in the HALEU solution. The U concentration increased from 9.72 g/L in the 2<sup>nd</sup> U Cycle Product to 30.8 g/L in the HALEU solution, which is a factor of about 3.2. However, the U-232 concentration was only reduced by a factor of 1.6 in the HALEU solution. The NU, which contained no U-232, should have diluted the U-232 concentration below the criterion in the ASTM C1462 standard. In future HALEU blending studies, a more comprehensive analysis of the 2<sup>nd</sup> U Cycle Product solution for U-232 should be performed to determine if the previous analysis (Table 3-2) was biased low to understand why the U-232 concentration was not diluted below the limit in the ASTM standard.

The Np-237 concentration measured in the HALEU solution was calculated using the data provided in Table C-2. The concentration (in units of Bq/g U) was calculated using the concentration of U in the HALEU solution  $(30.8 \pm 0.1 \text{ g/L})$ . The results of the calculation are summarized in Table 3-26. The ASTM C1462 criterion given in the table is for the transuranic alpha activity which would include the sum of the Np, Pu, Am, Cm, and Cf activities in the HALEU solution. The total TRU activity in the HALEU solution is discussed below following the presentation of the data from the analysis of the Am, Cm, and Cf isotopic concentrations measured in the solution.

Table 3-26. Np-237 Analysis for the HALEU Solution

Np-237	Uncertainty	ASTM C1462 TRU Activity
( <b>Bq/g U</b> )	(Bq/g U)	( <b>Bq/g U</b> )
96	5	≤250

The Pu isotopic concentrations measured in the HALEU solution were calculated using the data provided in Table C-3. The concentrations of the Pu-239/240 isotopes were below the MDA. The value reported is the average detection limit of the analysis for the three samples. The concentration of Pu-238 and the Pu-239/240 average detection limit (in units of Bq/g U) were calculated using the concentration of the U in the HALEU solution (30.8  $\pm$  0.1 g/L). The results of the calculations are summarized in Table 3-27. The ASTM C1462 criterion given in the table is for the transuranic (TRU) alpha activity which would include the sum of the Np, Pu, Am, Cm, and Cf activities in the HALEU solution.

Table 3-27. Pu Isotopic Analyses for the HALEU Solution

Isotope	2 <sup>nd</sup> U Cycle	Uncertainty	ASTM C1462 TRU Activity
_	(Bq/g U)	(Bq/g U)	(Bq/g U)
Pu-238	59	3	≤250
Pu-239/240	<2	MDA	_

The Am, Cm, and Cf isotopic concentrations measured in the HALEU solution were calculated using the data provided in Table C-4. The concentrations of the isotopes were all below the MDA. The values reported in Table 3-28 are the average detection limits of the analysis for the three samples. The concentrations (in units of Bq/g U) were calculated using the U concentration of the HALEU solution (30.8  $\pm$  0.1 g/L). The ASTM C1462 criterion given in the table is for the TRU alpha activity which would include the sum of the Np, Pu, Am, Cm, and Cf activities in the HALEU solution.

Isotope	2 <sup>nd</sup> U Cycle	Uncertainty	ASTM C1462 TRU Activity
_	(Bq/g U)	(Bq/g U)	(Bq/g U)
Am-241	<1.8	MDA	≤250
Am-243	<1.1	MDA	_
Am-242m	<0.6	MDA	_
Cm-242	<3.7	MDA	_
Cm-243	<3.0	MDA	_
Cm-244	<3.6	MDA	_
Cm-245	<4.0	MDA	_
Cm-247	<3.6	MDA	_
Cf-249	<0.5	MDA	_
Cf-251	<0.5	MDA	_

Table 3-28. Am, Cm, and Cf Isotopic Analyses for the HALEU Solution

The total TRU alpha activity measured in the HALEU solution was 155 Bq/g U which is less than the criterion (250 Bq/g U) in the ASTM C1462 standard for U enriched between 15 and 20%. Even if the Pu-239/240 and Am/Cm/Cf isotopes were present at the MDA, the TRU alpha activity would still be less than the ASTM limit. There appears to be an inconsistency with the TRU alpha activity measured in the 2<sup>nd</sup>U Cycle Product compared to the value measured in the HALEU solution. The U concentration increased from 9.72 g/L in the 2<sup>nd</sup> U Cycle Product to 30.8 g/L in the HALEU solution, which is a factor of about 3.2. However, the TRU alpha activity was only reduced by a factor of about 0.7 in the HALEU solution. The NU, which contained no TRU alpha activity, should have diluted the activity well below 100 Bq/g U

The inconsistent results could be explained by a low level of Pu cross contamination during the preparation of the samples for analysis; however, the measured Pu-238 and Pu-239/240 activities in the NU samples were below the MDA, except for the results from one of the Pu-239/240 analyses (see Table B-1) which was judged as insignificant compared to the MDA of theother samples.

The Sr-90 concentration measured in the HALEU solution was calculated using the data provided in Table C-5. The concentration (in units of Bq/g U) was calculated using the U concentration in the HALEU solution  $(30.8 \pm 0.1 \text{ g/L})$ . The results of the calculation are summarized in Table 3-29. The ASTM C1462 standard does not have a criterion for total beta activity which would include Sr-90 and Tc-99.

Sr-90 Uncertainty
(Bq/g U) (Bq/g U)

Table 3-29. Sr-90 Analysis for the HALEU Solution

The Tc-99 concentration measured in the HALEU solution was calculated using the data provided in Table C-6. The concentration of the isotope was below the MDA. The value reported is the average detection limit of the analysis for the three samples. The concentration (in units of Bq/g U) was calculated using the U concentration in the HALEU solution ( $30.8 \pm 0.1 \text{ g/L}$ ). The results of the calculation are summarized in Table 3-30. As noted above, the ASTM C1462 standard does not have a criterion for total beta activity which would include Tc-99.

Table 3-30. Tc-99 Analysis for the HALEU Solution

Tc-99	Uncertainty
(Bq/g U)	(Bq/g U)
<9	MDA

The concentrations of gamma-emitting isotopes measured in the HALEU solution were calculated using the data provided in Table C-7. The concentrations of a number of the isotopes were below the MDA. The values reported are the average detection limit of the analysis for the three samples. The concentration of the isotopes which were quantified and the average detection limit for the isotopes with concentrations below the MDA (in units of Bq/g U) were calculated using the U concentration in the HALEU solution  $(30.8 \pm 0.1 \text{ g/L})$ . The results of the calculations are summarized in Table 3-31. The ASTM C1462 criterion given in the table is for the total gamma activity. Although the limit is given as 600 Bq/g U, the standard notes that the limit may be as high as 6000 Bq/g U depending upon the requirements of the fuel fabricator. Gamma-emitting isotopes are not identified by the C1462 standard.

Table 3-31. Gamma-emitting Isotope Analyses for the HALEU Solution

Isotope	HALEU	Uncertainty	ASTM C1462 Activity Limit
_	(Bq/g U)	(Bq/g U)	(Bq/g U)
<sup>58</sup> Co	<4.4	MDA	≤600 (6000)
<sup>95</sup> Nb	<4.0	MDA	_
<sup>95</sup> Zr	<8.3	MDA	_
<sup>103</sup> Ru	<4.4	MDA	_
<sup>106</sup> Ru	<32	MDA	_
<sup>134</sup> Cs	<4.5	MDA	_
<sup>137</sup> Cs	<4.4	MDA	_
<sup>144</sup> Ce	<119	MDA	_
<sup>208</sup> Tl	155	1	_
<sup>212</sup> Bi	509	8	_
<sup>212</sup> Pb	555	125	_
<sup>237</sup> Np/ <sup>233</sup> Pa	87	1	_
<sup>234m</sup> Pa/ <sup>234</sup> Th/ <sup>238</sup> U	10969	299	_
<sup>241</sup> Am	<111	MDA	_

The gamma-emitting fission and activation product activities in the HALEU solution are low. The isotopes detected include Co-58, Nb-95, Zr-95, Ru-103, Ru-106, Cs-134, Cs-137, and Ce-144. The activity of all these isotopes were below the MDA. Even if the activity of each isotope was at the MDA, the total activity

would be less than 181 Bq/g U compared to a limit of less than or equal to 600 Bq/g U in the ASTM C1462 standard. The other gamma-emitting isotopes detected by the analysis are not fission or activation products. Thallium-208 is a decay product from U-232. Bismuth-212 and Pb-212 are daughter products from the decay of U-238. Protactium-234m and Th-234 are short-lived isotopes in secular equilibrium with U-238. Neptunium 237 and Am 241 are more accurately quantified by the specific methods used to characterize the HALEU solution.

The concentrations of trace metals in the HALEU solution were measured using ICPMS methods by SRNL AD and SRNL F/H Lab. In the ICPMS method utilized by AD, the high concentration of U was diluted prior to analysis. In the F/H Lab method, the U was removed using Eichrom Technologies' UTEVA® resin prior to the analysis. The concentrations of trace metals measured in the HALEU solution were calculated using the data provided in Table C-9 for the AD analyses and in Table C-10 for the F/H Lab analyses. The concentration of a number of the elements were below the MDA. The values reported are the average detection limit of the analysis for the three samples. The concentration of the elements which were quantified and the average detection limit for the elements with concentrations below the MDA (in units of  $\mu g/g$  U) were calculated using the U concentration of the HALEU solution (30.8  $\pm$  0.1 g/L). The results of the calculations are summarized in Table 3-32. The maximum concentrations allowed by the ASTM C1462 standard are provided in the table for most elements.

the ASTM C1462 standard and the

have a criterion for total impurities. The

maximum concentrations are ≤1500 μg/g U for the ASTM standard

Table 3-32. Trace Metal Analyses for the HALEU Solution

Element	U Dilution IC	PMS Method	U Removal IO	CPMS Method	<b>ASTM C1462</b>
_	Conc	Uncert	Conc	Uncert	Limits
_	(μg/g U)	(μg/g U)	(μg/g U)	(μg/g U)	(μg/g U)
Li	<0.1	MDA	< 0.06	MDA	≤10
Be	<0.1	MDA	< 0.03	MDA	≤10
В	0.6	0.1	< 0.26	MDA	≤1
Na	64	1	6.7	0.7	≤25
Mg	3.4	0.2	1.4	0.3	≤50
Al	2.7	0.2	2.3	0.7	≤150
Si	178	1	< 0.97	MDA	≤250
P	123	5	45.8	2.8	≤100
Ca	< 0.3	MDA	6.6	3.4	≤100
V	<0.1	MDA	< 0.02	MDA	≤30
Cr	7.8	0.2	0.540	0.002	≤50
Mn	0.98	0.02	< 0.05	MDA	≤50
Fe	159	43	9.6	0.6	≤250
Co	0.08	0.01	< 0.02	MDA	≤10
Ni	8.1	0.1	0.69	0.02	≤100
Cu	2.7	0.1	0.3	0.1	≤50
Zr	5.3	3.9	< 0.02	MDA	≤250
Mo	21	6	0.09	0.08	≤100

Element	U Dilution IC	CPMS Method	U Removal ICPMS Method		ASTM C1462
_	Conc	Uncert	Conc	Uncert	Limits
_	(μg/g U)	(μg/g U)	(μg/g U)	(μg/g U)	(μg/g U)
Sn	<0.1	MDA	< 0.02	MDA	≤100
W	0.250	0.001	< 0.03	MDA	≤100
Pb	0.18	0.02	< 0.03	MDA	≤10

The concentrations of all trace metals which were quantified in the HALEU solution are well below the limits in both the ASTM C1462 standard for U enriched between 15 and 20%

The total impurity concentrations based on the AD and F/H Lab analyses were 577 and 74  $\mu$ g/g U, respectively. The concentrations measured by F/H Lab using the ICPMS method in which the U was removed prior to analysis are more accurate due to the elimination of any signal attenuation by the presence of U in the solution.

The concentrations of TIC/TOC measured in the HALEU solution were calculated using the data provided in Table C-11. The concentration of both forms of carbon were below the MDA. The values reported are the average detection limit of the analysis for the three samples. The concentrations (in units of  $\mu g/g$  U) were calculated using the U concentration of the HALEU solution (30.8  $\pm$  0.1 g/L). The results of the calculations are summarized in Table 3-33.

Table 3-33. Carbon Analyses for the HALEU Solution

Carbon	Concentration	Uncertainty	ASTM C1462 Limit
_	(μg/g U)	(μg/g U)	(μg/g U)
Inorganic	<650	MDA	≤800
Organic	<650	MDA	_
Total	<1299	MDA	_

Although the MDA's for inorganic and organic carbon are below the total carbon in the ASTM C1462 standard, the sum of the two forms would exceed the limit in the standard if both forms of carbon were present at their maximum limit. However, the trace TIC/TOC in the U solution should mostly be destroyed in the thermal process used to produce a U oxide. The limit given in the ASTM C1462 standard is also for trace carbon in U metal.

The concentrations of anions measured in the HALEU solution were calculated using the data provided in Table C-12. The concentrations of all anions, except for the nitrate associated with the U and trace metals and HNO<sub>3</sub>, were below the MDA. The values reported for the anions with concentrations below the MDA are the average detection limit of the analysis for the three samples. The concentrations of the anions with reported values below the MDA (in units of  $\mu g/g$  U) were calculated using the U concentration in the HALEU solution (30.8  $\pm$  0.1 g/L). The results of the calculation are summarized in Table 3-34.

for anions; although, anionic impurities would fall under the criterion for total impurities which is

 $\leq$ 1500 µg/g U for the ASTM standard The trace quantities of some of the anionic impurities (e.g., formate, nitrite and oxalate) will be mostly destroyed or volatilized in the thermal process used to produce U oxide.

Anion	Concentration	Uncertainty
_	(μg/g U)	(μg/g U)
Fluoride	<3248	MDA
Formate	<3248	MDA
Chloride	<3248	MDA
Nitrite	<3248	MDA
Phosphate	<3248	MDA
Sulfate	<3248	MDA
Oxalate	<3248	MDA
Bromide	<3248	MDA

Table 3-34. Anion Analyses for the HALEU Solution

The HNO<sub>3</sub> concentration of the HALEU solution was measured as part of the characterization. The analyses for triplicate samples are provided in Table C-13. The average HNO<sub>3</sub> concentration for the H-Canyon product solution was  $0.56 \pm 0.02$  M.

#### 4.0 Conclusions

High assay low enriched U was prepared by blending solutions obtained from the H-Canyon Outside Facilities storage tanks for the  $2^{nd}$  U Cycle Product and NU. The HALEU blend targeted an approximate 30 g/L U solution containing nominally 19.5 wt % U-235. The composition of the solution met all isotopic and impurity requirements of the ASTM C1462 standard for U enriched between 15 and 20 wt %, which is applicable to advanced reactors, except for U-232. The measured U-232 concentration in the HALEU solution slightly exceeded the criterion (0.00229  $\mu$ g/g U versus  $\leq$ 0.002  $\mu$ g/g U) in the ASTM C1462 standard by about 14%; however, with the uncertainty associated with the measured value, the concentration at a significant confidence limit (e.g., 95%) is indistinguishable from the U-232 criterion in the standard. The measured U-234, U-235, and U-236 concentrations in the solution were 0.3, 19.4, and

1.3 wt % with the balance being U-238. The U-236 concentration was well below the 4 wt % limit in the ASTM C1462 standard. These results demonstrate that the purified U in the H-Canyon Outside Facilities could be used to produce 2 to 2.4 metric tons of HALEU (if blended to 19.75 wt % U-235) which meets the requirements in the ASTM C1462 standard with the qualification noted for U-232.

The impurity concentrations measured in the HALEU solution were all less than the maximum values permitted by the ASTM C1462 standard. The TRU activity in the solution which includes Np, Pu, Am, Cm, and Cf isotopes was 155 Bq/g U which is less than the criterion (≤250 Bq/g U) in the C1462 standard. The isotopes contributing to the total were Np-237 and Pu-238. All other isotopes were below the MDA. The total beta activity in the HALEU solution was minimal with Sr-90 contributing 31 Bq/g U. The Tc-99 activity was less than the MDA. The gamma-emitting fission and activation products in the HALEU solution were also low as all isotopes detected were below the MDA. All trace metal concentrations were below limits in the ASTM C1462 standard. The concentrations measured by F/H Lab by ICPMS following the removal of U were more accurate due to the elimination of any signal attenuation by the presence of high concentrations of U in the solution.

The HALEU solution was also analyzed for a number of anionic species and TIC/TOC; although, the ASTM C1462 standard does not have limits for anionic impurities and the limit for carbon in the standard applies to carbon in the U metal. Both inorganic and organic carbon concentrations in the solution were

below the MDA; however, the MDA values for both forms of carbon sum to a value which would exceed the limit in the standard. Most of the carbon present in the HALEU solution should be destroyed in the thermal process used to produce a U oxide and will not be present in the U metal. The concentrations of all anions except for nitrate were below the MDA, but the detection limits were greater than 3000  $\mu$ g/g U. The high detection limits can be attributed to the presence of a high concentration of U which required a significant dilution of the samples prior to analysis. The presence of anionic species in the HALEU is not expected to be a problem.

#### 5.0 Future Work

Inconsistencies in the analytical data were observed when the 2<sup>nd</sup> U Cycle Product and HALEU blend analyses for U-232 and TRU alpha activity were compared. The U concentration increased from 9.72 g/L in the 2<sup>nd</sup> U Cycle Product to 30.8 g/L in the HALEU solution, which is a factor of about 3.2. However, the reduction in both the U-232 and TRU alpha activity was not consistent with the dilution factors obtained for these species by adding the NU to the 2<sup>nd</sup> U Cycle Product. The NU contains no U-232 or TRU alpha activity. In future HALEU blending studies, a more comprehensive analysis of the 2<sup>nd</sup> U Cycle Product solution for U-232 and TRU alpha activity should be performed to understand why these isotopes were not diluted as expected; although, the measured TRU activity was less than required by the ASTM C1462 standard and the U-232 concentration was only slightly above the standard requirement.

Additional work could also be performed to demonstrate other HALEU blending scenarios. The future demand for HALEU will likely span a range of U-235 concentrations from greater than 5 to 19.75 wt %. Laboratory-scale demonstrations could be completed to validate that the blended materials can be produced and are compliant with specifications for U supplied to advanced reactors.

#### 6.0 References

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# Appendix A. Analytical Measurements for 2<sup>nd</sup> U Cycle Product Solution

The triplet analyses for each analytical method performed to characterize the 2<sup>nd</sup> U Cycle Product solution are summarized below. The uncertainties in the individual samples are provided as a 1 sigma relative standard deviation based on the results from the Analytical Development (AD) analysis method. The uncertainty in the average values was calculated from the three analyses.

#### U-232

The U-232 concentrations measured in the 2<sup>nd</sup> U Cycle Product Solution are summarized in Table A-1. The sample identification and AD Laboratory Information Management System (LIMS) number are provided for each sample.

Table A-1. U-232 Analyses for the 2<sup>nd</sup> U Cycle Product Solution

B31_U	J-232_1	B31_U-232_2		B31_U-232_3		B31_U-232	
LW	18133	LW18134		LW18135		Average	Std Dev
(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)
1.79E+03	20	8.27E+02	20	2.72E+03	20	1.78E+03	9.47E+02

### Np-237

The Np-237 concentrations measured in the 2<sup>nd</sup> U Cycle Product Solution are summarized in Table A-2. The sample identification and AD LIMS number are provided for each sample.

Table A-2. Np-237 Analyses for the 2<sup>nd</sup> U Cycle Product Solution

B31_	_Np_1	B31_Np_2		B31_	_Np_3	B31_Np	
LW	18148	LW	18149	LW	18150	Average	Std Dev
(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)
7.58E+00	20	8.26E+00	20	8.42E+00	20	8.09E+00	4.46E-01

## Pu TTA/APHA

The Pu isotopic concentrations measured by the Pu TTA/APHA method in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-3. The sample identification and AD LIMS number are provided for each sample. An average concentration was not calculated for the Pu-239/240 analyses since all values were below the MDA.

Table A-3. Pu Isotopic Analyses for the 2<sup>nd</sup> U Cycle Product Solution

Isotope	B31_TTA/	APHA_1	B31_TTA/APHA_2		B31_TTA/APHA_3		B31_TTA/APHA	
_	LW18	3136	LW18137		LW18138		Ave	Std Dev
_	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)
<sup>238</sup> Pu	8.97E+01	11.4	1.60E+02	8.22	1.07E+02	9.09	1.19E+02	3.66E+01
<sup>239/240</sup> Pu	<2.99E00	MDA	<2.22E00	MDA	<1.99	MDA	MDA	MDA

### Am/Cm/Cf Isotopes

The Am, Cm, and Cf isotopic concentrations measured by the Am/Cm/Cf method in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-4. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated since all analyses were either below the MDA or were provided as an upper limit on the concentration.

B31\_Am/Cm\_2 B31 Am/Cm 1 B31\_Am/Cm\_3 Isotope LW18139 LW18140 LW18141 (dpm/mL) (%) (dpm/mL) (%) (dpm/mL) (%) Am-241 <1.37E01 upper limit <1.21E01 upper limit <1.22E01 upper limit <2.38E00 Am-243 MDA <7.37E00 **MDA** <3.53E00 **MDA** Am-242m <2.43E00 **MDA** <5.76E00 upper limit <4.56E00 upper limit Cm-242 <2.01E00 **MDA** <4.76E00 upper limit <3.77E00 upper limit Cm-243 <2.56E01 **MDA** <2.31E01 **MDA** <2.13E01 **MDA** Cm-244 <2.43E00 <1.87E00 **MDA** <1.89E00 **MDA** upper limit Cm-245 <2.10E01 **MDA** <1.90E01 **MDA** <1.75E01 **MDA** Cm-247 <2.45E01 <2.42E01 <2.12E01 **MDA MDA MDA** Cf-249 <2.76E01 MDA <2.54E01 **MDA** <2.29E01 **MDA** Cf-251 <2.63E01 <2.26E01 <2.18E01 MDA MDA MDA

Table A-4. Am, Cm, and Cf Isotopic Analyses for the 2<sup>nd</sup> U Cycle Product Solution

#### Sr-90

The Sr-90 concentrations measured in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-5. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated since all analyses were below the minimum detection of the analysis.

B31 Sr-90 1 B31 Sr-90 2 B31 Sr-90 3 LW18142 LW18143 LW18144 dpm/mL (%) dpm/mL (%) dpm/mL (%) <6.73E01 **MDA** <5.58E01 MDA <6.44E01 **MDA** 

Table A-5. Sr-90 Analyses for the 2<sup>nd</sup> U Cycle Product Solution

#### Tc-99

The Tc-99 concentrations measured in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-6. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated since all analyses were below the MDA.

Table A-6. Tc-99 Analyses for the 2<sup>nd</sup> U Cycle Product Solution

B31_T	c-99_1	B31_T	c-99_2	B31_Tc-99_3		
LW18145		LW1	18146	LW18147		
dpm/mL	(%)	dpm/mL	(%)	dpm/mL	(%)	
<2.12E00	MDA	<1.75E00	MDA	<1.72E00	MDA	

#### **GPHA**

The concentrations of gamma-emitting isotopes measured by the GPHA method in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-7. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses which were below the MDA.

**B31\_GPHA\_2** Isotope B31\_GPHA\_1 B31 GPHA 3 **B31 GPHA** LW18130 LW18131 LW18132 Std Dev Ave (dpm/mL) (dpm/mL) (dpm/mL) (%) (%) (dpm/mL) (dpm/mL) (%) <sup>58</sup>Co <1.08E01 **MDA** <1.05E01 **MDA** <1.16E01 **MDA MDA MDA** <sup>60</sup>Co <1.15E01 MDA <1.20E01 MDA <1.18E01 MDA MDA MDA 95Nb <1.25E01 **MDA** <1.24E01 MDA <8.67E01 **MDA** MDA MDA <sup>95</sup>Zr <1.90E01 **MDA** <1.95E01 **MDA** <1.98E01 **MDA MDA MDA** <sup>103</sup>Ru <1.08E01 **MDA** <1.07E01 **MDA** <1.03E01 MDA MDA MDA <sup>106</sup>Ru <8.17E01 MDA <8.23E01 MDA <8.23E01 **MDA MDA MDA** 125Sb <3.29E01 **MDA** <3.23E01 MDA <3.31E01 **MDA** MDA MDA <sup>134</sup>Cs <1.13E01 MDA <1.15E01 MDA <1.14E01 MDA MDA **MDA** <sup>137</sup>Cs <1.33E01 **MDA** <8.81E01 MDA <1.22E01 MDA **MDA MDA** <sup>144</sup>Ce <3.31E02 **MDA** <3.35E02 MDA <3.35E02 MDA **MDA MDA** <sup>208</sup>Tl 2.18E+02 5.00 2.27E+02 5.00 2.23E+02 5.00 2.23E+02 4.51E+00 <sup>212</sup>Bi 7.11E+02 8.11 6.91E+02 7.76 7.14E+02 7.29 7.05E+021.25E+01 <sup>212</sup>Pb 7.35E+02 5.00 6.34E+02 5.00 5.00 6.75E+02 6.81E+02 5.08E+01 <sup>224</sup>Ra 6.49E+02 8.79 5.92E+029.40 6.19E+02 9.01 6.20E+02 2.85E+01 <sup>234m</sup>Pa 2.87E+03 14.8 2.73E+03 14.6 2.13E+03 20.0 2.58E+03 3.93E+02 235TJ 3.02E+04 5.00 3.06E+04 5.00 3.15E+04 5.00 3.08E+04 6.66E+02<sup>237</sup>Np/<sup>233</sup>Pa 7.14E+01 10.3 6.39E+01 7.58E+01 10.4 7.04E+01 6.02E+00 11.0

Table A-7. Gamma-emitting Isotope Analyses for the 2<sup>nd</sup> U Cycle Product Solution

### **ICPMS**

<sup>241</sup>Am

<2.91E2

**MDA** 

<3.04E2

The concentrations of the U isotopes and trace metals measured by the ICPMS method in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-8 and Table A-9, respectively. The concentrations provided in Table A-8 are for the U-234, U-235, U-236, and U-238 isotopes which were subsequently used to calculate the U isotopic composition of the 2<sup>nd</sup> U Cycle Product solution. The trace metal analyses provided in Table A-9 are the elements for which maximum concentrations are provided in the ASTM C1462 standard for U metal.<sup>7</sup> The sample identification and AD LIMS number are provided for the samples in each table. Average concentrations were not calculated for the analyses which were below the MDA.

MDA

<4.23E2

**MDA** 

MDA

 Table A-8. U Isotopic Concentrations Analyses for the 2<sup>nd</sup> U Cycle Product Solution

 ope
 B31\_ICPMS\_1
 B31\_ICPMS\_2
 B31\_ICPMS\_3
 B31\_ICPMS\_3

Isotope	B31_IC	CPMS_1	B31_ICPMS_2		B31_ICPMS_3		B31_ICPMS	
_	LW18154		LW18155		LW18156		Ave	Std Dev
_	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(μg/L)
U-234	7.82E+04	1.91E+00	7.82E+04	6.04E-01	7.93E+04	1.77E+00	7.86E+04	6.02E+02
U-235	6.39E+06	1.86E-01	6.31E+06	7.73E-01	6.28E+06	1.73E+00	6.33E+06	5.22E+04
U-236	4.43E+05	1.01E+00	4.41E+05	1.50E-03	4.38E+05	1.29E+00	4.41E+05	2.68E+03
U-238	2.91E+06	1.33E-01	2.87E+06	2.77E-01	2.84E+06	1.57E+00	2.88E+06	3.19E+04

MDA

Table A-9. Trace Metal Analyses for the 2<sup>nd</sup> U Cycle Product Solution

Isotope	B31_IC	PMS_4	B31_IC	PMS_5	B31_IC	PMS_6	B31_I	CPMS
-	LW1	9012	LW1	9013	LW1	9014	Ave	Std Dev
_	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(μg/L)
Li	6.50E+00	6.14E 01	5.48E+00	1.49E+00	6.12E+00	1.71E+00	6.03E+00	5.14E 01
Be	<5.00E-01	MDA	<5.00E-01	MDA	<5.00E-01	MDA	MDA	MDA
В	5.10E+03	1.33E+00	4.13E+03	4.17E-01	2.81E+03	3.86E-01	4.02E+03	1.15E+03
Na	<5.00E-01	MDA	<5.00E-01	MDA	<5.00E-01	MDA	MDA	MDA
Mg	1.35E+02	2.64E-0	1.22E+02	5.01E-01	3.10E+02	9.24E-01	1.89E+02	1.05E+02
Al	8.12E+02	3.18E-01	6.15E+02	8.77E-01	1.00E+03	1.06E+00	8.10E+02	1.94E+02
Si	7.55E+03	1.02E+00	7.05E+03	7.38E-01	8.83E+03	8.23E-01	7.81E+03	9.16E+02
P	2.66E+03	2.74E-01	2.68E+03	6.01E-01	2.77E+03	8.56E-01	2.70E+03	6.04E+01
K	1.56E+03	2.40E+00	1.10E+03	3.12E+00	1.43E+03	4.24E+00	1.36E+03	2.34E+02
Ca	6.56E+02	1.45E+00	6.07E+02	2.18E+00	4.66E+03	8.95E-01	1.97E+03	2.32E+03
V	<5.00E-01	MDA	5.05E-01	7.23E+00	8.50E-01	4.35E+00	6.77E-01	2.44E-01
Cr	8.83E+01	3.00E+00	9.04E+01	1.23E+00	9.02E+01	3.04E+00	8.96E+01	1.19E+00
Mn	1.78E+01	9.66E-01	1.81E+01	2.59E+00	2.66E+01	1.74E+00	2.08E+01	4.96E+00
Fe	2.71E+03	4.66E-01	2.71E+03	8.43E-01	2.82E+03	1.27E+00	2.75E+03	6.55E+01
Co	1.03E+00	6.10E+00	1.02E+00	6.50E+00	1.16E+00	2.72E+00	1.07E+00	7.98E-02
Ni	1.29E+02	3.02E-01	1.41E+02	4.24E-01	1.27E+02	1.63E+00	1.32E+02	7.80E+00
Cu	7.08E+01	6.70E-01	7.12E+01	1.96E+00	8.16E+01	8.90E-01	7.45E+01	6.15E+00
Zn	2.86E+01	1.51E+00	3.54E+01	4.59E+00	2.77E+02	1.50E+00	1.14E+02	1.41E+02
As	<5.00E-01	MDA	<5.00E-01	MDA	9.21E-01	1.55E+01	6.40E-01	2.43E-01
Zr	1.99E+00	4.06E+00	1.57E+00	1.10E+00	3.91E+00	3.55E+00	2.49E+00	1.25E+00
Nb	5.54E-01	3.41E+00	5.50E-01	9.06E+00	<5.00E-01	MDA	5.52E-01	2.68E-03
Mo	5.49E+00	7.10E+00	5.02E+00	3.76E+00	4.53E+00	5.81E+00	5.01E+00	4.83E-01
Ag	5.05E-01	2.46E+01	<5.00E-01	MDA	<5.00E-01	MDA	MDA	MDA
Cd	5.06E-01	1.96E+01	<5.00E-01	MDA	2.79E+00	1.17E+01	1.65E+00	1.62E+00
Sn	1.12E+00	1.13E+01	1.33E+00	7.87E+00	1.92E+00	4.28E+00	1.46E+00	4.15E-01
Sm	<5.00E-01	MDA	<5.00E-01	MDA	<5.00E-01	MDA	MDA	MDA
Eu	<5.00E-01	MDA	<5.00E-01	MDA	<5.00E-01	MDA	MDA	MDA
Gd	<5.00E-01	MDA	8.63E-01	3.96E+00	2.27E+00	4.03E+00	1.57E+00	9.95E-01
Dy	<5.00E-01	MDA	<5.00E-01	MDA	<5.00E-01	MDA	MDA	MDA
W	7.23E+00	1.52E+01	6.03E+00	1.70E+01	5.37E+00	2.06E+01	6.21E+00	9.41E-01
Pb	3.61E+00	4.83E+00	3.73E+00	5.95E+00	4.88E+01	9.18E-01	1.87E+01	2.61E+01

## Total Carbon

The concentrations of TIC/TOC measured by the total carbon method in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-10. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses since all values were below the MDA.

Table A-10. Total Carbon Analyses for the 2<sup>nd</sup> U Cycle Product Solution

Carbon	B31_TC_1		B31_	TC_2	B31_TC_3		
_	LW18157		LW1	8158	LW18159		
_	(μg/mL)	(%)	(μg/mL)	(%)	(μg/mL)	(%)	
Inorganic	<20	MDA	<20	MDA	<20	MDA	
Organic	<20	MDA	<20	MDA	<20	MDA	
Total	<40	MDA	<40	MDA	<40	MDA	

## IC-Anion

The concentrations of anions measured by the IC-Anions method in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-11. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses which were below the MDA.

Table A-11. IC-Anion Analyses for the 2<sup>nd</sup> U Cycle Product Solution

Anion	B31_IC-	B31_IC-Anion_1		·Anion_2	B31_IC-	Anion_3	B31_IC	C-Anion
_	LW1	8160	LW18161		LW18162		Ave	Std Dev
_	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(μg/L)
Fluoride	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA
Formate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA
Chloride	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA
Nitrite	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA
Nitrate	44900	10	45600	10	46200	10	45567	651
Phosphate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA
Sulfate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA
Oxalate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA
Bromide	<5000	MDA	<5000	MDA	<5000	MDA	MDA	MDA

## Free Acid

The HNO<sub>3</sub> concentration measured by the free acid method in the 2<sup>nd</sup> U Cycle Product solution are summarized in Table A-12. The sample identification and AD LIMS number are provided for each sample.

Table A-12. Free Acid Analyses for the 2<sup>nd</sup> U Cycle Product Solution

B31_Fre	eAcid_1	B31_FreeAcid_2		B31_Fre	eeAcid_3	B31_FreeAcid	
LW1	8163	LW1	8163	LW1	8163	Ave	Std Dev
(M)	(%)	(M)	(%)	(M)	(%)	(M)	(M)
0.605	10	0.627	10	0.600	10	0.611	0.014

### Appendix B. Analytical Measurements for NU Solution

The triplet analyses for each analytical method performed to characterize the NU solution are summarized below. The uncertainties in the individual samples are provided as a 1 sigma relative standard deviation based on the results from the AD or F/H Lab analysis method. The uncertainty in the average values was calculated from the three analyses.

#### Pu TTA/APHA

The Pu isotopic concentrations measured by the Pu TTA/APHA method in the NU solution are summarized in Table B-1. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses since all but one of the values were below the minimum detection of the analysis. The Pu-239/240 analysis for the third sample is not significant given the MDA's reported for the first and secondsamples.

E32\_TTA/APHA\_1 E32\_TTA/APHA\_2 E32\_TTA/APHA\_3 Isotope LW18166 LW18167 LW18168 (dpm/mL) (%) (dpm/mL) (%) (dpm/mL) (%) <sup>238</sup>Pu <3.01E00 MDA <3.12E00 MDA <3.25E00 **MDA** <sup>239/240</sup>Pu <2.69E00 MDA <5.01E00 MDA 2.36E+00 26

Table B-1. Pu Isotopic Analyses for the NU Solution

#### **ICPMS**

The concentrations of the U isotopes and trace metals measured by the ICPMS method in the NU solution are summarized in Table B-2. The trace metal analyses provided in the table are the elements for which maximum concentrations are provided in the ASTM C1462 standard for U metal.<sup>7</sup> The sample identification and AD LIMS number are provided for the samples in the table. Average concentrations were not calculated for the analyses which were below the MDA.

Table B-2. U Isotope and Trace Metal Concentrations for NU Solution

Isotope	E32_IC	PMS_1	E32_IC	PMS_2	E32_IC	PMS_3	E32_I	CPMS
_	LW1	8172	LW1	8173	LW1	8174	Ave	Std Dev
_	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(μg/L)
Co	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
As	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Zr	<1.00E+02	MDA	1.05E+02	1.22E+01	<1.00E+02	MDA	MDA	MDA
Nb	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Mo	5.28E+02	9.49E+00	5.19E+02	2.22E+00	4.58E+02	6.78E+00	5.02E+02	3.81E+01
Ag	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Cd	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Sn	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Sm	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Eu	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Gd	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
Dy	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
W	2.37E+02	1.30E+01	2.36E+02	3.62E+00	1.97E+02	1.19E+01	2.23E+02	2.29E+01
Pb	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
U-232	<1.00E+02	MDA	<1.00E+02	MDA	<1.00E+02	MDA	MDA	MDA
U-234	2.42E+04	3.05E-01	2.42E+04	8.73E-02	2.41E+04	2.02E-01	2.42E+04	9.21E+01
U-235	3.46E+06	1.04E-01	3.51E+06	7.51E-01	3.45E+06	9.46E-01	3.47E+06	3.75E+04
U-236	7.16E+02	2.78E+00	7.53E+02	5.07E-01	6.98E+02	2.71E+00	7.23E+02	2.81E+01
U-238	4.93E+08	7.53E-01	4.94E+08	4.11E-01	4.92E+08	8.19E-02	4.93E+08	7.94E+05

A second ICPMS analysis was performed in which the U was removed using Eichrom Technologies' UTEVA® resin prior to the analysis. The analysis of duplicate samples is shown in Table B-3. The ICPMS analyses includes elements for which maximum concentrations are provided in the ASTM C1462 standard.<sup>7</sup> The F/H Lab LIMS numbers are provided for the samples in the table. Average concentrations were not calculated for the analyses which were below the MDA.

Table B-3. Trace Metal Concentrations for NU Solution Following U Removal

Element	F/H Lab	LIMS No.	Average	Standard
_	200790355	200790356		Deviation
_	(μg/L)	(μg/L)	(μg/L)	(μg/L)
Li	< 2	< 2	MDA	MDA
Be	< 2	< 2	MDA	MDA
В	10.1	9.4	9.8	0.5
Na	278	263	271	11
Mg	14.9	13.7	14.3	0.8
Al	77.2	70.9	74.05	4.45
Si	62.9	69.5	66.2	4.67
P	172	158	165	10
K	< 182	< 182	MDA	MDA
Ca	< 160	< 160	MDA	MDA
V	0.43	0.52	0.48	0.06
Cr	85.1	84.6	84.9	0.4
Mn	6.5	6.2	6.4	0.2
Fe	428	410	419	13
Со	0.77	0.82	0.80	0.04
Ni	71.6	70.1	70.9	1.1
Cu	< 7	< 7	MDA	MDA
Zn	< 32	< 32	MDA	MDA

Element	F/H Lab	LIMS No.	Average	Standard
_	200790355	200790356		Deviation
_	(μg/L)	(μg/L)	(µg/L)	(μg/L)
As	< 3	< 3	MDA	MDA
Zr	2.2	2.3	2.3	0.1
Nb	< 1	< 1	MDA	MDA
Mo	12.2	12.1	12.2	0.1
Ag	< 0.3	< 0.3	MDA	MDA
Cd	< 2	< 2	MDA	MDA
Sn	< 0.8	< 0.8	MDA	MDA
Sm	< 2	< 2	MDA	MDA
Eu	< 1	< 1	MDA	MDA
Gd	< 2	< 2	MDA	MDA
Dy	< 1	< 1	MDA	MDA
W	3.8	3.2	3.5	0.4
Pb	< 1	< 1	MDA	MDA

## **Total Carbon**

The concentrations of TIC/TOC measured by the total carbon method in the NU solution are summarized in Table B-4. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses since all values were below the MDA.

Table B-4. Total Carbon Analyses for the NU Solution

Carbon	E32_	TC_1	E32_	TC_2	E32_TC_3		
_	LW18175		LW1	8176	LW18177		
-	(μg/mL)	(%)	(μg/mL) (%)		(μg/mL)	(%)	
Inorganic	<20	MDA	<20	MDA	<20	MDA	
Organic	<20	MDA	<20	MDA	<20	MDA	
Total	<40	MDA	<40	MDA	<40	MDA	

## IC-Anion

The concentrations of anions measured by the IC-Anions method in the NU solution are summarized in Table B-5. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses which were below the MDA.

Table B-5. IC-Anion Analyses for the NU Solution

Anion	E32_IC-Anion_1		E32_IC-Anion_4		E32_IC-Anion_3		E32_IC-	E32_IC-Anion_3	
_	LW18178		LW18179		LW18180		Ave	Std Dev	
_	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(μg/L)	
Fluoride	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA	
Formate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA	
Chloride	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA	
Nitrite	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA	
Nitrate	301000	10	316000	10	301000	10	306000	8660	
Phosphate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA	
Sulfate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA	
Oxalate	<1000	MDA	<1000	MDA	<1000	MDA	MDA	MDA	
Bromide	<5000	MDA	<5000	MDA	<5000	MDA	MDA	MDA	

# Free Acid

The  $HNO_3$  concentration measured by the free acid method in the NU solution are summarized in Table B-6. The sample identification and AD LIMS number are provided for each sample.

Table B-6. Free Acid Analyses for the NU Solution

E32_FreeAcid_1 E32_FreeAcid_2		E32_Fre	eeAcid_3	E32_FreeAcid			
LW1	LW18181 LW18182		LW1	8183	Ave	Std Dev	
(M)	(%)	(M)	(%)	(M)	(%)	(M)	(M)
0.360	10	0.301	10	0.303	10	0.321	0.034

### Appendix C. Analytical Measurements for HALEU Solution

The triplet analyses for each analytical method performed to characterize the HALEU solution are summarized below. The uncertainties in the individual samples are provided as a 1 sigma relative standard deviation based on the results from the AD or F/H Lab analysis method. The uncertainty in the average values was calculated from the three analyses.

#### U-232

The U-232 concentrations measured in the HALEU solution are summarized in Table C-1. The sample identification and AD LIMS number are provided for each sample. Each of the three samples was analyzed three times. The reported average and standard deviation are for the results of the analysis of the nine samples.

HALEU-Blend-1- AdvRct-U-232-1 HALEU-Blend-1- AdvRct-U-232-2			-Blend-1- -U-232-3	HALEU-Blend-1- AdvRct-U-232			
LW	LW19369 LW19370		LW	19371	Average	Std Dev	
(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)
3.58E+03	20	3.68E+03	20	3.72E+03	20	3.50E+03	3.92E+02
3.02E+03	20	3.91E+03	20	4.02E+03	20	_	_
2.84E+03	20	3.45E+03	20	3.32E+03	20	_	_

Table C-1. U-232 Analyses for HALEU Solution

## Np-237

The Np-237 concentrations measured in the HALEU solution are summarized in Table C-2. The sample identification and AD LIMS number are provided for each sample. The average and standard deviation of the Np-237 concentration does not include the value reported for sample HALEU-Blend-1-AdvRct-Np-237-1 which is clearly an outlier based on the reported values for the other two samples.

HALEU-Blend-1- AdvRct-Np-237-1			-Blend-1- Np-237-2		-Blend-1- Np-237-3	HALEU-Blend-1- AdvRct-Np-237	
LW1	LW19384		LW19385		19386	Average	Std Dev
(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)
781	20	183	20	171	20	177	8

Table C-2. Np-237 Analyses for the HALEU Solution

### Pu TTA/APHA

The Pu isotopic concentrations measured by the Pu TTA/APHA method in the HALEU solution are summarized in Table C-3. The sample identification and AD LIMS number are provided for each sample. An average concentration was not calculated for the Pu-239/240 analyses since all values were below the MDA.

Table C-3. Pu Isotopic Analyses for the HALEU	Solution
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	HALEU-Blend-1-		HALEU-Blend-1-		HALEU-I	Blend-1-	HALEU-Blend-1-		
Isotope	AdvI	Rct-	AdvRct- AdvRct- Ad		AdvRct-		Adv	Rct-	
	TTA/APHA-1		TTA/APHA-2		TTA/APHA-3		TTA/APHA		
	LW19372		LW19373		LW19374		Ave	Std Dev	
_	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)	
<sup>238</sup> Pu	1.07E+02	8.23	1.15E+02	8.36	1.06E+02	7.70	1.09E+02	4.93E+00	
<sup>239/240</sup> Pu	<2.74E00	MDA	<2.58E00	MDA	<3.09E00	MDA	MDA	MDA	

## Am/Cm/Cf Isotopes

The Am, Cm, and Cf isotopic concentrations measured by the Am/Cm/Cf method in the HALEU solution are summarized in Table C-4. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated since all analyses were below the MDA.

Table C-4. Am, Cm, and Cf Isotopic Analyses for the HALEU Solution

Isotope	HALEU-Blend-1- AdvRct-Am/Cm-1		HALEU- AdvRct-A		HALEU-Blend-1- AdvRct-Am/Cm-3		
_	LW1	9375	LW1	9376	LW19377		
-	(dpm/mL)	(dpm/mL) (%)		(%)	(dpm/mL)	(%)	
Am-241	<2.55E00	MDA	<4.48E00	MDA	<2.76E00	MDA	
Am-243	<1.52E00	MDA	<2.84E00	MDA	<1.59E00	MDA	
Am-242m	<1.33E00	MDA	<1.38E00	MDA	<5.74E-01	MDA	
Cm-243	<4.76E00	MDA	<1.00E01	MDA	<5.53E00	MDA	
Cm-245	<3.93E00	MDA	<8.26E00	MDA	<4.56E00	MDA	
Cm-247	<4.48E00	MDA	<1.08E01	MDA	<4.95E00	MDA	
Cf-249	<4.79E00	MDA	<1.15E01	MDA	<5.86E00	MDA	
Cf-251	<4.52E00	MDA	<1.00E01	MDA	<5.50E00	MDA	
Cm-242	<1.10E00	MDA	<1.14E00	MDA	<4.74E-01	MDA	
Cm-244	<5.90E-01	MDA	<8.61E-01	MDA	<1.07E00	MDA	

## <u>Sr-90</u>

The Sr-90 concentrations measured in the HALEU solution are summarized in Table C-5. The sample identification and AD LIMS number are provided for each sample.

Table C-5. Sr-90 Analyses for the HALEU Solution

HALEU-Blend-1- AdvRct-Sr-90-1			-Blend-1- t-Sr-90-2	0.000.000.000.000.000.000.000.000.000	-Blend-1- t-Sr-90-3	HALEU-Blend-1- AdvRct-Sr-90		
LW	LW19378		LW19379		19380	Average	Std Dev	
(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)	
63.9	35.7	60.9	35.4	46.3	35.0	57.0	9.4	

#### Tc-99

The Tc-99 concentrations measured in the HALEU solution are summarized in Table C-6. The sample identification and AD LIMS number are provided for each sample. An average concentration was not calculated since all analyses were below the MDA.

HALEU-Blend-1-HALEU-Blend-1-**HALEU-Blend-1-**AdvRct-Tc-99-1 AdvRct-Tc-99-2 AdvRct-Tc-99-3 LW19381 LW19382 LW19383 (dpm/mL) (%) (dpm/mL) (%) (dpm/mL) (%) <1.60E+01 MDA **MDA MDA** <1.76E+01 <1.75E+01

Table C-6. Tc-99 Analyses for the HALEU Solution

#### **GPHA**

The concentrations of gamma-emitting isotopes measured by the GPHA method in the HALEU solution are summarized in Table C-7. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses which were below the MDA.

Isotope	HALEU-Blend-1- AdvRct-GPHA-1		3000 CONTROL C	HALEU-Blend-1- AdvRct-GPHA-2		HALEU-Blend-1- AdvRct-GPHA-3		HALEU-Blend-1- AdvRct-GPHA	
_	LW19366		LW19367		LW19368		Ave	Std Dev	
-	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(%)	(dpm/mL)	(dpm/mL)	
<sup>58</sup> Co	<8.00E00	MDA	<8.14E00	MDA	<8.13E00	MDA	MDA	MDA	
<sup>95</sup> Nb	<5.75E00	MDA	<8.21E00	MDA	<8.36E00	MDA	MDA	MDA	
<sup>95</sup> Zr	<1.52E01	MDA	<1.54E01	MDA	<1.53E01	MDA	MDA	MDA	
<sup>103</sup> Ru	<8.03E00	MDA	<7.93E00	MDA	<8.20E00	MDA	MDA	MDA	
<sup>106</sup> Ru	<5.86E01	MDA	<5.87E01	MDA	<5.84E01	MDA	MDA	MDA	
<sup>134</sup> Cs	<8.22E00	MDA	<8.25E00	MDA	<8.27E00	MDA	MDA	MDA	
<sup>137</sup> Cs	<9.60E00	MDA	<7.70E00	MDA	<7.09E00	MDA	MDA	MDA	
<sup>144</sup> Ce	<2.21E+02	MDA	<2.20E02	MDA	<2.21E02	MDA	MDA	MDA	
<sup>208</sup> Tl	2.86E+02	5.00	2.84E+02	5.00	2.88E+02	5.00	2.86E+02	2.00E+00	
<sup>212</sup> Bi	9.27E+02	5.00	9.57E+02	5.00	9.35E+02	5.00	9.40E+02	1.55E+01	
<sup>212</sup> Pb	1.28E+03	5.00	9.70E+02	5.00	8.28E+02	5.00	1.03E+03	2.31E+02	
<sup>237</sup> Np/ <sup>233</sup> Pa	1.58E+02	5.00	1.61E+02	5.00	1.61E+02	5.00	1.60E+02	1.73E+00	
<sup>234m</sup> Pa/ <sup>234</sup> Th/ <sup>238</sup> U	2.00E+04	5.00	1.99E+04	5.00	2.09E+04	5.00	2.03E+04	5.51E+02	
235U	3.12E+04	5.00	3.10E+04	5.00	3.12E+04	5.00	3.11E+04	1.15E+02	
<sup>241</sup> Am	<2.06E02	MDA	<2.06E02	MDA	<2.05E02	MDA	MDA	MDA	

Table C-7. Gamma-emitting Isotope Analyses for the HALEU Solution

#### **ICPMS**

The U isotopic concentrations measured by the F/H Lab ICPMS method<sup>11</sup> for the HALEU solution are provided in Table C-8. The concentration of trace metals in the HALEU solution were measured using ICPMS methods by SRNL AD and SRNL F/H Lab. Uranium isotopic concentrations for the HALEU solution are also included in the AD data. In the F/H Lab method, the U was removed using Eichrom Technologies' UTEVA® resin prior to the analysis. The analyses performed by AD and F/H Lab are shown in Table C-9 and Table C-10, respectively. The trace metal analyses in Table C-9 and Table C-10 include elements for which maximum concentrations are provided in the ASTM C1462 standard for U metal.<sup>7</sup> The sample identification and AD LIMS number are provided for the samples in the three tables. Average concentrations were not calculated for the analyses which were below the MDA.

Table C-8. U Isotopic Concentrations Analyses for HALEU Solution

Isotope	HALEU-Blend-1- FHL-ICPMS-1	HALEU-Blend-1- FHL-ICPMS-2	HALEU-Blend-1- FHL-ICPMS-3	HALEU-Blend-1- FHL-ICPMS		
_	LW19407	LW19408	LW194079	Ave	Std Dev	
_	(wt %)	(wt %)	(wt %)	(wt %)	(wt %)	
U-234	0.278	0.277	0.278	0.278	0.001	
U-235	19.369	19.361	19.361	19.364	0.005	
U-236	1.285	1.282	1.285	1.284	0.002	
U-238	79.069	79.080	79.076	79.075	0.006	

Table C-9. Trace Metal and U Isotopic Analyses for HALEU Solution

Isotope	HALEU- -ICP			J-Blend-1B PMS-2		-Blend-1B PMS-3	HALEU-I	
-	LW1	9521	LW	19522	LW	19523	Ave	Std Dev
_	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(μg/L)
Li	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Be	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
В	1.92E+01	2.09E+00	1.62E+01	1.28E+01	1.68E+01	7.20E+00	1.74E+01	1.58E+00
Na	1.99E+03	1.31E+00	1.95E+03	1.25E+00	1.95E+03	2.02E-01	1.97E+03	2.42E+01
Mg	1.10E+02	1.15E-01	1.04E+02	1.55E+00	9.70E+01	2.63E-01	1.03E+02	6.36E+00
Al	8.47E+01	2.84E+00	7.89E+01	1.58E+00	8.96E+01	3.97E+00	8.44E+01	5.35E+00
Si	5.51E+03	1.38E+00	5.47E+03	1.30E+00	5.44E+03	8.79E-01	5.47E+03	3.57E+01
P	3.94E+03	1.11E+00	3.77E+03	1.04E+00	3.62E+03	1.64E+00	3.78E+03	1.57E+02
K	<1.00E+01	MDA	<1.00E+01	MDA	<1.00E+01	MDA	<1.00E+01	MDA
Ca	<1.00E+01	MDA	<1.00E+01	MDA	<1.00E+01	MDA	<1.00E+01	MDA
V	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Cr	2.46E+02	2.36E+00	2.40E+02	1.70E+00	2.37E+02	1.65E+00	2.41E+02	4.83E+00
Mn	3.09E+01	1.45E+00	3.00E+01	2.15E+00	2.97E+01	2.51E+00	3.02E+01	6.37E-01
Fe	3.41E+03	1.42E+00	5.27E+03	1.03E+01	5.98E+03	1.59E+01	4.89E+03	1.33E+03
Co	2.05E+00	9.07E+00	2.47E+00	9.94E+00	2.50E+00	1.33E+01	2.34E+00	2.48E-01
Ni	2.49E+02	3.17E+00	2.53E+02	1.17E+00	2.49E+02	8.23E-01	2.50E+02	1.96E+00
Cu	8.09E+01	9.66E-01	8.25E+01	1.67E+00	8.86E+01	6.63E-01	8.40E+01	4.06E+00
Zn	2.63E+01	4.81E+00	3.17E+01	2.09E+00	1.49E+01	7.20E+00	2.43E+01	8.59E+00
As	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Zr	2.65E+01	3.82E+00	2.01E+02	2.10E+01	2.58E+02	3.16E+01	1.62E+02	1.21E+02
Nb	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Mo	4.56E+02	4.76E+00	7.18E+02	1.13E+01	7.91E+02	2.35E+01	6.55E+02	1.76E+02
Ag	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Cd	<2.00E+00	MDA	<2.00E+00	MDA	2.68E+00	2.64E+01	2.23E+00	3.92E-01
Sn	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Sm	4.07E+00	1.02E+01	4.42E+00	4.13E+00	4.68E+00	4.90E+00	4.39E+00	3.10E-01
Eu	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Gd	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
Dy	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	MDA
W	7.68E+00	3.29E+00	7.67E+00	5.88E+00	7.72E+00	8.01E+00	7.69E+00	2.77E-02
Pb	6.42E+00	4.26E+00	5.03E+00	4.23E+00	5.52E+00	1.00E+01	5.66E+00	7.07E-01
<sup>234</sup> U	8.15E+04	3.10E+00	7.68E+04	3.47E+00	7.62E+04	4.92E+00	7.82E+04	2.91E+03
235U	6.21E+06	7.55E-01	6.17E+06	1.79E-01	6.13E+06	1.97E-01	6.17E+06	4.18E+04
236U	3.71E+05	4.79E-01	3.55E+05	3.38E-01	3.68E+05	3.91E-01	3.65E+05	8.59E+03
238U	2.42E+07	4.65E-01	2.42E+07	3.56E-01	2.42E+07	3.80E-01	2.42E+07	2.13E+04

Table C-10. Trace Metal Analyses for HALEU Solution Following U Removal

Isotope	HALEU-BI ICPI	end-1-FHL- MS-1	HALEU-BI ICPI	end-1-FHL- MS-2			HALEU-Blend-1-FHL- ICPMS	
= = =	LW1	9407	LW1	9408	LW19409		Ave	Std Dev
_	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(%)	(μg/L)	(μg/L)
Li	<2.00E+00	MDA	<2.00E+00	MDA	<2.00E+00	N/A	<2.00E+00	MDA
Be	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA
В	<8.00E+00	MDA	<8.00E+00	MDA	<8.00E+00	MDA	<8.00E+00	MDA
Na	2.05E+02	5.36E-01	1.86E+02	7.16E-01	2.30E+02	5.39E-01	2.07E+02	2.21E+01
Mg	4.65E+01	2.02E+00	5.19E+01	2.25E-01	3.46E+01	1.73E+00	4.43E+01	8.85E+00
Al	7.39E+01	3.15E-01	5.13E+01	1.51E-01	9.15E+01	3.29E-01	7.22E+01	2.02E+01
Si	3.70E+02	5.29E+00	<3.00E+01	MDA	<3.00E+01	MDA	<3.00E+01	MDA
P	1.46E+03	4.14E+00	1.46E+03	5.72E+00	1.31E+03	1.40E+00	1.41E+03	8.52E+01
K	<6.50E+01	MDA	<6.50E+01	MDA	<6.50E+01	MDA	<6.50E+01	MDA
Ca	1.31E+02	4.08E-01	2.77E+02	4.08E-01	<8.00E+01	MDA	2.04E+02	1.03E+02
V	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
Cr	1.66E+01	1.27E+00	1.66E+01	1.27E+00	1.67E+01	7.56E-01	1.66E+01	5.77E-02
Mn	<1.50E+00	MDA	<1.50E+00	MDA	<1.50E+00	MDA	<1.50E+00	MDA
Fe	2.73E+02	3.64E-01	3.05E+02	8.69E-01	3.08E+02	1.33E+00	2.95E+02	1.94E+01
Co	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
Ni	2.04E+01	1.53E+01	2.19E+01	1.24E+01	2.10E+01	6.47E+00	2.11E+01	7.55E-01
Cu	1.32E01L	3.89E+00	1.05E+01	2.99E+00	6.10E+00	1.88E+01	8.30E+00	3.11E+00
Zn	<3.00E+01	MDA	<3.00E+01	MDA	<3.00E+01	MDA	<3.00E+01	MDA
As	1.90E+00	3.89E+00	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA
Zr	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
Nb	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
Mo	4.40E+00	2.18E+00	1.00E+00	2.02E+00	<1.00E+00	MDA	2.70E+00	2.40E+00
Ag	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
Cd	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
Sn	<0.50E+00	MDA	<0.50E+00	MDA	5.90E+00	4.47E-01	<0.50E+00	MDA
Sm	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA
Eu	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
Gd	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA
Dy	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA	<0.50E+00	MDA
W	1.80E+00	7.75E+00	<1.00E+00	MDA	<1.00E+00	MDA	<1.00E+00	MDA
Pb	<1.00E+00	MDA	<1.00E+00	MDA	6.95E+01	3.15E-01	<1.00E+00	MDA

# Total Carbon

The concentrations of TIC/TOC measured by the total carbon method in the HALEU solution are summarized in Table C-11. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses since all values were below the MDA.

Table C-11. Total Carbon Analyses for the HALEU Solution

Carbon	Marian Control	Blend-1- t-TC-1		·Blend-1- et-TC-1	HALEU-Blend-1- AdvRct-TC-1		
_	LW19393		LW19394		LW19395		
_	(μg/mL)	(%)	(μg/mL)	(%)	(μg/mL)	(%)	
Inorganic	<20	MDA	<20	MDA	<20	MDA	
Organic	<20	MDA	<20	MDA	<20	MDA	
Total	<40	MDA	<40	MDA	<40	MDA	

## **IC-Anion**

The concentrations of anions measured by the IC-Anions method in the HALEU solution are summarized in Table C-12. The sample identification and AD LIMS number are provided for each sample. Average concentrations were not calculated for the analyses which were below the MDA.

Table C-12. IC-Anion Analyses for the HALEU Solution

Anion				HALEU-Blend-1- AdvRct-IC_A-2		HALEU-Blend-1- AdvRct-IC_A-3		HALEU-Blend-1- AdvRct-IC	
_	LW19396		LW19397		LW19398		Ave	Std Dev	
_	(μg/mL)	(%)	(μg/mL)	(%)	(μg/mL) (%)		(μg/mL)	(μg/mL)	
Fluoride	<100	MDA	<100	MDA	<100	MDA	<100	MDA	
Formate	<100	MDA	<100	MDA	<100	MDA	<100	MDA	
Chloride	<100	MDA	<100	MDA	<100	MDA	<100	MDA	
Nitrite	<100	MDA	<100	MDA	<100	MDA	<100	MDA	
Nitrate	60000	10	64000	10	61000	10	61667	2082	
Phosphate	<100	MDA	<100	MDA	<100	MDA	<100	MDA	
Sulfate	<100	MDA	<100	MDA	<100	MDA	<100	MDA	
Oxalate	<100	MDA	<100	MDA	<100 MDA		<100	MDA	
Bromide	<100	MDA	<100	MDA	<100	MDA	<100	MDA	

## Free Acid

The HNO<sub>3</sub> concentration measured by the free acid method in the HALEU solution are summarized in Table C-13. The sample identification and AD LIMS number are provided for each sample.

Table C-13. Free Acid Analyses for the HALEU Solution

HALEU-Blend-1- AdvRct-FA-1		HALEU-Blend-1- AdvRct-FA-2		HALEU-Blend-1- AdvRct-FA-3		HALEU-Blend-1- AdvRct-FA	
LW19363		LW19364		LW19365		Ave	Std Dev
(M)	(%)	(M)	(%)	(M)	(%)	(M)	(M)
0.57	20	0.56	20	0.54	20	0.56	0.02

#### Distribution

natraj.iyer@srnl.doe.gov william.bates@srnl.doe.gov greg.chandler@srnl.doe.gov kerry.dunn@srnl.doe.gov dave.dunn@srnl.doe.gov douglas.lowry@srnl.doe.gov philip.almond@srnl.doe.gov catherine.mussi@srnl.doe.gov kristine.zeigler@srnl.doe.gov marissa.reigel@srnl.doe.gov jonathan.duffey@srnl.doe.gov gene.daniel@srnl.doe.gov tracy.rudisill@srnl.doe.gov christopher.armstrong@srnl.doe.gov justin.doman@srnl.doe.gov harris.eldridge@srnl.doe.gov jarrod.gogolski@srnl.doe.gov nicholas.karay@srnl.doe.gov eddie.kyser@srnl.doe.gov michael.lee@srnl.doe.gov robert.pierce@srnl.doe.gov john.scogin@srnl.doe.gov thomas.shehee@srnl.doe.gov tracy.stover@srs.gov Records Administration (EDWS)