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Cesium and Isopar® L Concentrations in Samples Collected during Modular Caustic Side Solvent Extraction Unit (MCU) Simulant Testing

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SUMMARY

During simulant testing, the Modular Caustic Side Solvent Extraction Unit (MCU) collected samples and submitted them to Savannah River National Laboratory (SRNL) personnel for analysis of cesium, Isopar® L, and Modifier [1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol]. SRNL personnel analyzed the aqueous samples for cesium by Inductively-Coupled Plasma Mass Spectroscopy (ICP-MS) and the solvent samples for cesium using a Parr Bomb Digestion followed by ICP-MS. They analyzed aqueous samples for Isopar® L and Modifier by gas chromatography (GC). They submitted control samples to verify the accuracy of the analytical methods.

The conclusions from the cesium analyses follow.

- The cesium in the feed samples measured 15.8 (±10%) mg/L, in agreement with expectations.
- The decontamination factor measured 90 – 1580 for Mass Transfer Test A (conducted at 3.5 gpm salt solution flow rate), 106 – 252 for Mass Transfer Test B (conducted at 6.0 gpm salt solution flow rate), and 138 – 878 for Mass Transfer Test C (conducted at 8.5 gpm salt solution flow rate).
- The concentration factor measured 11.0 – 11.1 for Mass Transfer Test A (3.5 gpm salt solution flow rate), 12.8 – 13.2 for Mass Transfer Test B (6.0 gpm salt solution flow rate), and 12.0 – 13.2 for Mass Transfer Test C (8.5 gpm salt solution flow rate).
- The organic carryover from the final extraction contactor (#7) varied between 22 and 710 mg/L Isopar® L. The organic carryover was less in Test A (3.5 gpm salt solution flow rate) than in Tests B and C (6.0 and 8.5 gpm salt solution flow rate, respectively).
- The organic carryover from the final strip contactor (#7) varied between 80 and 180 mg/L Isopar® L
- The organic carryover in the Decontaminated Salt Solution Hold Tank and the Strip Effluent Hold Tank was less than 10 mg/L Isopar® L, indicating good recovery of the solvent by the coalescers and decanters.
- The measured concentrations in the control samples agreed with the prepared concentrations.

INTRODUCTION

The Department of Energy identified the Caustic Side Solvent Extraction (CSSX) process as the preferred technology for removing cesium from radioactive waste solutions at the Savannah River Site (SRS).[1,2] As a result, the Washington Savannah River Company (WSRC) designed and built the MCU facility in the SRS Tank Farm to process liquid waste for an interim period until the Salt Waste Processing Facility (SWPF) begins operations.

SRS personnel have completed construction and assembly of the MCU facility. Following assembly, they conducted testing to evaluate the ability of the process to remove non-radioactive cesium and to separate the aqueous and organic phases. These tests are referred to as the Mass Transfer Tests.

They conducted the tests as follows. A vendor (Blue Line Chemical) prepared simulated SRS salt solution. MCU personnel added non radioactive cesium to the salt solution to achieve a cesium concentration of 14.9 mg/L (equivalent to 1.1 Ci/gal ^{137}Cs). They processed the salt solution through the MCU process at flow rates of 3.5, 6.0, and 8.5 gpm (referred to as Tests A, B, and C, respectively). During the testing, they collected samples from the inlet and outlet of selected contactors to measure cesium removal from the salt solution, cesium transfer from the solvent to the strip acid, and organic solvent carryover into the Decontaminated Salt Solution (DSS) and Strip Effluent (SE). They collected samples from the Decontaminated Salt Solution Hold Tank (DSSHT) and the Strip Effluent Hold Tank (SEHT) to measure the effectiveness of the coalescers in recovering solvent from the aqueous streams. Following the Mass Transfer Tests, they performed a Solvent Cleanup Test which they fed Decontaminated Salt Solution through the contactors to remove cesium from the solvent. Following that test, they stopped and restarted the MCU process – designated as the System Shutdown/Restart Test – to determine its ability to rapidly reestablish process efficiency after shutdown and restart.

During the Mass Transfer Tests, the MCU facility collected samples from the Salt Solution Receipt Tank 1 (SSRT#1), the Salt Solution Feed Tank (SSFT), Extraction Contactor #1 aqueous inlet, Extraction Contactor #7 solvent inlet, Extraction Contactor #7 aqueous outlet, Strip Contactor #7 aqueous outlet, the DSSHT, the SEHT, the Solvent Hold Tank (SHT), and the Contactor Drain Tank (CDT). They submitted the samples to SRNL for analysis of cesium, Isopar® L, and Modifier. In addition to the samples listed above, the authors prepared and submitted a set of controls with selected batches of samples being analyzed.

This document describes the analyses.

ANALYSES

The Analytical Development (AD) group of SRNL performed the ^{133}Cs analysis by ICP-MS. The aqueous samples (Decontaminated Salt Solution and Strip Effluent) were submitted directly to the ICP-MS. The solvent samples were digested using a Parr Bomb Digestion prior to analysis by ICP-MS. The AD group analyzed DSS and SE samples for Isopar® L and Modifier by gas chromatography-flame ionization detection (GC-FID) and gas chromatography-mass spectrometry (GC-MS).

The ICP-MS used for the analyses is a Thermo-Elemental Plasma Quad II. This instrument provides multi-element analyses of aqueous solutions, and the analytical results can be expressed as either elemental or isotopic concentrations. The instrument aerosolizes the sample and transports the aerosol to the argon plasma. In the high temperature plasma (~10,000 K) metallic species are ionized. The ions generated by the plasma enter the mass spectrometer through a sampling cone set near the end of the plasma. The ions are separated by a quadrapole mass filter and focused on a detector. The detector provides either an ion count or an analog signal. The signal from the detector is amplified, measured, and stored in a multi-channel analyzer, and these measurements are used to calibrate the instrument and determine the concentrations of the elements of concern.

AD personnel performed the solvent sample digestions as follows. Approximately 0.1-0.2 g of the well-mixed sample was transferred to the Teflon™ cup of a Parr Bomb dissolution container.

A 3 mL aliquot of high-purity concentrated nitric acid was added and the dissolution container sealed. Typically eight containers were heated simultaneously in an oven pre-heated to 175 °C. Heating was continued for at least three hours after the oven temperature re-equilibrated to 175 °C. After cooling to room temperature, the containers were opened and the nitric acid solutions were diluted to 10 mL with de-ionized water. No immiscible organic fraction or solution cloudiness was evident after this treatment, indicating that the oxidation of the organic fraction in the samples was complete.

Personnel performed the GC-FID and GC-MS analyses as follows. They weighed the sample bottle. They either added hexane to the sample bottle (1/4 of sample volume) or transferred the sample to a larger bottle and rinsed the sample bottle with the hexane. They recorded the weight of the bottle, sample, and hexane. They removed the top layer of liquid and placed it in a vial with a Teflon™ cap. They recorded the empty bottle weight. They dried the hexane with sodium sulfate, collected aliquots, and analyzed them.

GC-MS analysis or GC-FID analysis was employed to identify organic compounds in the samples. Analytical separations were carried out on a Hewlett Packard 6890 gas chromatograph, equipped with a 30 m DB-XLB column, with 0.18 mm diameter and 0.20 μ film thickness for GC-MS. The GC-FID uses a 30 m DB-5ms column, with 0.2 mm diameter and 0.33 μ film thickness. Quantitation was performed using a Hewlett Packard 5973 mass selective detector. The mass spectrometer tuning was confirmed within 24 hours prior to each measurement using perfluorotributylamine.

The authors prepared controls for each set of samples analyzed. They prepared the controls by adding solvent and/or Isopar® L to simulated SRS supernate solution and 0.001 M nitric acid. They prepared some of the controls gravimetrically by adding a known mass of solvent and/or Isopar® L to a known mass of supernate solution or acid. They prepared other controls by serial dilution. They dissolved a known volume of solvent and/or Isopar® L in hexane, and added a known volume of hexane to a known volume of supernate or nitric acid.

RESULTS

Cesium Removal

Table 1 shows the analysis of the feed solution along with the control submitted. The feed cesium concentration measured 15.8 mg/L in both samples versus a target of 15 mg/L. The 15 mg/L control sample measured 14.6 mg/L (3% difference), well within the standard analytical uncertainty of ±10%. The analytical uncertainty on all measured values is ±10%, unless otherwise stated.

Table 1. Feed Solution Cesium Concentration

Sample	Cesium (mg/L)
MCU-CS-I-SSRT	15.8
MCU-CS-I-SSFT	15.8
15 mg/L Control	14.6

Table 2 shows the cesium concentration in the samples from Mass Transfer Test A. The Decontamination Factor (DF) varied between 181 and 1580, with an average value of 348. The Concentration Factor (CF) varied between 11.0 and 11.1. The cesium concentration in the solvent entering the extraction contactors was less than 1.1 mg/L.

Table 2. Cesium Concentration (mg/L) during Mass Transfer Test A

	Feed	DSS	SE	Solvent	DF	CF
MCU-CS-I-SSRT	15.8					
MCU-CS-I-SSFT	15.8					
MCU-CS-A-EC-A-O-1		< 0.010				
MCU-CS-A-EC-A-O-2		0.017				
MCU-CS-A-EC-A-O-3		0.069				
MCU-CS-A-EC-A-O-5		0.087				
MCU-CS-A-EC-A-O-6		0.044				
MCU-CS-A-SC-A-O-1			174			
MCU-CS-A-SC-A-O-3			176			
MCU-CS-A-SC-A-O-5			174			
MCU-CS-A-EC-O-I				1.062		
MCU-CS-A-EC-O-I-1				0.209		
MCU-CS-A-EC-O-I-3				0.849		
MCU-CS-A-EC-O-I-5				0.400		
MCU-CS-A-EC-O-I-6				0.588		
Minimum		< 0.010	174		181	11.0
Maximum		0.087	176		>1580	11.1
Average		0.045	175		348	11.1
Standard Deviation		0.033	1.1			

Table 3 shows the cesium concentration in the samples from Mass Transfer Test B. The DF varied between 211 and 252, with an average value of 227. The CF varied between 12.8 and 13.2. The cesium concentration in the solvent entering the extraction contactors was less than 1 mg/L.

Table 4 shows the cesium concentration in the samples from Mass Transfer Test C. The DF varied between 275 and 878, with an average value of 470. The CF varied between 12.0 and 13.2. The cesium concentration in the solvent entering the extraction contactors was less than 1 mg/L.

Tables 5 and 6 show the cesium concentration in the samples collected during the Solvent Cleanup Test. The purpose of this test was to assess the removal of cesium from the solvent by contacting it with decontaminated salt solution. Table 5 shows the cesium in the DSS samples. The cesium concentration decreased with time during this test, and all samples contained less than 0.2 mg/L cesium.

Table 3. Cesium Concentration (mg/L) during Mass Transfer Test B

	Feed	DSS	SE	Solvent	DF	CF
MCU-CS-I-SSRT	15.8					
MCU-CS-I-SSFT	15.8					
MCU-CS-B-EC-A-O-1		0.075				
MCU-CS-B-EC-A-O-3		0.071				
MCU-CS-B-EC-A-O-5		0.063				
MCU-CS-B-SC-A-O-1			204			
MCU-CS-B-SC-A-O-3			202			
MCU-CS-B-SC-A-O-5			209			
MCU-CS-B-SC-A-O-6			207			
MCU-CS-B-EC-O-I				0.749		
MCU-CS-B-EC-O-I-1				0.227		
MCU-CS-B-EC-O-I-3				0.214		
MCU-CS-B-EC-O-I-5				0.191		
Minimum		0.063	202		211	12.8
Maximum		0.075	209		252	13.2
Average		0.070	205.5		227	13.0
Standard Deviation		0.006	3.1			

Table 4. Cesium Concentration (mg/L) during Mass Transfer Test C

	Feed	DSS	SE	Solvent	DF	CF
MCU-CS-I-SSRT	15.8					
MCU-CS-I-SSFT	15.8					
MCU-CS-C-EC-A-O-1		0.057				
MCU-CS-C-EC-A-O-3		0.032				
MCU-CS-C-EC-A-O-5		0.028				
MCU-CS-C-EC-A-O-6		0.018				
MCU-CS-C-SC-A-O-1			208			
MCU-CS-C-SC-A-O-3			190			
MCU-CS-C-SC-A-O-5			199			
MCU-CS-C-EC-O-I				0.29		
MCU-CS-C-EC-O-I-1				0.14		
MCU-CS-C-EC-O-I-3				0.46		
MCU-CS-C-EC-O-I-5				0.077		
MCU-CS-C-EC-O-I-6				0.099		
Minimum		0.018	190		275	12.0
Maximum		0.057	208		878	13.2
Average		0.034	199		470	12.6
Standard Deviation		0.017	9			

Table 5. Cesium Concentration (mg/L) in Salt Solution during the Solvent Cleanup Test

Sample	Cesium (mg/L)
MCU-CS-W-EC-A-I-1	0.129
MCU-CS-W-EC-A-I-3	0.078
MCU-CS-W-EC-A-I-5	0.038
MCU-CS-W-EC-A-I-7	0.046
MCU-CS-W-EC-A-I-9	0.043

Table 6 shows the cesium concentration in the SHT. The cesium concentration measured less than 0.3 mg/L. The solvent cesium concentration at the conclusion of Mass Transfer Test C – and hence at the start of the Solvent Cleanup Test – measured 0.21 ± 0.16. The initial SHT sample, collected 50 minutes after the start of the Solvent Cleanup Test, had a cesium concentration of 0.030 mg/L versus an average concentration of 0.40 mg/L during the Mass Transfer Tests, showing a large fraction (~ 90 %) of the cesium had been removed from the solvent. Subsequent samples showed similar cesium concentrations. The last sample collected showed a higher cesium concentration. We are unsure of the reason for this increase.

Table 6. Cesium Concentration (mg/L) in Solvent Hold Tank during Solvent Cleanup Test

Sample	Cesium (mg/L)
MCU-CS-SHT-1	0.030
MCU-CS-SHT-3	0.025
MCU-CS-SHT-5	0.037
MCU-CS-SHT-7	0.051
MCU-CS-SHT-9	0.293

Table 7 shows the cesium concentration in the DSS, SE, and SHT 30 minutes after starting the System Shutdown/Restart Test. In this test, the MCU system was shut down and restarted. The DF was 268, and the CF was 11.8. These results are consistent with the results from Mass Transfer Tests A, B, and C. During the Mass Transfer Tests, the cesium in the Decontaminated Salt Solution averaged 0.048 ± 0.026 mg/L, the cesium in the Strip Effluent averaged 194 ± 15 mg/L, and the cesium in the solvent averaged 0.40 ± 0.31 mg/L. The DF averaged 491 ± 461, and the CF averaged 12.2 ± 0.9.

Table 7. Cesium Concentration during System Shutdown/Restart Test

Sample	Sample	Cesium (mg/L)
MCU-CS-I-SSFT	Feed	15.8
MCU-CS-D-EC-A-O-1	DSS	0.059
MCU-CS-D-SC-A-O-1	SE	187
MCU-CS-SHT-11	SHT	0.851
	DF	268
	CF	11.8

Table 8 shows the cesium concentration in the inlet to Extraction Contactor #1 for each test (MCU-CS-x-EC-A-I, x = A, B, or C). The concentration is slightly higher than in the SSRT#1 and the SSFT (15.8 mg/L).

Table 8. Cesium Concentration (mg/L) in Inlet to Extraction Contactor #1

Sample	Test	Cesium (mg/L)
MCU-CS-A-EC-A-I	A	16.8
MCU-CS-B-EC-A-I	B	16.9
MCU-CS-C-EC-A-I	C	18.3
MCU-CS-I-SSRT		15.8
MCU-CS-I-SSFT		15.8

Table 9 shows the target and measured cesium in the control samples prepared. The samples showed good agreement between the target and measured values. The difference between the target concentration and the measured concentration was 2 – 27%, and in 5 of the 7 samples, the difference was less than 10%.

Table 9. Cesium Concentration in Control Samples

Target Concentration (mg/L)	Measured Concentration (mg/L)	Digestion (Y/N)
15	15.4	N
150	159	N
0.14	0.11	Y
1.48	1.49	Y
1000	1020	N
15	16.1	N
1	1.27	N

During previous contactor testing conducted at Wright Industries[3], control samples with cesium concentrations less than 1 mg/L showed a negative bias of approximately 50% when analyzed by SRNL. When calculating DF values from those tests, the researchers multiplied any measured DSS values less than 1 mg/L by a factor of 2.[4] Applying that approach to this data results in the calculated values shown in Table 10.

Table 10. DF Values Calculated Applying Same Correction as Wright Industries Test

Test	A	B	C
Minimum DF	90	106	138
Maximum DF	790	136	439
Average DF	174	114	235
Minimum CF	11.0	12.8	12.0
Maximum CF	11.1	13.2	13.2
Average CF	11.1	13.0	12.6

Organic Carryover

Table 11 shows the measured Isopar® L concentration in aqueous samples collected from the outlet of Extraction Contactor #7. The organic carryover varied between 22 and 709 mg/L Isopar® L. The carryover measured during Test A was much less than measured during Tests B and C (31 mg/L Isopar® L versus 444 – 524 mg/L Isopar® L). This result is consistent with the results from the Integrated Test conducted at Wright Industries, where the organic carryover was

~ 80 mg/L Isopar® L at salt solution flow rates of 3.5 gpm, and 130 – 100 mg/L Isopar® L at flow rates of 4.5 – 8.5 gpm salt solution.[4]

Table 11. Isopar® L concentration in Extraction Contactor #7 Outlet

Sample ID	Test	Isopar® L (mg/L)	Modifier (mg/L)	Isopar® L/Modifier
MCU-ISO-A-EC-A-O-1	A	37.0	22.2	1.66
MCU-MS-1	A	33.2	14.2	2.34
MCU-ISO-A-EC-A-O-3	A	21.8	20.1	1.08
MCU-MS-3	A	33.6	19.7	1.71
MCU-ISO-A-EC-A-O-5	A	35.1	23.9	1.47
MCU-ISO-A-EC-A-O-6	A	31.7	23.6	1.34
MCU-MS-5	A	26.9	12.0	2.24
Average	A	31.3	19.4	1.61
Standard Deviation	A	5.3	4.6	
MCU-ISO-B-EC-A-O-1	B	487	233	2.09
MCU-MS-7	B	501.5	154.2	3.25
MCU-ISO-B-EC-A-O-3	B	366.8	174.6	2.10
MCU-ISO-B-EC-A-O-5	B	419.6	201.7	2.08
Average	B	443.7	190.9	2.32
Standard Deviation	B	62.5	34.2	
MCU-ISO-C-EC-A-O-1	C	709.1	315.0	2.25
MCU-ISO-C-EC-A-O-3	C	210.8	137.6	1.53
MCU-ISO-C-EC-A-O-5	C	651.0	286.9	2.27
Average	C	523.6	246.5	2.12
Standard Deviation	C	272.4	95.4	

Table 12 shows the measured Isopar® L concentration in aqueous samples collected from the DSSHT. In all samples, the Isopar® L concentration is less than 10 mg/L.

Table 12. Isopar® L concentration in Decontaminated Salt Solution Hold Tank

Sample ID	Test	Isopar® L (mg/L)	Modifier (mg/L)
MCU-ISO-A-DT-A-1	A	< 3	< 3
MCU-ISO-A-DT-A-3	A	< 2	< 2
MCU-ISO-A-DT-A-5	A	< 2	< 2
MCU-ISO-A-DT-A-6	A	< 2	< 2
MCU-ISO-B-DT-A-1	B	< 2	< 2
MCU-ISO-B-DT-A-3	B	< 2	< 2
MCU-ISO-B-DT-A-5	B	< 3	< 3
MCU-ISO-B-DT-A-6	B	< 3	29.9
MCU-ISO-C-DT-A-1	C	< 2	14.2
MCU-ISO-C-DT-A-3	C	4.9	7.3
MCU-ISO-C-DT-A-5	C	7.7	9.5

Table 13 shows the measured Isopar® L concentration in aqueous samples collected from the outlet of Strip Contactor #7. The organic carryover varied between 80 and 182 mg/L Isopar® L. No significant difference in organic carryover was observed between the different tests. These results are consistent with the Integrated Test conducted at Wright Industries, in which the organic carryover varied between 170 and 370 mg/L Isopar® L.

Table 13. Isopar® L concentration in Strip Contactor #7 Outlet

Sample ID	Test	Isopar® L (mg/L)	Modifier (mg/L)	Isopar® L/Modifier	Insoluble Modifier (mg/L)	Mod. Isopar® L/Modifier
MCU-ISO-A-SC-A-O-1	A	80.5	66.8	1.21	43.2	1.86
MCU-ISO-A-SC-A-O-3	A	145.7	108.2	1.35	84.6	1.72
MCU-ISO-A-SC-A-O-4	A	130.4	81.0	1.61	57.4	2.27
MCU-ISO-A-SC-A-O-5	A	181.8	96.7	1.88	73.1	2.49
Average	A	134.6	88.2	1.53	64.6	2.07
Standard Deviation	A	42.0	18.1			
MCU-ISO-B-SC-A-O-1	B	161.6	92.7	1.74	69.1	2.34
MCU-ISO-B-SC-A-O-3	B	158.3	89.9	1.76	66.3	2.39
MCU-ISO-B-SC-A-O-4	B	147.8	87.7	1.69	64.1	2.31
MCU-ISO-B-SC-A-O-5	B	167.8	112.0	1.50	88.4	1.90
Average	B	158.9	95.6	1.66	72.0	2.23
Standard Deviation	B	8.4	11.1			
MCU-ISO-C-SC-A-O-1	C	113.9	79.7	1.43	56.1	2.03
MCU-ISO-C-SC-A-O-3	C	98.4	74.9	1.31	51.3	1.92
MCU-ISO-C-SC-A-O-5	C	99.8	73.5	1.36	49.9	2.00
Average	C	104.0	76.0	1.37	52.4	1.98
Standard Deviation	C	8.6	3.3			

Table 14 shows the measured Isopar® L concentration in aqueous samples collected from the strip effluent hold tank (SEHT). In all samples, the Isopar® L concentration is less than 10 mg/L.

The modifier concentration in SEHT was 18 – 29 mg/L. The modifier concentration is higher than the Isopar® L concentration due to the Modifier's solubility in dilute nitric acid. The average Modifier concentration in the SEHT was 23.6 mg/L. Prior studies at Oak Ridge National Laboratory suggest a solubility of approximately 25 mg/L for Modifier in 1 mM nitric acid (i.e., strip acid).[7]

Table 14. Isopar® L concentration in SEHT

Sample ID	Test	Isopar® L (mg/L)	Modifier (mg/L)
MCU-ISO-A-ST-A-1	A	< 3	22.3
MCU-ISO-A-ST-A-3	A	< 3	23.6
MCU-ISO-A-ST-A-5	A	< 3	26.5
MCU-ISO-B-ST-A-1	B	< 3	25.2
MCU-ISO-B-ST-A-2	B	< 3	21.3
MCU-ISO-B-ST-A-3	B	< 2	20.1
MCU-ISO-B-ST-A-5	B	< 3	24.5
MCU-ISO-C-ST-A-1	C	< 3	25.9
MCU-ISO-C-ST-A-3	C	< 3	17.9
MCU-ISO-C-ST-A-5	C	< 3	28.8
Average		< 3	23.6

Subtracting the average soluble concentration (23.6 mg/L) for the Modifier from the measured Modifier in the Strip Effluent Contactor outlet samples, we calculate an insoluble Modifier concentration (see Table 13). Using the insoluble Modifier concentration, we calculate a modified Isopar® L to Modifier ratio, which varies between 1.72 and 2.49 with an average of 2.1. This average agrees well with the calculated Isopar® L to Modifier ratio of 2.32 for the fresh solvent. The low bias (~ 10%) could be due to analytical uncertainty or to evaporation of Isopar® L. If 9% of the Isopar® L evaporated, the Isopar® L:Modifier ratio would decrease to 2.1 and the density would increase to 0.869 g/mL versus 0.852 for the prepared solvent. Samples collected during the mass transfer tests had a measured density of 0.863, indicated some loss of Isopar® L. The low bias observed in the Isopar® L:Modifier ratio is likely due to Isopar® L evaporation and analytical uncertainty.

Table A-1 in Appendix A shows the analysis of the control samples submitted. The results show good agreement between the prepared and measured concentrations of Isopar® L and Modifier.

Figure A-1 in Appendix A shows the difference between the prepared Isopar® L concentration and the measured Isopar® L concentration in the control samples as a function of prepared Isopar® L concentration. At Isopar® L concentrations greater than 400 mg/L, the difference is less than 20%. At Isopar® L concentrations between 80 and 200 mg/L, the difference is less than 40%. At concentrations less than 80 mg/L, the difference is as much as 100%. In most of the samples, the measured concentration is less than the prepared concentration. The larger differences with the lower concentration controls could be a sign of incomplete recovery, as the little bit remaining in the sample bottles is a larger fraction of the total amount. The differences between prepared and measured Isopar® L appear larger when the controls were prepared by serial dilution. However, this difference is largest at low Isopar® L concentrations and is most likely due to the low Isopar® L concentration rather than the serial dilution preparation method.

Figure A-2 in Appendix A shows the difference between the prepared Modifier concentration and the measured Modifier concentration in the control samples as a function of prepared Modifier concentration. At Modifier concentrations greater than 150 mg/L, the difference is less than 25%. At Modifier concentrations between 30 and 100 mg/L, the difference is less than 30%. At concentrations less than 30 mg/L, the difference is as much as 60%. In most of the samples, the measured concentration is less than the prepared concentration. The larger differences with the lower concentration controls could be a sign of incomplete recovery, as the little bit remaining in the sample bottles is a larger fraction of the total amount.

Figure A-3 in Appendix A shows the differences between prepared Isopar® L concentration and the measured Isopar® L concentration. The figure compares the difference when the internal standards were added before and after the analytical method extraction of the solvent into hexane. At prepared Isopar® L concentrations greater 30 mg/L, there appears to be no statistical difference in the error between the samples in which the internal standard was added before the solvent extraction step and the samples in which it was added after the solvent extraction step.

Figure A-4 shows the ratio of Isopar® L to Modifier in the control samples. Based upon the solvent composition, this ratio should be 2.32. The figure shows that all but two samples are within 20% of this ratio. Those samples occurred at low Isopar® L concentrations (~10 mg/L). In most of the samples prepared by serial dilution, the ratio is greater than 2.32. In most of the samples prepared gravimetrically, the ratio is less than 2.32.

CONCLUSIONS

The conclusions from the cesium analyses follow.

- The cesium in the feed samples measured 15.8 (±10%) mg/L, in agreement with expectations.
- The decontamination factor measured 90 – 1580 for Mass Transfer Test A (conducted at 3.5 gpm salt solution flow rate), 106 – 252 for Mass Transfer Test B (conducted at 6.0 gpm salt solution flow rate), and 138 – 878 for Mass Transfer Test C (conducted at 8.5 gpm salt solution flow rate).
- The concentration factor measured 11.0 – 11.1 for Mass Transfer Test A (3.5 gpm salt solution flow rate), 12.8 – 13.2 for Mass Transfer Test B (6.0 gpm salt solution flow rate), and 12.0 – 13.2 for Mass Transfer Test C (8.5 gpm salt solution flow rate).
- The organic carryover from the final extraction contactor (#7) varied between 22 and 710 mg/L Isopar® L. The organic carryover was less in Test A (3.5 gpm salt solution flow rate) than in Tests B and C (6.0 and 8.5 gpm salt solution flow rate, respectively).
- The organic carryover from the final strip contactor (#7) varied between 80 and 180 mg/L Isopar® L
- The organic carryover in the Decontaminated Salt Solution Hold Tank and the Strip Effluent Hold Tank was less than 10 mg/L Isopar® L, indicating good recovery of the solvent by the coalescers and decanters.
- The measured concentrations in the control samples agreed with the prepared concentrations.

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APPENDIX A. ANALYSIS OF ISOPAR® L CONTROL SAMPLES

Table A-1. Analysis of Control Samples

Sample ID	Isopar® L prepared (mg/L)	Isopar® L measured (mg/L)	Modifier prepared (mg/L)	Modifier measured (mg/L)
MCU-ISO-101	667	660	290	267
MCU-ISO-102	689	613	300	233
MCU-ISO-103	710	741	309	271
MCU-ISO-104	168	102	73	52
MCU-ISO-105	158	143	69	62
MCU-ISO-106	101	87	44	42
MCU-ISO-107	112	102	49	52
MCU-ISO-108	60	47	26	25
MCU-ISO-109	48	45	21	22
MCU-ISO-110	70	37	30	16
MCU-ISO-111	43	42	19	17
MCU-ISO-112	26	21	11	10
MCU-ISO-113	26	25	11	11
MCU-ISO-114	15	10	7	3
MCU-ISO-115	16	10	7	3
MCU-ISO-116	16	10	7	4
MCU-ISO-117	16	9	7	3
MCU-ISO-118	671	586	292	276
MCU-ISO-119	645	526	280	253
MCU-ISO-120	677	548	294	265
MCU-ISO-121	43	38	19	16
MCU-ISO-122	43	36	19	16
MCU-ISO-123	44	37	19	16
MCU-ISO-124	16	8	7	3
MCU-ISO-125	16	8	7	3
MCU-ISO-126	16	8	7	3
MCU-ISO-127	426	427	185	206
MCU-ISO-128	529	535	230	265
MCU-ISO-129	168	149	73	78
MCU-ISO-130	172	166	75	81
MCU-ISO-131	572	584	0	0
MCU-ISO-132	626	690	0	0
MCU-ISO-133	466	426	0	0
MCU-ISO-134	449	447	0	0
MCU-ISO-135	176	136	0	0
MCU-ISO-136	193	141	0	0
MCU-ISO-137	45	46	0	0
MCU-ISO-138	46	42	0	0
MCU-ISO-139	680	625	227	219
MCU-ISO-140	592	618	197	180
MCU-ISO-141	610	643	203	206

Table A-1. Analysis of Control Samples (continued)

Sample ID	Isopar® L prepared (mg/L)	Isopar® L measured (mg/L)	Modifier prepared (mg/L)	Modifier measured (mg/L)
MCU-ISO-142	693	682	231	242
MCU-ISO-143	151	116	50	51
MCU-ISO-144	148	157	49	57
MCU-ISO-145	43	47	14	14
MCU-ISO-146	46	46	15	14
MCU-ISO-201	151	126	66	56
MCU-ISO-202	154	126	67	55
MCU-ISO-203	45	31	19	18
MCU-ISO-204	44	38	19	17
MCU-ISO-205	27	19	12	9
MCU-ISO-206	26	18	11	10
MCU-ISO-207	9	0.4	4	4
MCU-ISO-208	9	0.4	4	4
MCU-ISO-209	43	27	19	16
MCU-ISO-210	44	33	19	15
MCU-ISO-211	44	33	19	17
MCU-ISO-212	44	34	19	16
MCU-ISO-213	9	0	4	3
MCU-ISO-214	10	0.2	4	3
MCU-ISO-215	9	0.5	4	4
MCU-ISO-216	9	0.2	4	4

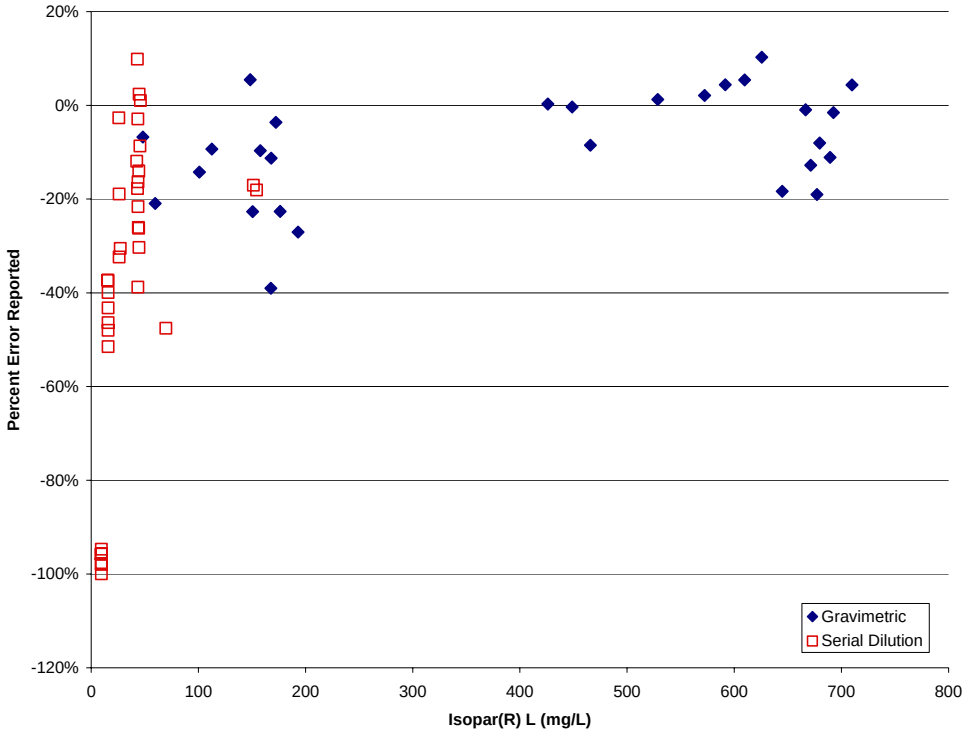

Figure A-1. Difference between Measured and Prepared Isopar® L concentration in Control Samples

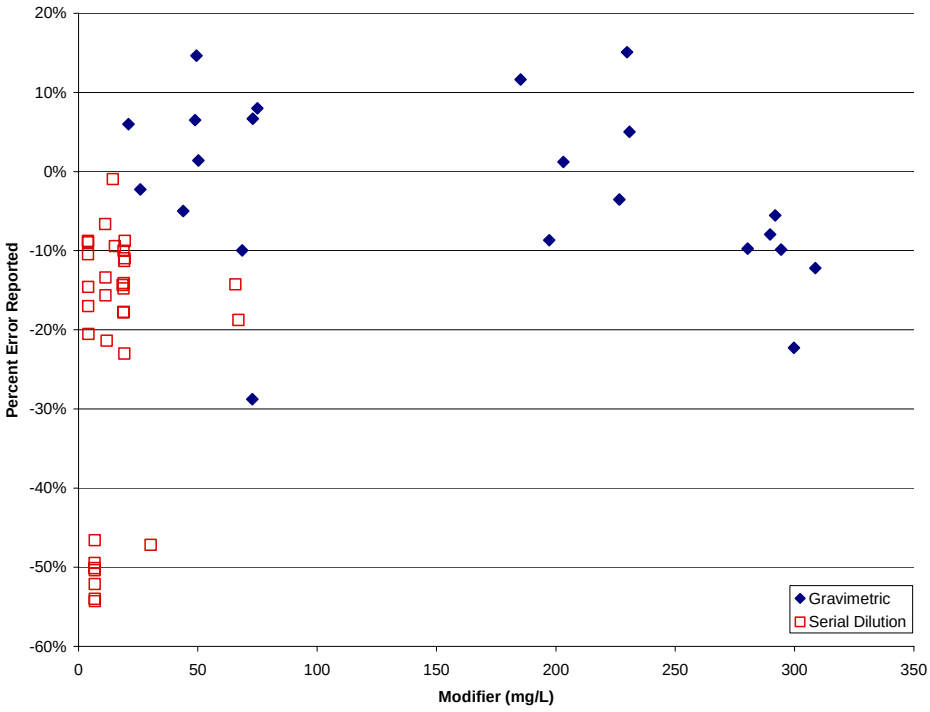

Figure A-2. Difference between Measured and Prepared Modifier concentration in Control Samples

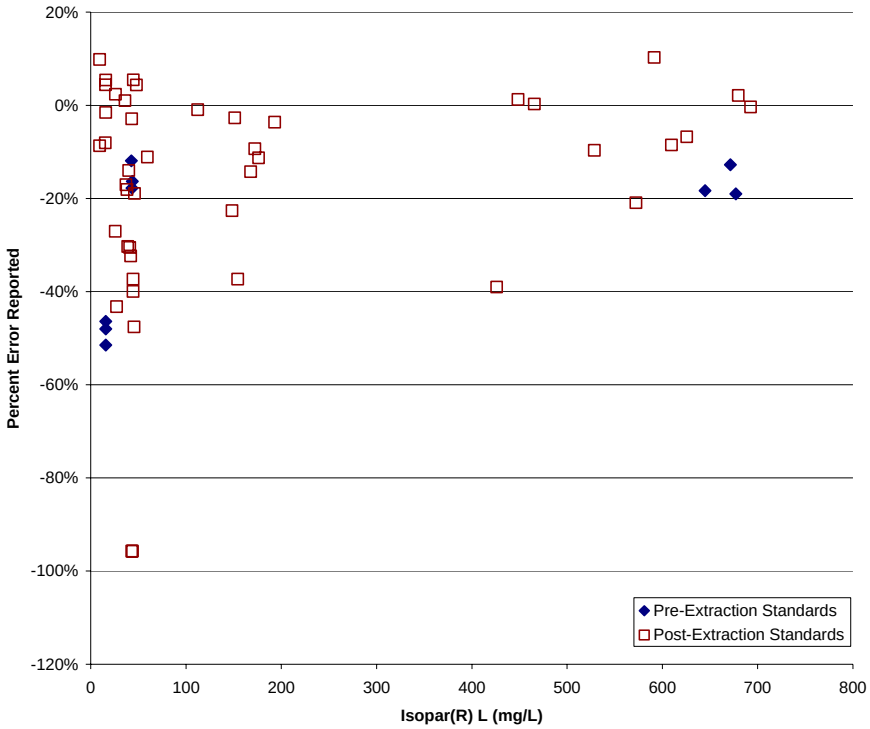

Figure A-3. Difference between Measured and Prepared Isopar® L concentration in Control Samples as a Function of when Internal Standard was Added

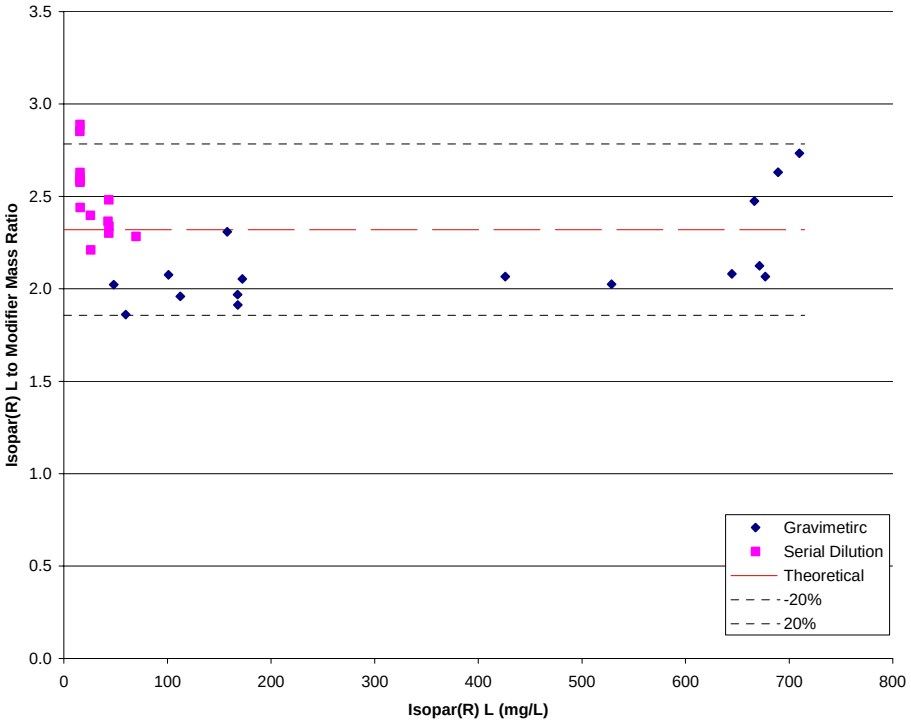

Figure A-4. Ratio of Isopar® L to Modifier in Control Samples