Small Column Testing of Superlig 639 for Removing ^{99}Tc from Hanford Tank Waste Envelope C (Tank 241-AN-107)

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SUMMARY

The current BNFL Inc. flow sheet for pretreating Hanford High-Level tank wastes includes the use of Superlig® 639 (SL-639) in a dual column system for removing technetium-99 (^{99}Tc) from the aqueous fraction of the waste. This sorbent material has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT. This report documents the results of testing the SL-639 sorbent with diluted waste ($[Na^+] \approx 5$ M) from Tank 241-AN-107 (an Envelope C waste, abbreviated AN-107) at Battelle Northwest Laboratories (BNW)

The equilibrium behavior was assessed with batch contacts between the sorbent and the waste. Two AN-107 samples were used: 1) an archived sample from previous testing and 2) a more recent sample collected specifically for BNFL. A portion of the archive sample and all of the BNFL sample were treated to remove Sr-90 and transuranic elements (TRU). All samples had also been Cs decontaminated by ion exchange (IX), and were spiked with a technetium-95m (^{95m}Tc) pertechnetate tracer, $^{95m}TcO_4^-$. The TcO_4^- and total Tc K_d values, assumed equal to the ^{95m}Tc and ^{99}Tc K_d's, respectively, are shown in Table S1. Values are averages of duplicates, which showed significant scatter. The total Tc K_d for the BNFL sample is much lower than the TcO_4^- K_d, indicating that a large fraction of the ^{99}Tc is not pertechnetate.

Table S1. TcO_4^- and Total Tc K_d Values

Sample	Feed conditions	TcO_4^- K_d (mL/g)/ % Removal (1)	Total Tc K_d (mL/g)/ % Removal (2)
Archive AN-107 (3)	Na = 5.70 M Density = 1.256 g/mL ^{99}Tc=55 µCi/L (3.2 mg/L) $NO_3/Tc = 6.8 \times 10^4$	350 / 75%	Not Determined
Treated Archive AN-107	Na = 4.70 M Density = 1.226 g/mL ^{99}Tc=48 µCi/L (2.8 mg/L) $NO_3/Tc = 7.3 \times 10^4$	200 / 66%	Not Determined
BNFL AN-107 (Treated)	Na = 4.84 M Density = 1.241 g/mL ^{99}Tc=57 µCi/L (3.3 mg/L) $NO_3/Tc = 5.4 \times 10^4$	500 / 80%	22 / 17%

1) Assumed equal to the ^{95m}Tc K_d.
2) Assumed equal to the ^{99}Tc K_d.
3) Archive and Treated Archive ^{99}Tc and NO_3/Tc estimated from Blanchard et al. (1997) and Hallen, Bredt, Brooks and Jagoda (2000)

Small column testing was performed using a dual column system. The columns were arranged in series, and each contained a 4.7 mL bed, length/diameter = 6.0. The columns were used in a previous test, but the order was reversed. As a result of the previous test, the lead column at the start of this test was partially loaded and the lag column had been loaded and eluted to $C/C_0(^{99}Tc)$= 1%. Approximately 0.8 L of the BNFL AN-107 sample was processed in this column test. A $^{95m}TcO_4^-$ tracer was used to follow the progress of the test. After elution of the first column with warm (53.5°C) de-ionized water, a second set of tests was performed to investigate the impact on subsequent loading of residual Tc on the column. A Tank AN-107 waste feed simulant and actual

Tank AN-107 waste feed were run through the eluted column to determine the amount of the residual ^{99}Tc that would bleed from the column, and to get an estimate of the fraction of extractable Tc in the waste.

A summary of performance measures from the small column tests is shown in Table S2. The λ value is the number of column volumes processed when C/C_0 reaches 50%, and is a measure of the effective capacity of the resin. The λ value for the $^{95m}TcO_4^-$ tracer is approximately half that observed in a previous test of Tank 241-AW-101 waste. The initial DF's for the $^{95m}TcO_4^-$ tracer for the 1st and 2nd columns were much lower than the corresponding values for AW-101 (180 and 433, respectively), reflecting the partial loading of the columns from the previous test.

Results for ^{99}Tc were dramatically different. The concentration of ^{99}Tc in the effluent during "loading" was the same as or higher than in the feed, indicating that ^{99}Tc was actually being eluted during this phase. Thus λ values could not be calculated, and the initial DF's were less than or equal to 1. Approximately 170 bed volumes of the diluted AN-107 feed was processed through the SL-639 columns. The initial ^{99}Tc C/C_0 for the lead column was approximately 144%, and dropped to 126% at the end of the run. The initial ^{99}Tc C/C_0 for the lag column was approximately 98%, and rose to 127% at the end of the run. The difference between the ^{99}Tc and $^{95m}TcO_4^-$ results is attributed to the high concentration of non-pertechnetate Tc in AN-107 feed. The concentration of ^{99}Tc in the effluent composite is 59.3 μCi/L (3.47 mg/L), almost 50% higher than the expected average maximum allowed concentration.

Table S2. Summary of Performance Measures from Small Column Tests

Feed	Column Effluent Sampled	Flow rate, BV/hr	λ		Comp DF, $^{95m}TcO_4^-$	Comp DF, ^{99}Tc	Initial DF	
			$^{95m}TcO_4^-$ Tracer	^{99}Tc			$^{95m}TcO_4^-$ Tracer	^{99}Tc
BNFL AN-107	Col 1	6.1	125	(Eluted)	NA	NA	9.9	0.7
BNFL AN-107	Col 2	6.1	NA	(Eluted)	22	0.96	74	1
AN-107 Unspiked Simulant	Col 1, Post-Elution	6.3	NA	NA	NA	NA	20 (rel. to AN-107)	48 (rel. to AN-107)
BNFL AN-107	Col 1, Post-Elution	6.1	NA	NA	NA	NA	19	1.3

Only the lead column was eluted. The peak ^{99}Tc concentration was 27 times the ^{99}Tc concentration in the feed, and was found in the 2nd bed volume. After this the elution proceeded very slowly, requiring 46 BV of eluant (DI water at 50°C) for the ^{99}Tc concentration to drop to $C/C_0 = 0.05$. Tank AN–107 waste feed simulant containing no Tc was subsequently passed through the eluted column. The levels of ^{99}Tc and ^{95m}Tc found in the effluent were 2.5% and 5%, respectively, of that in the actual feed. Actual AN-107 waste feed was then passed through the column, and the ^{99}Tc was found to immediately break through with a C/C_0 of almost 80%. The ^{95m}Tc tracer immediately broke through with an initial C/C_0 of about 5%. Washing of the loaded columns with 0.1 M NaOH was found to elute Tc slightly.

TERMS, SYMBOLS, AND ABBREVIATIONS

AEA	alpha energy analysis
ALARA	as low as reasonably achievable
BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd.
BV	Bed Volume
DF	decontamination factor
DL	detection limit
EQL	estimated quantitation level
GEA	gamma energy analysis
HLRF	High Level Radiation Facility
IC	ion chromatography
ICP	inductively coupled plasma/atomic emission spectrometry
ICP-MS	inductively coupled plasma/mass spectrometry
λ	lambda; the number of BV processed at 50% breakthrough
L/D	ratio of bed height to bed diameter
MDL	method detection limit
MRQ	minimum reportable quantity
RPL	Radiochemical Processing Laboratory
SAL	Shielded Analytical Laboratory
TC	total carbon
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic

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1.0 INTRODUCTION

The current BNFL Inc. flow sheet for pretreating Hanford High-Level tank wastes includes the use of Superlig® 639 (SL-639) in a dual column system for removing technetium-99 (^{99}Tc) from the aqueous fraction of the waste. This material has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT.

This report documents the results of testing the SL-639 sorbent with Hanford tank waste samples from Tank 241-AN-107 (the 241 prefix, which is common to all Hanford tanks, will not be used hereafter). Batch contacts were performed with small samples to assess equilibrium behavior and to plan small column tests. A 0.8 L sample of diluted waste (@ 4.8 M Na) was processed in a small column test. The samples had been previously treated to reduce the concentrations of Sr-90 and transuranic (TRU) components and filtered to remove particulates (Hallen, Brooks and Jagoda, 2000; Hallen, Bredt, Brooks and Jagoda, 2000), and processed by IX to remove cesium-137 (Kurath et al., 2000). The Tc removal process steps tested include loading, feed displacement, water rinse, elution, resin regeneration, and subsequent reloading of the eluted column. The Tc removal column system was used in a previous test of Tc removal from Tank AW-101 waste (Envelope A; Blanchard, Kurath and Bontha, 2000); the lead column in this test was already partially loaded from the previous test.

Most of the ^{99}Tc in the liquid fraction of Hanford tank wastes is expected to be present as the pertechnetate anion (TcO_4^-). Analyses of candidate LAW Envelope A and B feed samples indicate that 95% (or greater) of the ^{99}Tc is indeed present as TcO_4^-. However, analyses of candidate LAW Envelope C feed samples (e.g., Tank 241-AN-102 and Tank 241-AN-107 samples) indicates that only 15% to 20% of the ^{99}Tc is present as the TcO_4^-. The BNFL Inc. Waste Treatment Plant (WTP) project uses a sorbent material that selectively removes the TcO_4^- from LAW solutions. Therefore, the amount of Tc separated from LAW Envelope A and B feeds can approach 95% and that from Envelope C feed only ~20%. In order to comply with the ILAW glass product acceptance criteria by averaging 80% removal of the Tc present in the feed, the amount of Envelope C processed by the WTP will be limited. Similarly, the ^{99}Tc concentration in the ILAW glasses produced from Envelope C solutions are expected to exceed 0.1 Ci/m^3, but the average concentration of Envelope A, B, and C glasses together will be less than 0.1 Ci/m^3.

The objectives of this work were to:

- Demonstrate the ^{99}Tc decontamination of Envelope C (Tank AN-107) and provide a technetium-decontaminated sample for downstream process testing (i.e. corrosion testing, Low Activity Waste (LAW) melter feed testing and LAW vitrification).
- Demonstrate the effectiveness of all SL-639 process steps including loading, feed displacement, DI water washing, elution, resin regeneration, and subsequent reloading.
- Obtain process performance data for SL-639 at conditions different than those previously tested.
- Obtain process performance data for loading of SL-639 by AN-107 waste following partial loading by AW-101 waste.
- Investigate sorbent/waste chemistry, particularly the differences in the interaction of TcO_4^- and non-pertechnetate Tc with the sorbent.
- Investigate the potential for exchanger fouling.

2.0 EXPERIMENTAL

2.1 Tc Removal Column System

A schematic of the Tc Removal column system is shown in Figure 2.1. The system, which is mounted in a radiological fume hood, consists of 2 small columns containing the sorbent resin, a small metering pump, 3 valves, a pressure gauge and a pressure relief valve. The pump inlet tube was manually switched between the waste feed and various process solutions. Valves 1, 2 and 3 are three-way valves that can be turned to a flow position, a sample position or a no-flow position. Valve 1 is placed at the outlet of the pump and is used to eliminate air from the system, purge the initial volume of the system or isolate the columns from the pump. Valves 2 and 3 are primarily used for obtaining samples and may also be used to isolate the columns from the rest of the system.

The columns are Kontes Chromaflex chromatography columns made of glass with adjustable plungers on the bottom and the top. The inside diameter of the columns is 1.0 cm which corresponds to a volume of 0.785 mL per cm of length. The columns are jacketed with a clear plastic to provide temperature regulation and a safety shield. The connecting tubing is a polyfluorinated plastic with 1/8-in OD and 1/16-in ID. The columns are connected in series with the first column referred to as the lead column and the second column referred to as the lag column. The pump is a FMI piston pump with the flow rate controlled from outside of the hood with a FMI stroke rate controller. The pump was calibrated with the stroke rate controller and can provide pumping rates of approximately 0.5-50 mL/hr. The volume actually pumped is determined using the mass of the fluid and the fluid density.

The pressure relief valve is set at 40 psi, which is below the maximum operating pressure for the columns. The pressure indicated on the pressure gauge remained below 5 psi during the run. The total holdup volume of the system was estimated to be 14 mL (3 BV) with the holdup volume to valve 1 being approximately 2 mL (0.4 BV). The columns were used in a previous test of Tc removal from AW-101 waste (Blanchard, Kurath and Bontha, 2000), but the order was reversed. As a result, the lead column at the start of this test was partially loaded and the lag column had been loaded and eluted to $C/C_0(^{99}Tc)= 1\%$, where C and C_0 are the concentrations of ^{99}Tc in the effluent and feed, respectively.

2.2 SL-639 Resin and Bed Preparation

The SL-639 resin was supplied by IBC Technologies Inc. (American Fork UT) from batch # 980624001DC. The resin consists of a proprietary organic compound (ligand) attached to spherical styrene beads. The mean diameter of the beads (D_p) is reported by the manufacturer as 0.5 mm. SL-639 functions by extracting the sodium pertechnetate salt-pair ($NaTcO_4$) from either acidic or basic solutions.

The resin was used for batch contacts with no pre-conditioning. The resin and beds were prepared for the column testing in the previous test of Tc removal from AW-101 (Blanchard, Kurath, and Bontha, 2000). The resin was slurried into the columns in DI water. The bed height was 6.0 cm, giving a bed volume of 4.7 mL, and a length-to-diameter ratio (L/D) of 6. The number of beads per column diameter (1 cm) is approximately 20:1, which is at the low end of the range (20 –30) commonly assumed necessary to prevent wall affects during small-scale column testing. (The column size was chosen based on the bed size required for 50% breakthrough given the amount of sample available, and the desired L/D.) The columns were prepared for loading by flushing them with 14.4 BV (67.5 mL) of 1 M NaOH. This was done primarily to flush water from the beds to prevent precipitation of solids on introduction of the feed.

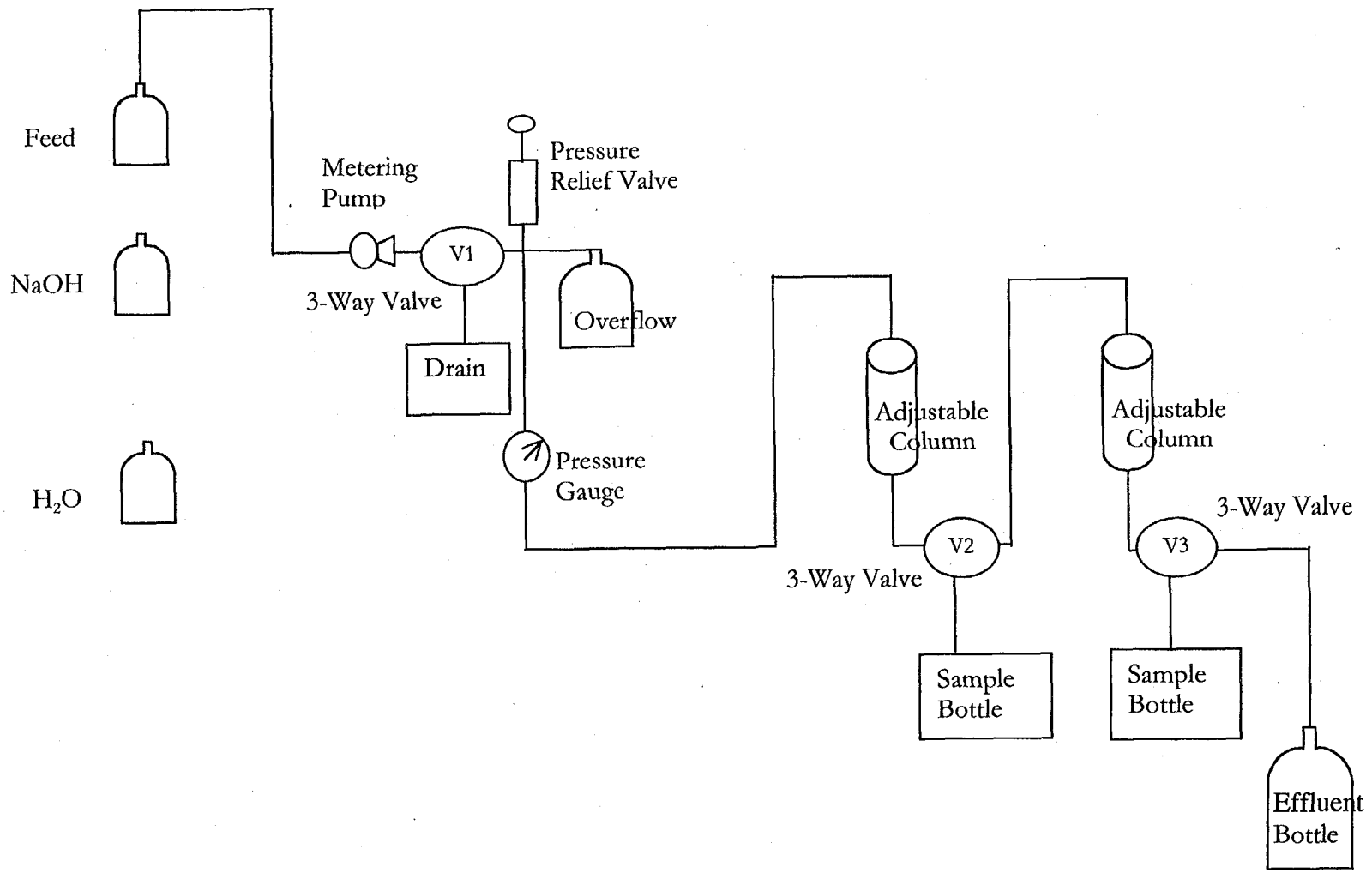

Figure 2.1. Technetium Removal Column System

2.3 Feed Preparation

The first set of batch contacts used a sample of waste from Tank AN-107 (Envelope C) archived after previous testing (Hendrickson et al., 1997). The original sample was removed from Tank AN-107 as a grab sample in January 1997. It was diluted with 0.53 M NaOH to approximately 5 M Na and 0.126 M OH. Particulates were allowed to settle, and the decantate was then Cs decontaminated using a crystalline silicotitanate ion exchanger, IE-911 (UOP). Details may be found in Hendrickson (1997). Several liters of this waste were received in six 1 L bottles in the Shielded Analytical Laboratory (SAL) at the Radiochemical Processing Laboratory (RPL) on May 23, 1997. Several of these bottles were loaded into the SAL hot cells later that summer for archival. This archived material was transferred to the High Level Radiochemistry Facility (HLRF) (also in the RPL) in July 1999, where the [OH^-] of a subsample was adjusted to approximately 1 M. The subsample was then spiked with $Sr(NO_3)_2$ and $NaMnO_4$ to precipitate native Sr and TRU. Development of this Sr/TRU removal method is reported in Hallen, Bryan and Hoopes (2000), and treatment of the archived AN-107 is reported in Hallen, Brooks and Jagoda (2000). Portions of the untreated and treated samples were transferred to a radiological fume hood for batch contact testing. The untreated solution was opaque dark brown. The supernate of the treated sample was clear light brown, and a fine-grained black precipitate had settled to the bottom of the vial. Only the clear supernate was used for the contacts of the treated material. The "untreated archive AN-107" and "treated archive AN-107" samples referred to in this report are subsamples of samples MN-21 and MN-22, respectively, described in Hallen, Brooks and Jagoda, (2000).

Approximately 0.3 µCi of ^{95m}Tc ($t_{1/2}$ = 61 days, decays to stable ^{95}Mo) was added as ammonium pertechnetate (NH_4TcO_4) in 1 M ammonium hydroxide (NH_4OH) to each of the archive AN-107 samples to act as a tracer for following the removal of TcO_4^-. A 10-µL aliquot of the tracer solution was added to each 15 mL sample. The amounts of NH_4TcO_4 and NH_4OH added to the waste are not expected to significantly change the physical or chemical properties of the waste. The amount of ^{95m}Tc added to the waste (1.4E-10 moles) is small relative to the ^{99}Tc already in each 15 mL waste sample (approximately 4.8E-7 moles, based on data in Blanchard et al., 1997), and is not expected to change either the TcO_4^- K_d or the effective total Tc K_d. The additional ^{95}Mo produced by ^{95m}Tc decay is also not expected to change the waste properties. Batch contacts using these samples were performed September 27 - October 1, 1999.

A second sample of waste from Tank AN-107 (Envelope C) was received in the HLRF during the 4th quarter of 1998. The homogenization, dilution, and subsampling are described in Urie et al. (1999). This sample was processed to remove Sr-90 and TRU in September 1999 in the same fashion as the archive sample. The [OH^-] of the sample was adjusted to approximately 1 M and it was spiked with $Sr(NO_3)_2$ and $NaMnO_4$ to precipitate native Sr and TRU. In addition, it was filtered in a single tube cross-flow filter to remove entrained solids using a 0.1 micron sintered metal Mott filter. A complete description may be found in Hallen, Bredt, Brooks and Jagoda (2000). The clarified AN-107 sample was then transferred from the HLRF to the SAL hot cells for Cs IX testing using Superlig®644. Following Cs IX, described in Kurath et al. (2000), the feed was removed from the hot cell and transferred to a radiological hood containing the Tc removal column system.

A 100-mL aliquot of this feed was set aside, and then approximately 0.5 mCi of ^{95m}Tc was added to the remainder (approx. 1.5 L), to act as a tracer to follow the progress of TcO_4^- removal. The tracer was added as NH_4TcO_4 in 1 M NH_4OH. The amounts of NH_4TcO_4 and NH_4OH added to the waste (4E-5 g and 0.875 g) are not expected to significantly change the physical or chemical properties of the waste. The amount of ^{95m}Tc added to the waste (2.2E-05 g) is small relative to the ^{99}Tc already in the waste sample (8.4E-03g), and is not expected to change either the TcO_4^- K_d or the effective total

Tc K_d. The additional ^{95}Mo produced by decay is also not expected to change the waste properties. Batch contacts with this feed were performed November 8 - 12, 1999. Column tests with this feed were performed November 4 - 11, 1999. The feed is referred to in this report as the "BNFL sample" or "BNFL feed" or "Tc removal feed." The density of this feed was determined with a 25 mL volumetric flask and a 4-place analytical balance.

Based on previous work (Blanchard, Kurath and Bontha, 2000; Blanchard, Lawrence, Kurath and Golcar, 2000), the ^{95m}Tc pertechnetate tracer is expected to remain as TcO_4^- after addition to the waste samples, and not form ^{95m}Tc non-pertechnetate species. Because the amount of the ^{95m}Tc tracer added to the samples is very small relative to the amount of ^{99}Tc already present (less than 1/100th for the archive samples and less than 1/1000th for the BNFL sample) the added tracer is not expected to change the observed K_d's. It acts only as an indicator of the TcO_4^- K_d.

2.4 Batch Contact Procedure

The batch K_d tests were performed at a phase ratio of approximately 100 (liquid volume to exchanger mass), with 0.05 g of exchanger contacted with 5 mL of solution. The exchanger mass was determined to an accuracy of 0.0001 g. The waste volume was transferred by pipette and the actual volume was determined from the mass difference (0.0001 g accuracy) and the solution density. Agitation was provided by a back-and-forth shaker set at 180 cpm for approximately 96 hours. The temperature was not controlled, and varied between approximately 22.5°C and 25.0°C over the course of the 4-day contacts.

The archive AN-107, treated archive AN-107, and BNFL AN-107 samples were tested. All K_d measurements were made in duplicate and blank samples were used to determine the initial concentration of the species of interest. All solutions were analyzed by gamma spectroscopy to determine the $^{95m}TcO_4^-$ concentrations and K_d's. The ^{99}Tc concentrations in the BNFL samples were determined by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS). The concentrations of various components of the BNFL feed were determined as described in Section 2.5 below.

The batch distribution coefficient, K_d (with units of mL/g), was determined using the following relationship;

$$K_d = \frac{(C_0 - C_1)}{C_1} * \frac{V}{M * F}$$

where C_0 and C_1 are the initial and final concentrations, respectively, of the species of interest (i.e., $^{95m}TcO_4^-$ and ^{99}Tc), V is the volume of the liquid sample (mL), M is the mass of resin used for the contact (g), and F is the mass of a sample of dried resin divided by its mass before drying. The resin was sampled for F factor determination at the same time resin samples for the contacts were tared in order to minimize changes due to atmospheric humidity.

2.5 Column Test Procedure and Conditions

The experimental conditions for each process step of the column test are shown in Table 2.1. In general the flow rates were maintained as close as possible to the recommended values but in some steps the total volume of the process solutions was increased in an attempt to ensure adequate flushing of the system, as for instance, in the column preparation with 1.0 M NaOH. Only the lead column was eluted. The bed preparation, loading, feed displacement and regeneration steps were conducted by passing these solutions through both resin beds connected in series. The DI water rinse,

elution and elution rinse were conducted on only the lead column, as were the follow-on tests to reload the eluted column.

Table 2.1. Experimental Conditions

Process step	Solution	Total Vol., BV (mL)	Flow Rate, BV/hr (mL/hr)	Time (hr)
Column prep	1.0 M NaOH	18 (83)	11 (50)	1.67
Loading	AN-107 Feed	170.5 (801.3)	6.1 (28.8)	28.1
Feed displacement	0.1 M NaOH	7.5 (35.1)	3.0 (14.0)	2.5
Rinse (Lead Column)	DI water	2.1 (9.7)	3.1 (14.5)	0.7
Elution (Lead Column)	DI water @ @ 53.5°C	46.1 (216.8)	1.0 (4.8)	45.7
Eluant rinse (Lead Column)	DI water	4.1 (19.2)	6.1 (28.9)	0.7
Regeneration	1.0 M NaOH	6.4 (30.2)	6.4 (30.2)	1.0
Simulant Loading (Lead Column)	AN-107 Simulant (No Tracer)	13.4 (62.8)	6.3 (29.4)	2.1
Feed Displacement (Lead Column)	0.1 M NaOH	4.9 (23.0)	3.7 (17.2)	1.3
Loading (Lead Column)	AN-107 Feed	29.8 (139.9)	6.1 (28.8)	4.9
Regeneration (Lead Column)	1.0 M NaOH	4.6 (21.8)	3.5 (16.4)	1.3

Elution was performed using DI water at 53.5°C. A recirculating water bath was used to circulate heated water through the column jacket, and the feed tube to the column was taped (approx. 12" length) to the much larger tube carrying warm water to the jacket. Although faster than in a previous test using room temperature 0.5 M nitric acid as the eluant, elution was still relatively slow. It was halted when C/C_0 for the $^{95m}TcO_4^-$ tracer reached 13%, instead of the target 1%, in order to limit the cost of the run, as per discussion with the BNFL Pretreatment Technical Manager (M.E. Johnson). To determine the extent to which the residual Tc on the column would affect the next loading cycle, simulant without the tracer was passed through the column after regeneration. The BNFL actual AN-107 feed (with tracer) was then passed through the column for the same reason and to assess the fraction of pertechnetate Tc.

The adjustable plungers at the top of each column were used to minimize the volume of solution above each of the resin beds. The bed volumes changed less than 0.2 cm (0.16 mL or 3.3 %) during the run. The height of liquid above the beds was kept to less than 0.5 cm (0.4 mL).

The sampling and analysis protocol is shown in Table 2.2. The TcO_4^- C/C_0 was determined in all samples by counting the ^{95m}Tc gamma emission at 205 KeV with a portable gamma spectrometer. The C/C_0 were determined by taking the ratio of the peak areas of the feed and the effluent samples. The feed sample was recounted periodically (at least every 24 hours) to minimize the effects of the ^{95m}Tc decay. This method allowed near real time analysis of the behavior of TcO_4^- in the samples. The

response time was limited by the rate at which samples could be removed from the hood. The ^{99}Tc activities were determined in selected samples by ICP-MS after the run was concluded. ICP was used to determine sodium and other elemental concentrations. Free hydroxide ion concentrations were determined by titration with standard hydrochloric acid solutions. Total inorganic carbon was determined by evolution of CO_2 from an acidified, heated sample, and total organic carbon was determined by evolution of CO_2 from the same sample after subsequent addition of persulfate with heating. A sample of the feed was analyzed for ^{99}Tc by ICP-MS, and for nitrate by IC. Data on other analytes collected during these analyses are also reported.

Table 2.2. Sampling Interval and Analyses

Process Step	Lead Column BV	Lag Column BV	Approx. Sample Vol. (mL)	Analyses
Column prep	-	-	-	-
Loading	Every 10 BV	Every 20 BV	2	ICP-MS
Feed displmt	-	Every 1 BV	5	ICP, ICP-MS, OH
DI water rinse	Every 1 BV	-	5	ICP, ICP-MS, OH
Elution	Every 1 BV	-	5	ICP-MS
Eluant rinse	Every 1 BV	-	5	ICP-MS
Regeneration	-	1 composite	30	ICP, ICP-MS, OH
Sim. Loading	Every 1 BV	-	5	ICP-MS
Feed displmt	Every 1 BV	-	5	ICP-MS
Loading	Every 1 BV	-	5	ICP-MS
Regeneration	Every 1 BV	-	5	ICP-MS
Composite Samples				
Effluent (Initial Loading)	-	1 composite	5	ICP-MS
Eluate	1 composite	-	22	ICP, TIC, TOC, OH ICP-MS, Radchem

During the loading phase, the treated effluent was collected in an effluent bottle except for the small (2 mL) samples that were taken. A composite sample from the effluent bottle was analyzed for ^{99}Tc by ICP-MS. The regenerate was collected in one 30 mL batch. A sample was analyzed for ^{99}Tc, Na and OH^-. The rest of the samples were collected in approximately 1 BV aliquots. A composite sample of the eluate was prepared and submitted for ICP-MS, ICP, TOC, TIC, OH and various radiochemical analyses (total alpha, Sr-90, GEA).

The λ value, which is the number of column volumes of feed processed when $C/C_0 = 50\%$, may be predicted from a batch contact K_d by the relationship $\lambda = K_d * \rho_B$, where ρ_B is the bed density of the resin in the waste. Since the SL-639 resin did not swell or shrink in any of the solutions processed, ρ_B is equal to its dry bed density, 0.49 g/mL (Kurath et al., 1999). The experimental λ values from breakthrough curves were determined from a linear fit of the breakthrough data plotted on a linear-linear scale. Initial DF's were calculated as C_0/C_1, where C_1 is the concentration in the first sample collected from each column.

3.0 RESULTS AND DISCUSSION

3.1 Feed Composition

The compositions of the treated and untreated archive AN-107 samples used for batch contacts are shown in Table 3.1. A limited number of analyses were performed, as testing of these samples was primarily in preparation for testing the BNFL AN-107 sample. The ^{99}Tc concentration and the $NO_3^-/^{99}Tc$ mole ratio in the untreated archive sample are estimated from previous work with this material (Blanchard et al., 1997). The ^{99}Tc concentration and the $NO_3^-/^{99}Tc$ mole ratio in the treated archive sample are estimated based on dilution of, and addition of NO_3 to, the untreated sample during treatment (Hallen, Brooks and Jagoda, 2000).

The composition of the BNFL Tc removal feed is also shown in Table 3.1. The concentrations of Na and Al were determined by ICP-AES and the values presented in Table 3.1 are averages of the analyses for the filtrate from the Sr/TRU removal process, cesium IX feed, Tc removal feed, Tc removal effluent and sulfate removal feed. The concentrations of K, Cr and P were also determined by ICP and are averages of analyses for the filtrate from the Sr/TRU removal process and the cesium IX feed. Anionic forms for Al, Cr and P are assumed on the basis of waste chemistry. Additional anion concentrations were determined by IC analyses of the feed and effluent for the Tc removal tests. This compilation is assumed valid since the non-radioactive cations and anions indicated are not greatly affected by the Cs and Tc removal processes. The hydroxide (OH^-) concentration is estimated based on data in Urie et al. (1999) corrected for chemical additions during the Sr/TRU precipitation. The feed had a light brown color that has been attributed to the presence of organic compounds. The ^{99}Tc concentration was determined by ICP-MS of the Tc removal feed. Data from individual analyses may be found in Appendix A, Kurath et al. (2000), and Hallen, Bredt, Brooks and Jagoda (2000).

The total anion normality, 4.62 N, is lower than the total cation normality, 4.86 N. This is probably mostly due to the presence of anionic complexants for which analyses were not performed. For example, previous analyses of an AN-107 tank waste grab sample (Campbell et al., 1998) indicate concentrations of acetate and formate of 0.13 mole/kg and 0.12 mole/kg, respectively. Data in the same report indicate concentrations of nitroso-iminodiacetate (NIDA), nitrilotriacetate (NTA), citrate, N-(2-hydroxyethyl)ethylenediaminetriacetate (HEDTA), ethylenediaminetriacetate (ED3A), and ethylenediaminetetraacetate (EDTA) ranging from 1.7×10^{-3} moles/kg (HEDTA) to 3.1×10^{-2} moles/kg (citrate). All of these complexants are expected to be in their anionic form in the waste, and carry full anionic charges of -2 to -4, resulting in normalities 2 to 4 times their observed molarities. The sum of the anionic normality of these species is 0.48 equivalents per kg waste. (See appendix for details.) Campbell et al. (1998) claim 86% of the TOC in the sample was accounted for by their analyses, so there may be more anionic complexants that were not discovered. The feed sample used in this study was treated with permanganate and diluted relative to the sample analyzed by Campbell et al. (1998), so a direct comparison is not appropriate, but the data indicate that the complexants and complexant fragments present in the waste represent a significant contribution to the anionic concentration.

Table 3.1. Composition of Envelope C (AN-107) Tc Removal Feeds

	Archive AN-107 (Untreated)	Treated Archive AN-107	BNFL AN-107 (Treated)
Cations, M			
Na^+	5.70	4.70	4.84
K^+	2.14E-2	1.90E-2	1.90E-2
Anions, M			
AlO_2^- (1)	6.19E-3	4.82E-3	8.7E-2
Cl^-	N. D.	N. D.	1.4E-2
CO_3^{2-}	N. D.	N. D.	5.7E-1
CrO_4^{-2} (1)	1.37E-3	8.20E-4	8.81E-4
NO_2^-	N. D.	N. D.	6.2E-1
NO_3^-	N. D.	N. D.	1.82
OH^-	0.87	0.74	8E-1 (2)
PO_4^{-3} (1)	7.46E-3	6.49E-3	1E-2
SO_4^{-2}	N. D.	N. D.	4.2E-2
Oxalate	N. D.	N. D.	1.6E-2
^{99}Tc and Competing Ion Ratios			
^{99}Tc, µCi/L (mg/L) (ICP-MS)	55 (3.2) (3)	48 (2.8) (3)	57.1 (3.34)
$NO_3^-/^{99}Tc$ mole ratio	6.8E+4 (3)	7.3E+4 (3)	5.39E+4
$CrO_4^{-2}/^{99}Tc$ mole ratio	N. D.	N. D.	26.1
Solution Density, g/mL	1.256	1.226	1.241

1) Determined by ICP. Anionic form is assumed on the basis of waste chemistry.
2) Estimated. See text.
3) Estimated using data from Blanchard et al. (1997) and Hallen, Brooks and Jagoda (2000).
4) N. D. indicates that the concentration of this analyte was not determined.
5) The raw analytical results may be found in the appendix.

A series of analyses were also performed to determine the fraction of ^{99}Tc present as TcO_4^- in the archive sample, in the treated archive sample, and in the BNFL sample. The samples were prepared for counting of ^{99}Tc in three different ways: 1) extraction of TcO_4^- (no oxidation), 2) extraction of TcO_4^- following oxidation by Ce(IV)/8 M HNO_3/heating and 3) extraction of TcO_4^- following oxidation by potassium permanganate ($KMnO_4$). The procedures are given in more detail with the raw data in the appendix. Results are shown in Table 3.2.

The treated archive sample used for this analysis was processed differently than that used for the batch contacts. The target concentrations of OH^-, $Sr(NO_3)_2$ and $NaMnO_4$ were slightly lower. Details may be found in Hallen, Brooks and Jagoda (2000). Results in Table 3.2 for the "Archive AN-107" are from the samples designated MR-01 and MR-02 in that report, except they were not acid digested prior to the

indicated analyses. MR-02 is the same as MR-01, but was filtered prior to analysis. Results were within error for these two samples, as well as for a duplicate of MR-01, so all three sets were averaged. Results for "Treated Archive AN-107" are from sample MR-03, prior to acid digestion. Note that MR-03 was diluted relative to the untreated archive samples, so the total Tc concentration should be lower, as observed. The difference is not completely accounted for by the dilution; the remaining Tc loss is probably due to precipitation of some of the non-pertechnetate Tc with the Sr and TRU. Radiochemical analysis of the precipitated solids for the archive sample was not performed, but analysis of precipitated solids from the BNFL AN-107 sample did show significant Tc-99 present (Hallen, Bredt, Brooks and Jagoda, 2000). The "BNFL AN-107" results are from a sample of the same material used for the batch contacts and the column test reported herein. The raw data for all samples may be found in the appendix.

Table 3.2. TcO_4^- and Total Tc Concentrations in Envelope C (AN-107) Tc Removal Feed

	Archive AN-107	Treated Archive AN-107	BNFL AN-107 (Treated)
TcO_4^-, µCi/L (No Oxidation)	16.5	17.4	13.0
Total Tc, µCi/L (Ce(IV) Oxidation)	53.0	43.5	36.3
TcO_4^- Percentage	31%	40%	36%
Total Tc, µCi/L ($KMnO_4$ Oxidation)	54.6	43.5	38.6
TcO_4^- Percentage	30%	40%	34%

Results using the two different oxidation methods are consistent. The absolute concentration of TcO_4^- in the treated archive sample is slightly higher than in the untreated sample, suggesting that some of the non-pertechnetate was oxidized to TcO_4^- by the permanganate added for the Sr/TRU precipitation. Combined with the apparent precipitation of non-pertechnetate Tc, the percentage of TcO_4^- increases significantly due to the treatment. The lower TcO_4^- percentage found in the (treated) BNFL AN-107 relative to the treated archive sample is consistent with the observation that the non-pertechnetate slowly converts to TcO_4^- if the sample is not located in a high radiation field (i.e., after removal from the tank; Schroeder, 2000). The archive sample was removed from Tank AN-107 approximately 18 months before the BNFL sample.

The total ^{99}Tc concentrations determined for the BNFL AN-107 sample by the two oxidation methods are approximately 34% lower than that determined by ICP-MS (57.1 µCi/L; see Table 3.1 above). Recovery of a matrix spike added to duplicate samples was above 93% in all cases, so the discrepancy is evidently not due to losses during the analytical procedures. It's possible that even these vigorous oxidations did not recover all the ^{99}Tc. In a previous study, the same Ce(IV) oxidation recovered 82% of the Tc found by ICP-MS (Blanchard et al., 1997).

It's also possible that the concentration determined by ICP-MS is higher than the actual. This technique determines the mass of material at a given mass-to-charge ratio (m/z). Ruthenium-99 (^{99}Ru) is one of seven stable, naturally occurring Ru isotopes. If present, it will appear at the same m/z as ^{99}Tc. Examination of the mass spectrum indicated that there were some ruthenium (Ru) isotopes present, but they did not exhibit a natural abundance distribution, so they were assumed to be fission products. Since fission produces only a vanishingly small amount of stable ^{99}Ru, all signal at m/z=99 was assumed to be ^{99}Tc. If some natural Ru was present from impurities in another chemical added during processing,

however, there would be ^{99}Ru present that was counted as ^{99}Tc. An error in the ^{99}Tc activity due to this source is expected to be very small, but it allows us to assume that the activity determined this way is an upper bound to the total Tc present. If this higher value of the total Tc is used, the fraction of TcO_4^- present is calculated to be 23%. So taking the true value to be intermediate between this value and those given in Table 3.2, the fraction of TcO_4^- is given as (29 ± 10)%, where the error estimate is conservative.

3.2 Batch Contacts

Results of the batch contacts are shown in Table 3.3. (The complete data table may be found in the appendix.) The Sr/TRU precipitation used for the treated archive sample and the BNFL sample diluted the ^{99}Tc relative to the archive sample. The use of permanganate (MnO_4^-) for the precipitation is expected to have oxidized various organic and inorganic components. These changes are expected to result in different observed TcO_4^- K_d's for the three different solutions. However, the relatively large differences between the archive duplicates and between the BNFL duplicates makes it difficult to discern these true differences. The large differences between duplicates are attributed to poor resin/solution contact. This would prevent true equilibrium, and is suspected because transfer of resin beads out of the liquid and onto the sides of the vials or inside of the caps has been previously observed when the resin floats in or on the solution.

At least some of the resin floated in all three solutions. The largest fraction of the resin floated in the treated archive sample, with almost all the resin beads at the surface and the remainder suspended in the liquid. In the untreated archive sample and the BNFL sample most of the resin was suspended in the liquid, with small amounts of resin at the bottom and at the surface. It is not clear why a greater percentage of the resin floated in the treated archive sample (density = 1.226) than in the untreated archive sample (density = 1.256) and the BNFL AN-107 sample (density = 1.241). Vials were checked once or twice daily for resin on the vial sides and cap. None was observed, but it may have occurred between inspections. Transfer of relatively few resin beads to the sides or cap may significantly skew the results due to the small sample of resin used for the contacts. The small resin sample was driven by the required phase ratio and the small amounts of sample available.

The large difference between TcO_4^- removal (measured by the $^{95m}TcO_4^-$ tracer) and total Tc removal (measured by ^{99}Tc) in the BNFL sample is easily discerned. The lowest TcO_4^- removal was 64.5%, while the highest total Tc removal was 22.3%. The observed total Tc removal is expected to be an average of the removal of each different Tc species, weighted by the molar amounts of each species. The difference between the TcO_4^- and total Tc results therefore indicates a significant amount of inextractable non-pertechnetate. If the non-pertechnetate is assumed to be completely inextractable, the fraction of non-pertechnetate is calculated to be 79%. The large difference between the K_d's indicates that it is reasonable to assume that little or none of the ^{95m}Tc pertechnetate tracer is converted to non-pertechnetate.

The total Tc K_d's for the BNFL AN-107 sample are very similar to those observed previously at Battelle for untreated samples of the same feed (K_d's of 13 – 21, representing 11% to 16% removal; (Kurath et al., 1999), and are similar to those observed previously at SRTC using a phase ratio of 10 instead of 100 (K_d's of 4 to 5, representing 27% removal; Hassan, 1997).

Based on these K_d's the predicted λ value for $^{95m}TcO_4^-$ in BNFL AN-107 is between 150 BV and 347 BV. Comparison with the treated archive AN-107 K_d's suggests it will be closer to 150 BV than to 347 BV. The predicted λ value for ^{99}Tc in BNFL AN-107 is between 6.4 BV and 14.7 BV. This prediction, however, is based on the implicit assumption that all the ^{99}Tc present is extractable, which is not correct. Therefore this prediction is of limited use.

Table 3.3. TcO_4^- and Total Tc K_d Values

Sample	Feed conditions	TcO_4^- K_d (mL/g)/ % Removal (1)	Total Tc K_d (mL/g)/ % Removal (2)
Archive AN-107 (3)	Na = 5.70 M Density = 1.256 g/mL ^{99}Tc=55 µCi/L (3.2 mg/L) NO_3/Tc = 6.8 x 10^4	257 / 70.5%	Not Determined
Archive AN-107 Dup	Na = 5.70 M Density = 1.256 g/mL ^{99}Tc=55 µCi/L (3.2 mg/L) NO_3/Tc = 6.8 x 10^4	445 / 80.9%	Not Determined
Treated Archive AN-107	Na = 4.70 M Density = 1.226 g/mL ^{99}Tc=48 µCi/L (2.8 mg/L) NO_3/Tc = 7.3 x 10^4	186 / 64.5%	Not Determined
Treated Archive AN-107 Dup	Na = 4.70 M Density = 1.226 g/mL ^{99}Tc=48 µCi/L (2.8 mg/L) NO_3/Tc = 7.3 x 10^4	211 / 67.3%	Not Determined
BNFL AN-107 (Treated)	Na = 4.84 M Density = 1.241 g/mL ^{99}Tc=57 µCi/L (3.3 mg/L) NO_3/Tc = 5.4 x 10^4	708 / 87.0%	30 / 22.3%
BNFL AN-107 (Treated) Dup	Na = 4.84 M Density = 1.241 g/mL ^{99}Tc=57 µCi/L (3.3 mg/L) NO_3/Tc = 5.4 x 10^4	303 / 74.1%	13 / 11.3%

1) Assumed equal to the ^{95m}Tc K_d.
2) Assumed equal to the ^{99}Tc K_d.
3) Archive and Treated Archive ^{99}Tc and NO_3/Tc estimated from Blanchard et al. (1997) and Hallen, Bredt, Brooks and Jagoda (2000)

3.3 Column Test: Loading (Breakthrough Curves) and Feed Displacement

Column preparation with 1.0 M NaOH was completed by 11:00 AM on November 3, 1999. The columns were left in 1.0 M NaOH overnight, and column loading with the BNFL AN-107 sample was started at 6:00 AM the next morning. Small samples (about 2 mL) were collected from the lead column every 10 BV of feed and from the lag column every 20 BV of feed. The initial samples were collected from the columns after just 10 BV to determine the maximum decontamination factors (DFs). (The DF of a sample is defined as C_0/C.) The loading phase generally went well. A few (less than 50) resin beads were observed to float above the top of the column.

The ^{99}Tc and $^{95m}TcO_4^-$ concentrations in the load effluent samples are shown in Figure 3.1 as % C/C_0 vs. the bed volumes of feed processed through each column. The C_0 value for ^{99}Tc was 57.1 µCi/L (3.34 mg/L). The initial $^{95m}TcO_4^-$ C_0 was 1820 counts/min/mL of sample. This decayed to 1664 counts/min/mL of sample by the end of the run. The $^{95m}TcO_4^-$ C_0 was recounted periodically to account for this decay. The C/C_0 value of 0.70 (i.e., 70%) is marked on the plot. This corresponds to the expected maximum allowed effluent concentration needed to meet the average ^{99}Tc limit in the LAW glass (0.1 Ci/m^3). The % C/Co values for ^{99}Tc and $^{95m}TcO_4^-$ in the effluent composite are also shown on the plot.

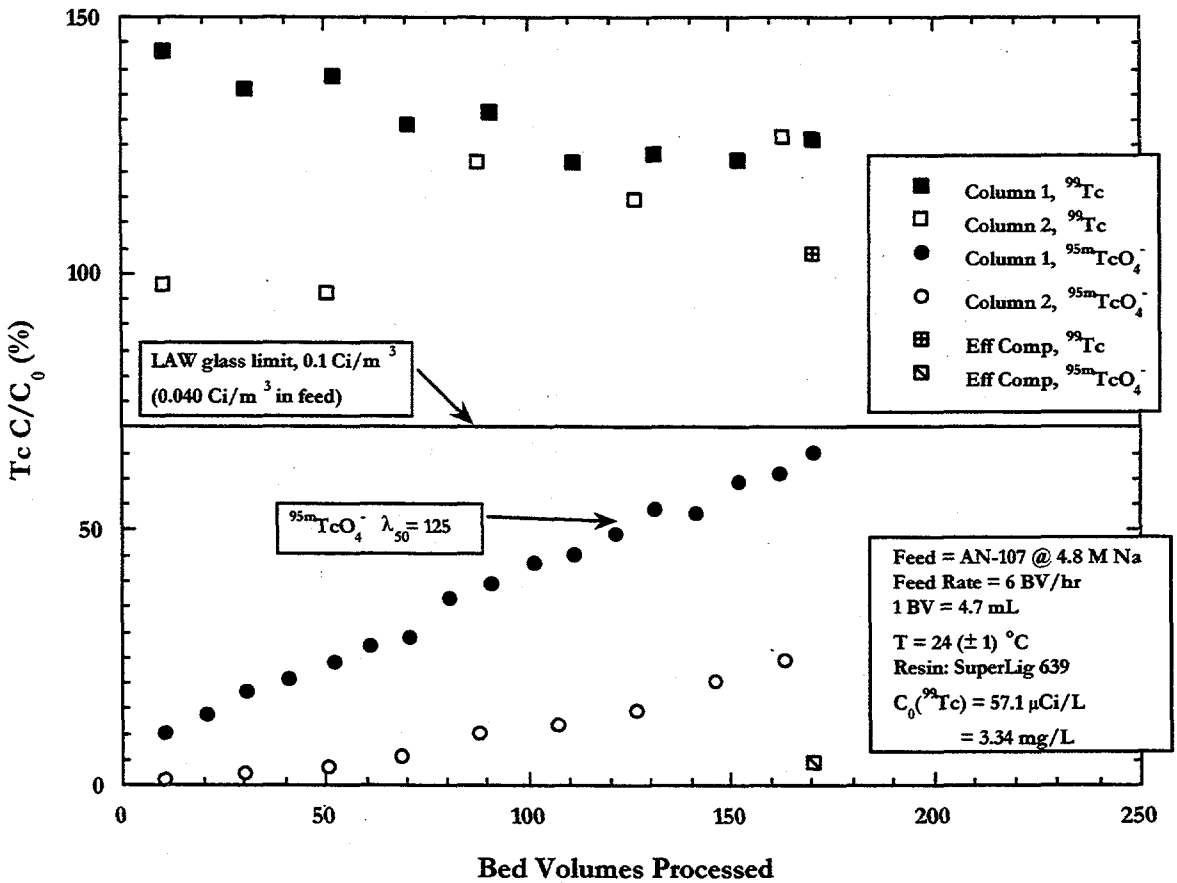

Figure 3.1. ^{99}Tc and $^{95m}TcO_4^-$ Breakthrough Curves, First and Second Columns

The $^{95m}TcO_4^-$ data form fairly typical breakthrough curves, with the exception that the breakthrough curve for the first column is about 10 percentage points higher than expected for a fresh column. This is attributed to the use of a column that was still partly loaded from a previous run, as described in the experimental section. The breakthrough is comparable to that on the last sample taken from this column at the end of the previous run (C/C_0 = 14.8%). However, a direct comparison cannot be made as the ratios of ^{95m}Tc to ^{99}Tc are not the same on the column and in the feed due to the decay of the ^{95m}Tc loaded on the column, and probable differences in the amount of tracer added to the two different feeds. There is a relatively high initial breakthrough on the second column (C/C_0 = 1.3%). This may be due to bleed of residual Tc from the column, which was previously loaded and eluted. For comparison, C/C_0 = 0.6% on the second column when C/C_0 = 9% on the first column during the AW-101 test.

In sharp contrast to the $^{95m}TcO_4^-$ data, the ^{99}Tc data indicate that ^{99}Tc was eluted from the column during the "loading" phase. The initial C/C_0 of approximately 145% decreased over the first 100 BV to approximately 125%, where it levels off. The initial ^{99}Tc C/C_0 for the lag column effluent is slightly less than 100%, then also climbs to approximately 125%. The behavior of the ^{99}Tc is attributed to the presence of a large fraction of non-pertechnetate Tc in the AN-107 feed and to the use of a column that was still partially loaded from a previous run, as described.

The initial DFs for ^{99}Tc for the first and second columns (derived from the ^{99}Tc concentration in the first sample from each column) are 0.7 and 1, respectively. The corresponding initial DFs for TcO_4^- (similarly derived from the $^{95m}TcO_4^-$ C/C_0 in the first sample from each column) are 9.9 and 74. These were also the maximum DFs observed for $^{95m}TcO_4^-$.

The λ value is the number of bed volumes of processed feed at which the C/C_0 value reaches 50% (0.5) and is a direct indicator of the effective capacity of the resin. Since the ^{99}Tc C/C_0 was never below 50% for either the lead or lag columns, ^{99}Tc λ values could not be calculated. The $^{95m}TcO_4^-$ λ value for the lead column is 125. The $^{95m}TcO_4^-$ breakthrough on the second column at the conclusion of the loading phase was sufficiently low (approximately 25%) that extrapolation to determine the λ value is not practical.

The overall ^{99}Tc DF was 0.96 (^{99}Tc was added to the feed by the process) and the concentration of ^{99}Tc in the effluent composite is 59.3 µCi/L (3.47 mg/L), 1.5 times the expected maximum allowed concentration needed to meet the average ^{99}Tc limit in the Envelope C LAW glass (0.1 Ci/m^3). The overall $^{95m}TcO_4^-$ DF was 22.5. The corresponding C/C_0 values are shown in Figure 3.1.

The $^{95m}TcO_4^-$ breakthrough curves for AN-107 are compared to those for AW-101 in Figure 3.2. The affect of the partially loaded lead column on the initial C/C_0 for AN-107 is clear.

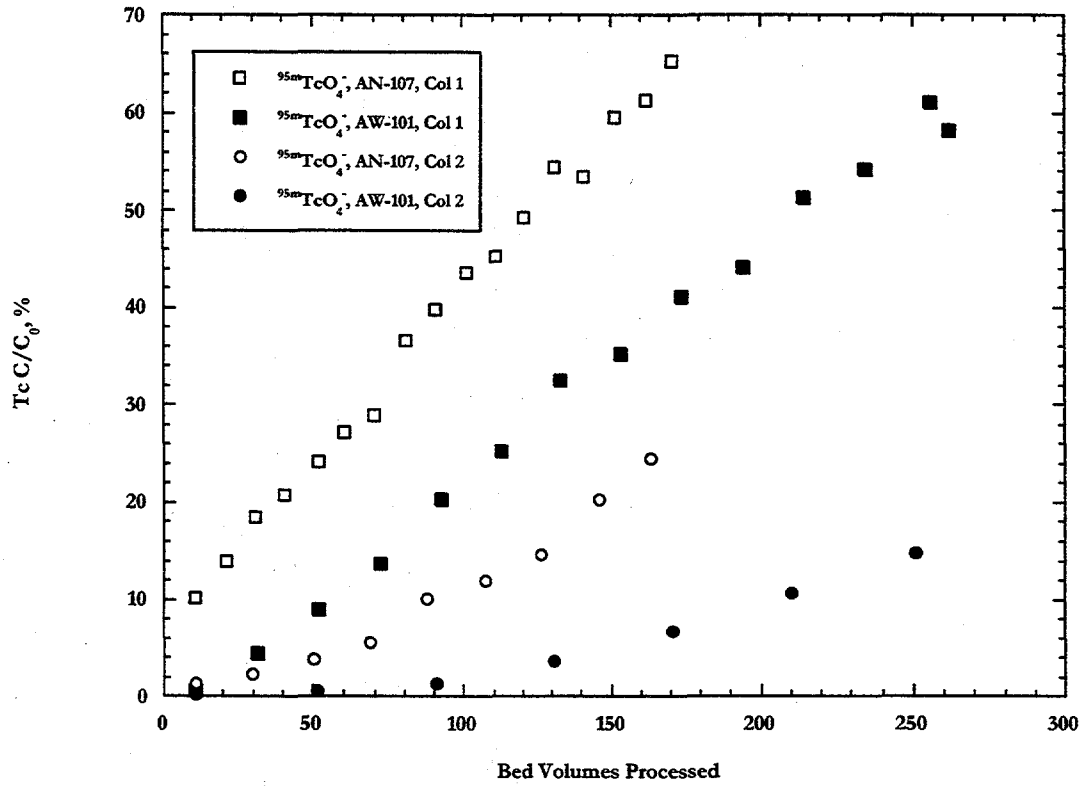

Figure 3.2. Comparison of $^{95m}TcO_4^-$ Breakthrough Curves for AW-101 and AN-107, First and Second Columns

Feed displacement was started immediately after the last load sample was collected. The beds were flushed in series with 7.5 BV of 0.1 M NaOH at 3.0 BV/hr to displace the feed prior to elution. (Direct contact of the feed and the eluant, DI water, would result in precipitation of some of the feed components.) The feed tube was transferred to the feed displacement solution prior to collection of the last load sample to reduce feed holdup in the feed displacement samples. The brown color indicative of feed began to lighten with the 4th feed displacement sample.

The ^{99}Tc and $^{95m}TcO_4^-$ concentrations in the feed displacement samples taken after the second column are shown in Figure 3.3 as C/C_0 vs. the bed volumes of feed displacement processed through the columns. The C/C_0 from the last load effluent sample from the second column is also shown on the plot. The $^{95m}TcO_4^-$ C/C_0 of the feed displacement samples drop off only slightly (from 25% to 20%) for the first 5 BV. After this the C/C_0 rises significantly, signaling elution. After the second point confirmed that the $^{95m}TcO_4^-$ was eluting, the feed displacement was stopped, at C/C_0 = 55%. The subsequent DI rinse was performed only on the first column to minimize this elution.

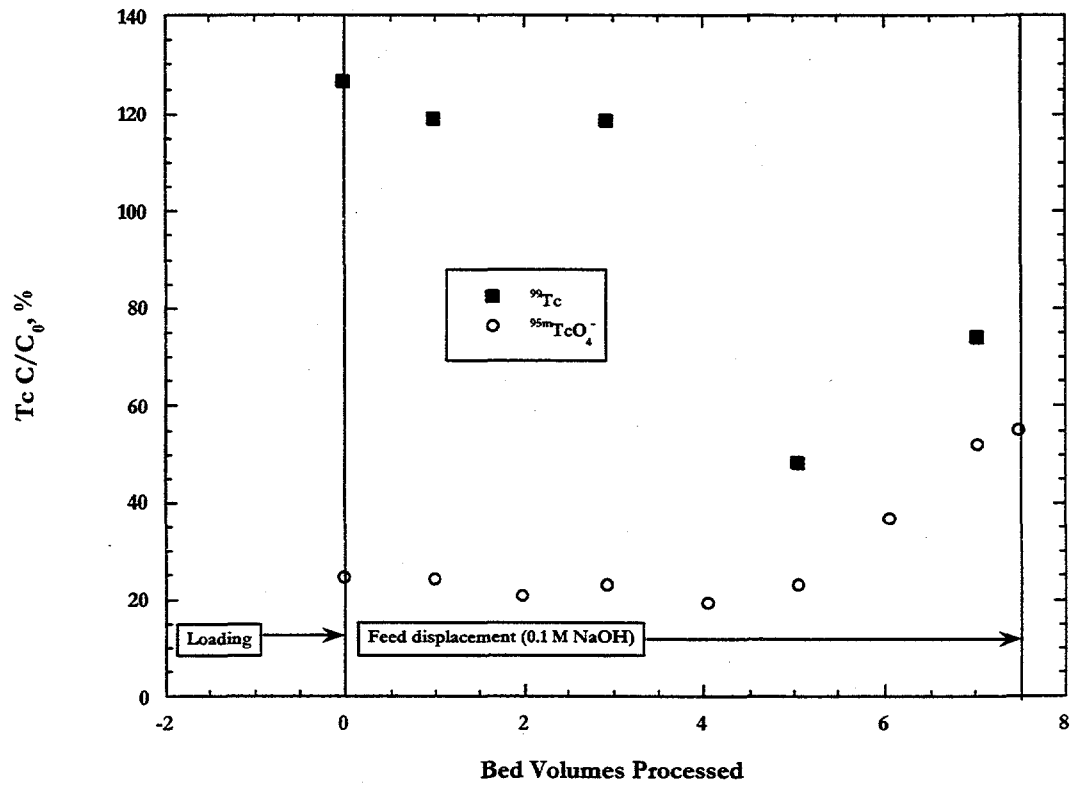

Figure 3.3. ^{99}Tc and $^{95m}TcO_4^-$ C/C_0 for Feed Displacement

The general behavior of ^{99}Tc is similar to $^{95m}TcO_4^-$, although the changes are much larger. The ^{99}Tc starts at a much higher C/C_0 (127%), but also drops off only slightly for at least the first 3 BV. At the 5th BV the C/C_0 drops quite strongly to just under 50%, before rising again to almost 75% by the 7th BV. This behavior is attributed to displacement of ^{99}Tc (with the feed) in the first 3 BV to 4 BV, followed by elution of $^{99}TcO_4^-$ from the bed by the 0.1 M NaOH. The observed elution of both ^{99}Tc and $^{95m}TcO_4^-$ with 0.1 M NaOH is consistent with information provided by the resin manufacturer that decreasing the ionic strength of the solution in contact with the SL-639 resin will elute perrhenate (ReO_4^-), a surrogate for TcO_4^-, from the resin (King, 1999).

The concentrations of sodium (Na), potassium (k), aluminum (Al) and hydroxide (OH^-) in the feed displacement are shown in Figure 3.4. The concentrations of Na, K, and Al are indicated on the left hand axis while the OH^- concentration is shown on the right hand axis. Both Y-axes are logarithmic scales in order to clearly show the large decreases in the concentrations. The samples were taken at the effluent line from the lag column, after the solutions had passed sequentially through both columns. Analytical results and calculations may be found in the appendix.

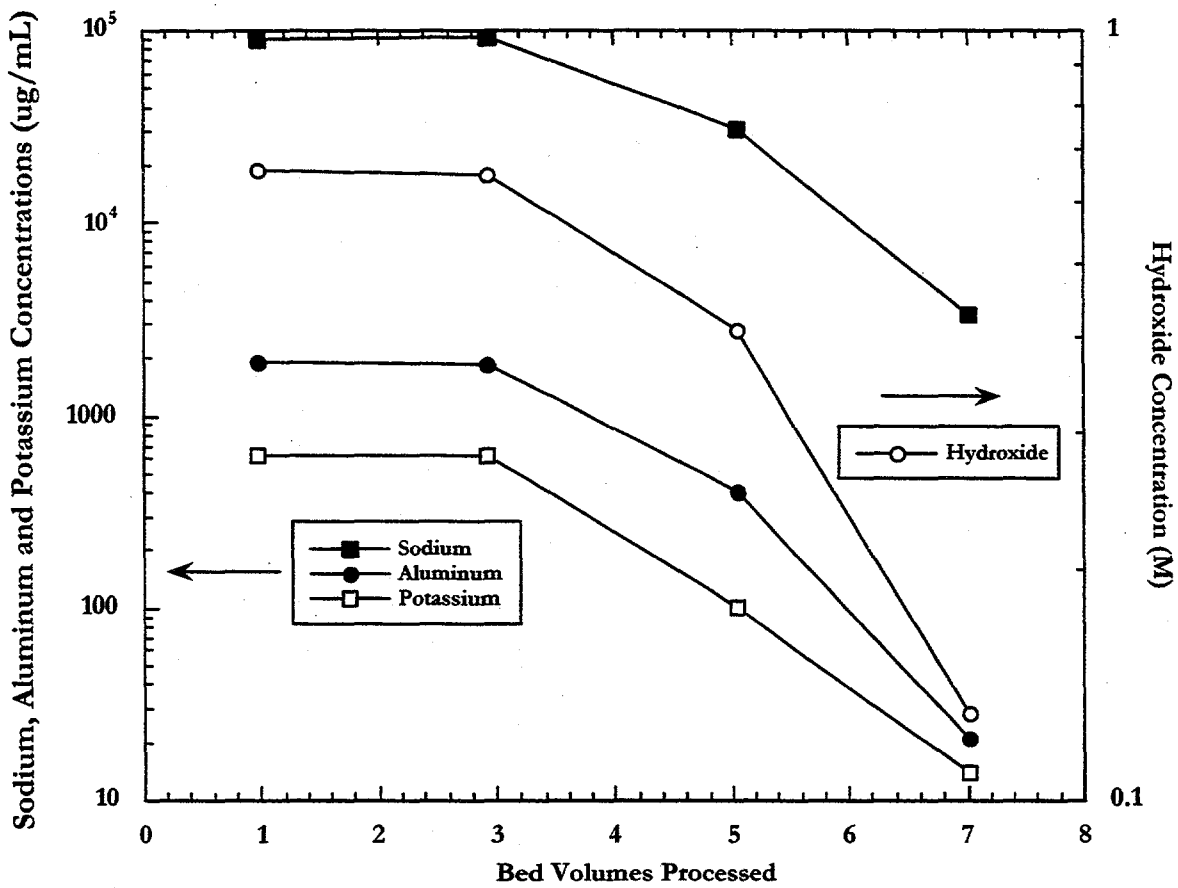

Figure 3.4. Component Concentrations in Feed Displacement Samples

The concentrations of the four components do not change between the first and second samples (first three BV) of the feed displacement, but a large decrease in all concentrations is observed in the third sample (5th BV). The displacement of the feed is reasonably sharp, with the concentrations of most components dropping 1.5 to 2 orders of magnitude between the 3rd and 7th bed volumes. The Na and OH^- concentrations do not drop as low as the others as they are present in the displacement solution. The feed components (excluding OH) are 66% to 84% reduced in the 5th BV, and 96% to 99% reduced in the 7th BV.

The observed color change in the samples and the behavior of ^{99}Tc, $^{95m}TcO_4^-$, and the other feed analytes are all consistent with the conclusion that the first 3 BV to 4 BV consist of feed holdup. This is consistent with the estimated system holdup volume of 3 BV, as stated in Section 2.1. Based on this data, it is recommended that the feed displacement be terminated after the 5th bed volume. Elution continued with

the room temperature DI water rinse (see appendix for data). If a small amount of OH^- can be tolerated in the eluate, it may be desirable to omit the room temperature DI water rinse and proceed directly to the 50°C DI water elution. Assuming 2 BV of 0.1 M NaOH and 50 BV of DI eluate, the OH^- concentration in the eluate would be approximately 0.004 M.

3.4 Column Test: Water Rinse, Elution, Eluant Rinse and Regeneration

The 0.1 M NaOH feed displacement was followed by a room temperature de-ionized (DI) water rinse of 2.1 BV at 3.1 BV/hr (lead column only). The rinse was started on November 8, 1999, 67 hours after the feed displacement. (The column was left in the feed displacement solution over the weekend.) At the completion of the DI water rinse the lead column was eluted with DI water at 53.5°C at a rate of 1 BV/hr. The requested elution temperature was 50(+/- 5)°C; the water bath set point was set to 53.5°C to compensate in case the actual bed temperature was lower. As shown below, the bed temperature was most likely at the same temperature (+/- 0.3°C) as the water bath. The water bath reached 50°C within 12 minutes while circulating water through the column jacket. The column was allowed to warm for about 30 minutes before elution was started. The total time between the last rinse sample and the start of the elution was 2 hours 40 minutes. The eluate was collected in 1 BV increments. The elution was stopped at 20 BV for 22 minutes to check the liquid level above the bed. Elution was stopped again at 37 BV for 7.2 hours (overnight) to reduce RCT overtime and expense, and to investigate the effect of allowing the column to stand in DI water at temperature (53.5°C) for an extended period of time. The heater was left on during both breaks.

When the elution was stopped and the lead column had cooled to room temperature (overnight), it was rinsed with room temperature DI water at 6 BV/hr. Samples were collected in 1 BV increments. After 2 BV, the feed tube was transferred to the 1 M NaOH reservoir for the regeneration, but the next 2 BV were collected as DI rinse due to the holdup in the system, giving a total of 4 BV of DI rinse. The regenerate was then immediately collected (both columns) in a 6.4 BV batch. (The flow rate had increased to 6.4 BV/hr.) Collection of the regenerate was complete at 10:15 AM on November 11, 1999.

The $^{95m}TcO_4^-$ C/C_0 for each sample was determined soon after collection (generally within an hour). The ^{99}Tc concentrations in selected samples were determined later by ICP-MS. The elution, rinse and regeneration data are shown in Figure 3.5. The Y axis is a logarithmic scale to clearly show the large range of C/C_0 values. (Note these are <u>not</u> $\%C/C_0$ values.)

The concentrations of ^{99}Tc and $^{95m}TcO_4^-$ jumped sharply from the last room temperature DI rinse sample to peak in the 2nd eluate bed volume at C/C_0 of 27 and 51, respectively. This is probably due both to the higher temperature of the water and to the 2.7 hours during which the loaded column sat in the water eluant. The elution tailed off slowly, however, only dropping to C/C_0 of 0.24 at 22 BV. After a 22-minute break, C/C_0 jumped to about 0.4, and then tailed off slowly again. A larger, sharper rise in C/C_0 was observed following a longer (7.2 hour) break, but again the concentration tailed off very slowly.

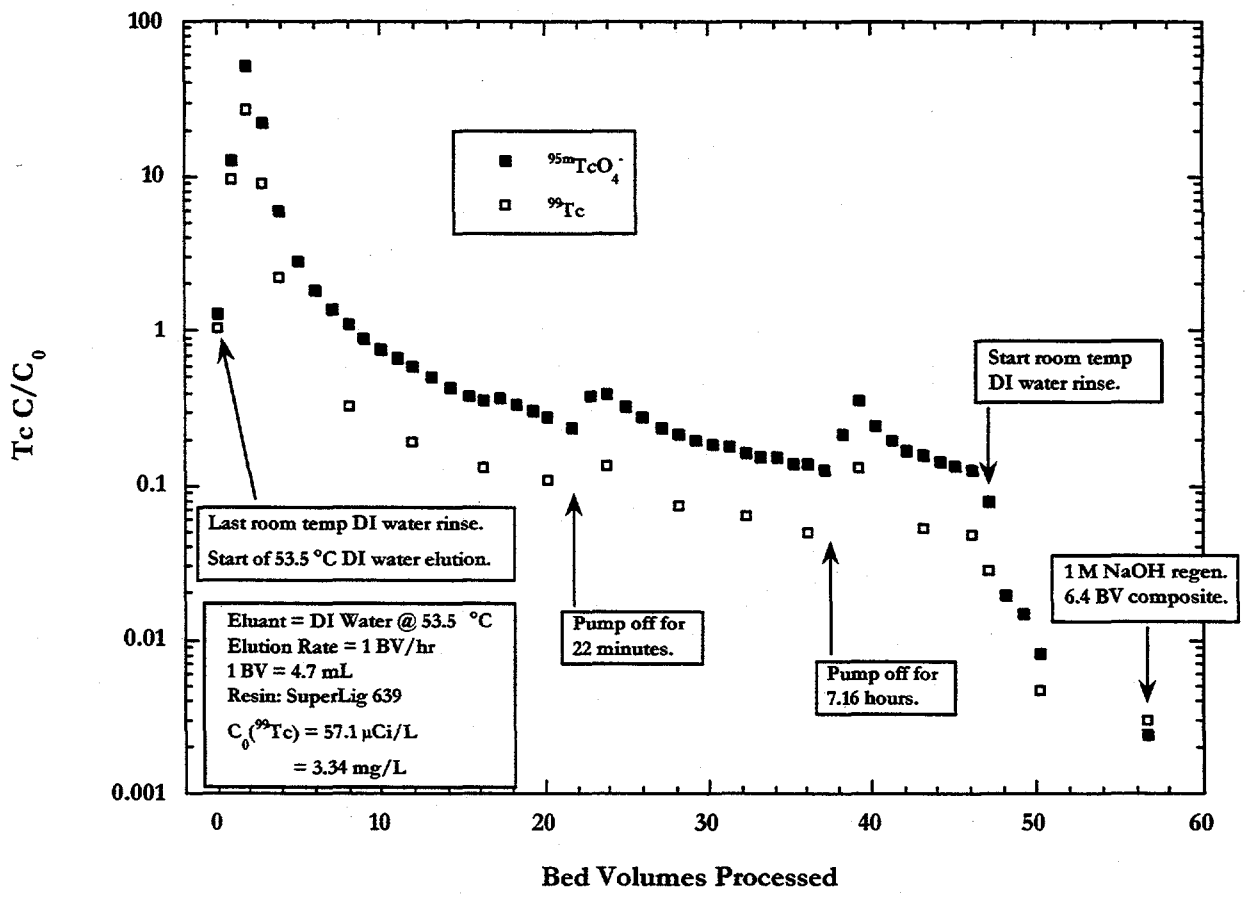

Figure 3.5. Elution, Eluant Rinse and Regeneration of Lead Column

The elution was continued for 8 hours after the last break. At that time C/C_0 of the $^{95m}TcO_4^-$ tracer had dropped to 0.13. Subsequent analysis of ^{99}Tc concentration by ICP-MS indicated that the ^{99}Tc C/C_0 was 0.05 in the last sample. The water bath was turned off, and the column was allowed to cool overnight (about 16 hours), and then rinsed with 4 BV of room temperature DI water at 6.1 BV/hr. This rinse was collected in 1 BV increments. The C/C_0 of both Tc isotopes in the first rinse sample were much lower than in the last elution sample, in spite of standing overnight in contact with the resin. When compared with the rises in C/C_0 seen when the resin stood in contact with warm (50°C) eluant, the drop illustrates the large effect temperature has on the equilibrium of Tc between resin and eluant.

The C/C_0 of both Tc isotopes dropped nearly an order of magnitude over the 4 BV rinse; both were less than 0.01 in the last sample. The concentration of ^{99}Tc in the last elution sample was 2.7 µCi/L (0.160 mg/L) and 0.26 µCi/L (0.015 mg/L) in the last rinse sample.

Although the ^{99}Tc and $^{95m}TcO_4^-$ elution and rinse data track well, at any given point the magnitude of C/C_0 for ^{99}Tc is significantly lower than that for the tracer. This indicates that the ratio of Tc on the column to that in the feed is lower for ^{99}Tc than it is for ^{95m}Tc. This is consistent with the observation that ^{99}Tc did not load on the column while $^{95m}TcO_4^-$ did. The ^{99}Tc observed to elute is that left from the initial partial loading with AW-101, minus the amount that eluted during the "loading" phase. The $^{95m}TcO_4^-$ observed to elute is that left from the initial partial loading with AW-101, plus the large amount that loaded during the loading phase. This difference between the ratio of ^{99}Tc to ^{95m}Tc in the feed and on the column is reflected in the elution and rinse C/C_0 values.

The ^{99}Tc concentration in the 6.4 BV regenerate batch was found to be 0.14 µCi/L (0.008 mg/L), for a C/C_0 of 0.003. The $^{95m}TcO_4^-$ C/C_0 was found to be 0.002. Results of various analyses of a sample of the regenerate are shown in Table 3.4.

Table 3.4. Composition of Regenerate

	Concentration, µg/mL	Concentration, M
Na	19200	0.83
K	< 33.8	< 8.6E-4
Al	11.2	4.15E-4
OH^-	-	0.85
^{99}Tc	0.008 (0.14 µCi/L)	8.1E-8
Density, g/mL	-	1.04

The temperature of the resin bed during elution was assumed to be 50(+/- 5)°C. A thermocouple was subsequently placed at the top of a resin bed and the temperature was measured while the elution conditions were reproduced in order to check this assumption. The thermocouple was a K type enclosed in stainless steel. It was inserted along the long axis of the column and sealed in an elbow at the bottom, allowing flow during temperature measurement. The tip of the thermocouple, where the temperature is measured, was barely visible emerging from the top of the resin bed. This location, where the eluant first contacts the bed, is expected to be the coldest one in the bed. The resin bed used for the loading and elution test had been removed from the column for chemical analysis, so another bed was packed in the column. A shorter resin bed (3.4 cm) was required in order to locate the tip of the thermocouple at the top of the bed. The volume of the bed was therefore only 2.7 mL, about half the size of the bed used for the actual loading and elution test. The temperature at the top of the bed is expected to be independent of the bed volume when the flow rate (in mL/hr) is the same.

A plot of the temperature measured by the thermocouple at the top of the bed during ramp up and elution is shown in Figure 3.6. The zero is taken when the water bath started heating. The temperature was recorded every 5 sec. The bed temperature at ambient hood conditions was collected for 15 minutes before heating was started (not shown). The setpoint on the water bath was set to the same value as for the actual elution (53.5°C). The bed temperature was observed to lag behind the water bath temperature by about 1°C during heating. The bed temperature reached 50°C within 28 minutes. The temperature never dropped below 50°C again, but took 80 minutes (from the start of heating) to stabilize at 53.6°C. At 1 hr 30 min elapsed time, the water bath setpoint was unintentionally reduced to 53.1°C, and the temperature dropped to 53.2°C, where it quickly restabilized (15 – 20 min).

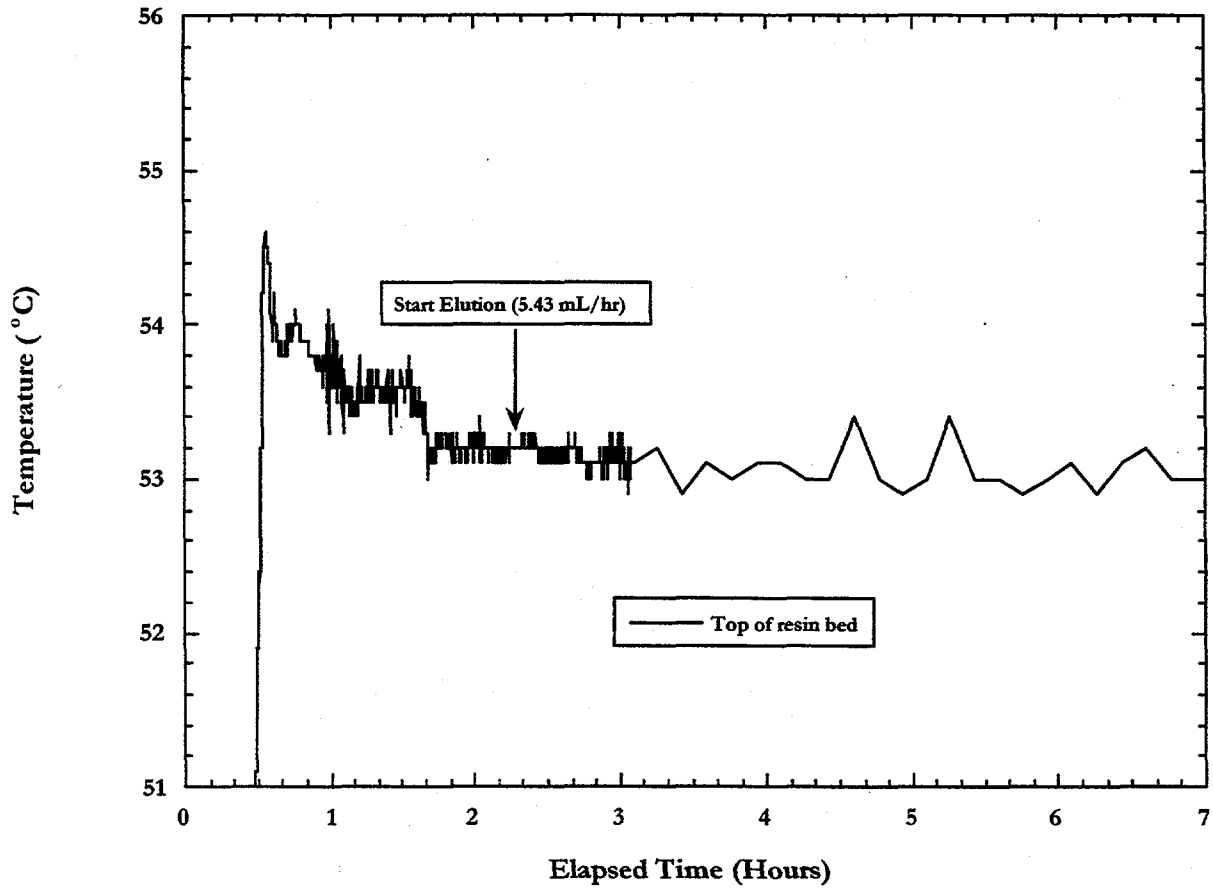

Figure 3.6. Bed Temperature During Elution

Elution was started at about 2.3 hrs elapsed time (137 min). A slight (0.1 - 0.2°C) drop in the bed temperature is observed starting 20 min after the elution was begun. Evidently the drop was insufficient to change the water bath temperature and trigger a correction. The elution was run for 4.5 hours. The data collection frequency was reduced to once every 10 min at about 3 hrs elapsed time. During the elution the bed temperature varied between a low of 52.9°C and a high of 53.4°C. The average feed rate was found to be 5.46 mL/hr, somewhat faster than for the actual elution test (4.75 mL/hr).

The temperature drop observed 20 min after elution was started is attributed to the introduction of cold eluant at the top of the bed. The 20 min lag is attributed to preheating of the eluant in the flow adapter. The adapter assembly is in direct, tight contact with the water jacket, so it and the eluant in it were heated with the column. The time required for the eluant to descend the full length of the adapter is estimated to be 15 - 20 min at the flow rate used. The fact that the drop was only 0.2°C is attributed to preheating of the eluant and the slow feed rate. The eluant was warmed as it traversed sections of tubing taped to the much larger water bath circulation tubing, as it descended the flow adapter, and in the small head (approx. 0.16 mL) of eluant above the resin bed.

In summary, the eluant temperature measured at the top of the resin bed remained between 52.9°C and 53.4°C, within 0.3°C of the water bath setpoint, during the 4.5 hour elution. The top of the bed is expected to be the lowest temperature location, and the faster feed rate used in this elution test is expected

to lead to a greater temperature drop than in the initial elution test. Therefore the assumption that the bed temperature was between 50°C and 55°C during the initial elution test appears justified. Note that preheating of the eluant is particularly important if the resin beds are loaded and eluted in the same direction, as in this test. The highest concentration of Tc will occur at the upstream end of the bed. If the bed is eluted in the same direction, the eluant must be at temperature when it first contacts the bed to ensure maximum elution of Tc.

The elution was faster than observed in a previous test when 0.5 M nitric acid (HNO_3) at ambient (room) temperature was used as an eluant (Blanchard, Kurath, and Bontha, 2000). This is evident in a plot of the data from the two elutions, shown in Figure 3.7. The concentrations of ^{99}Tc and $^{95m}TcO_4^-$ both peaked much higher and much more rapidly when DI water at 53.5°C was used. The comparison must be made with caution, however, since the Tc load on the column was almost certainly much lower for the elution reported here.

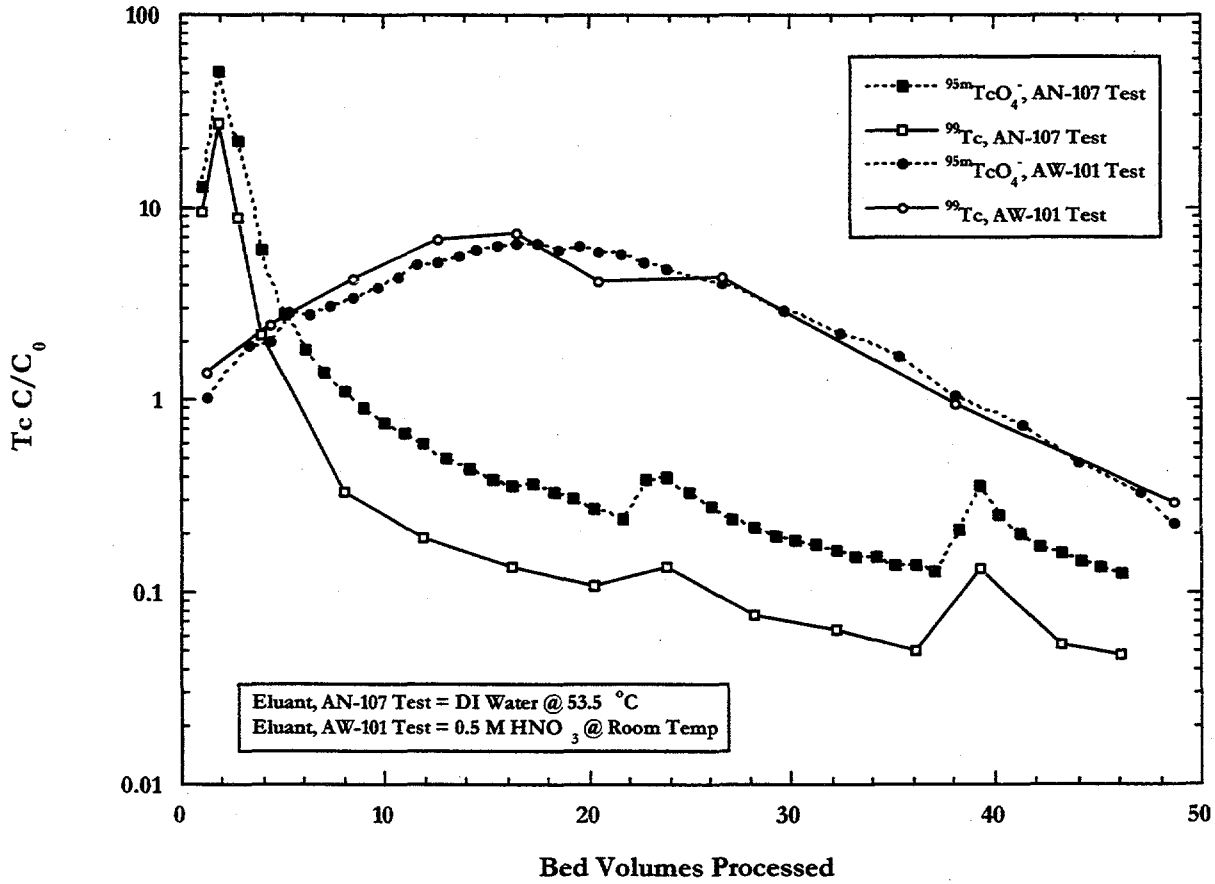

Figure 3.7. Comparison of Elution with 0.5 M Nitric Acid at Ambient Temperature To Elution with De-ionized Water at 53.5°C

More testing to determine optimum elution conditions to minimize the overall process cycle time may be desirable. Testing could include: 1) investigating the relationship between extent of column loading and rate of Tc elution; 2) investigating the relationship between the length of soak time prior to the start of the elution and the rate of subsequent Tc elution; 3) investigating the relationship between C/C_0 when the

elution is terminated and the amount of Tc that bleeds from the column with the next loading cycle; 4) investigating the relationship between temperature and rate of Tc elution.

The eluate samples were combined to produce a 145.9 mL composite, and a sample was submitted to the analytical laboratory for a number of analyses. The results are shown in Table 3.5. The BNFL specified minimum reportable quantity (MRQ) levels for the components determined by ICP were all met. Sodium is the dominant constituent at 105 µg/mL (5 mM). Other significant components may have leached from the glassware used to hold the samples (Si, B). The total organic carbon and total inorganic carbon were both less then 20 µg C/mL, meeting the MRQ. The MRQ levels for the anionic components determined by IC were all met. Nitrate was the dominant anion at 19 µg/mL (0.3 mM). Chlorine, nitrite, sulfate and hydroxide were also present. ^{99}Tc and ^{95m}Tc were the dominant radionuclides.

Table 3.5. Analysis of Eluant Composite and Minimum Reportable Quantities

ICP Components		BNFL MRQ	Anions and Carbon		BNFL MRQ
	µg/mL	µg/mL		µg/mL	µg/mL
Al	[0.46]	7.50E+01	TOC	<20	1.50E+03
Ba	[0.10]	7.80E+01	TIC	<20	1.5E+02
Ca	[1.9]	1.50E+02	F	<0.3	1.5E+02
Cd	<0.078	7.50E+00	Cl	15	3.0E+00
Co	<0.26	3.00E+01	NO_2	8	NMRQ
Cr	<0.10	1.50E+01	Br	<0.3	NMRQ
Cu	<0.13	1.70E+01	NO_3	19	3.0E+03
Fe	<0.13	1.50E+02	SO_4	1	2.3E+03
K	<10.	7.50E+01	PO_4	< 0.5	2.5E+03
La	<0.26	3.50E+01	C_2O_4	< 0.5	NMRQ
Mg	<0.52	1.50E+02	OH	0.002 M	NMRQ
Mn	<0.26	1.50E+02			
Mo	<0.26	9.00E+01	Radionuclides		BNFL MRQ
Na	105	7.50E+01		µCi/mL	µCi/mL
Ni	[0.18]	3.00E+01	Cs -137	< 3.E-4	9.00E+00
Pb	<0.52	3.00E+02	Cs -134	<2.E-4	NMRQ
Si	[22]	1.70E+02	Sr-90	8.70E-4	1.50E-01
Sn	<7.8	1.50E+03	Tc-99	6.08E-2	1.50E-03
Ti	<0.13	1.70E+01	Tc-95m	4.50E-1	NMRQ
U	<10.	6.00E+02	Am-241	<7E-4	7.20E-04
Zn	<0.26	1.65E+01	Eu-154	<1.E-4	2.00E-03
B	10.5	NMRQ	Eu-155	<7.E-4	9.00E-02
P	<5.2	NMRQ	Total Alpha	5.73E-6	2.30E-01

Notes:
Total volume of eluate = 145.9 mL
MRQ = minimum reportable quantity
NMRQ = no minimum reportable quantity
Overall error is estimated to be within +/- 15%
Values in brackets are within 10-times the detection limit and errors are likely to exceed +/- 15%
Tc-95m (61 day half-life) activity measured as of 12/17/99, 8:00 AM PST

3.5 Column Test: Reloading of Eluted Column

Following the elution, rinse and regeneration of the lead column, AN-107 simulant and feed were processed through the column to determine the amount of residual Tc on the resin that would bleed into the effluent with the next loading cycle. Simulant loading was started on December 9, 1999 at 10:30 AM. The column had been left in 1.0 M NaOH from the regeneration, so the first 3 BV of effluent were discarded. The simulant contained no $^{95m}TcO_4^-$ or ^{99}Tc tracers. However, ^{95m}Tc was detected in the effluent indicating that some of the residual $^{95m}TcO_4^-$ on the column was bleeding into the effluent. Once a $^{95m}TcO_4^-$ baseline was established (13.4 BV), the column was immediately flushed with 4.9 BV of 0.1 M NaOH. No effort was made to eliminate holdup. The 0.1 M NaOH was observed to elute the $^{95m}TcO_4^-$ slightly. The column was left overnight in the 0.1 M NaOH, and then loaded with actual AN-107 starting at 9:00 AM the next morning. Once a $^{95m}TcO_4^-$ baseline was established (29.8 BV), the column was immediately flushed with 4.6 BV of the regenerant, 1.0 M NaOH.

As for the other phases of testing, the $^{95m}TcO_4^-$ C/C_0 for each sample was determined soon after collection and the ^{99}Tc concentrations in selected samples were determined later by ICP-MS. The ^{99}Tc C_0 used for calculating C/C_0 values for the simulant was taken as C_0 of the actual feed. (This was found to be 45.0 µCi/L, 21% lower than the previous value of 57.1 µCi/L. Possible causes for this discrepancy are discussed below.) The data are plotted in Figure 3.8. The Y axis is linear and the C/C_0 values are given as percentages for ease of comparison with the breakthrough curves in Figure 3.1. The last elution, rinse and regeneration data are shown for comparison. The start of simulant loading is taken as 0 BV. Raw data may be found in the appendix. The 89% C/C_0 value corresponding to the expected maximum allowed effluent concentration needed to meet the average ^{99}Tc limit in the LAW glass (0.1 Ci/m^3) is indicated on the plot. This value is different from that in Figure 3.1 due to the difference in the measured C_0 value.

Both $^{95m}TcO_4^-$ and ^{99}Tc bleed from the column into the simulant at levels higher than observed in the last rinse sample and the regeneration sample. Both appear to dip and then rise very slightly, with $^{95m}TcO_4^-$ near 5% and ^{99}Tc near 2.5% after 13.4 BV. For comparison, the last rinse sample C/C_0 were 0.8% and 0.5%, for $^{95m}TcO_4^-$ and ^{99}Tc, respectively. After a 2 BV lag due to holdup, the 0.1 M NaOH begins to elute both $^{95m}TcO_4^-$ and ^{99}Tc.

The $^{95m}TcO_4^-$ continues to climb during another 2 BV lag as actual AN-107 is started loading, then drops to 5%. The contribution from the residual Tc on the column in this case is significant. The $^{95m}TcO_4^-$ C/C_0 then rise in a fairly typical breakthrough curve, offset by the bleed from the residual $^{95m}TcO_4^-$ on the column. After another 2 BV lag, the C/C_0 begin to drop with the 1.0 M NaOH regeneration, but more slowly than expected.

In contrast to $^{95m}TcO_4^-$, the ^{99}Tc C/C_0 rise sharply to 78% as the actual AN-107 is loaded. This breakthrough is attributed mostly to the non-pertechnetate present in the feed. The 22% ^{99}Tc that was removed is slightly better than the target of 20% for Envelope C. After the initial sharp rise, the ^{99}Tc C/C_0 continue to rise slightly, as the pertechnetate ^{99}Tc breakthrough increases on top of the presumably constant non-pertechnetate ^{99}Tc breakthrough. The ^{99}Tc C/C_0 then drop sharply with the 1.0 M NaOH regeneration as the feed is displaced.

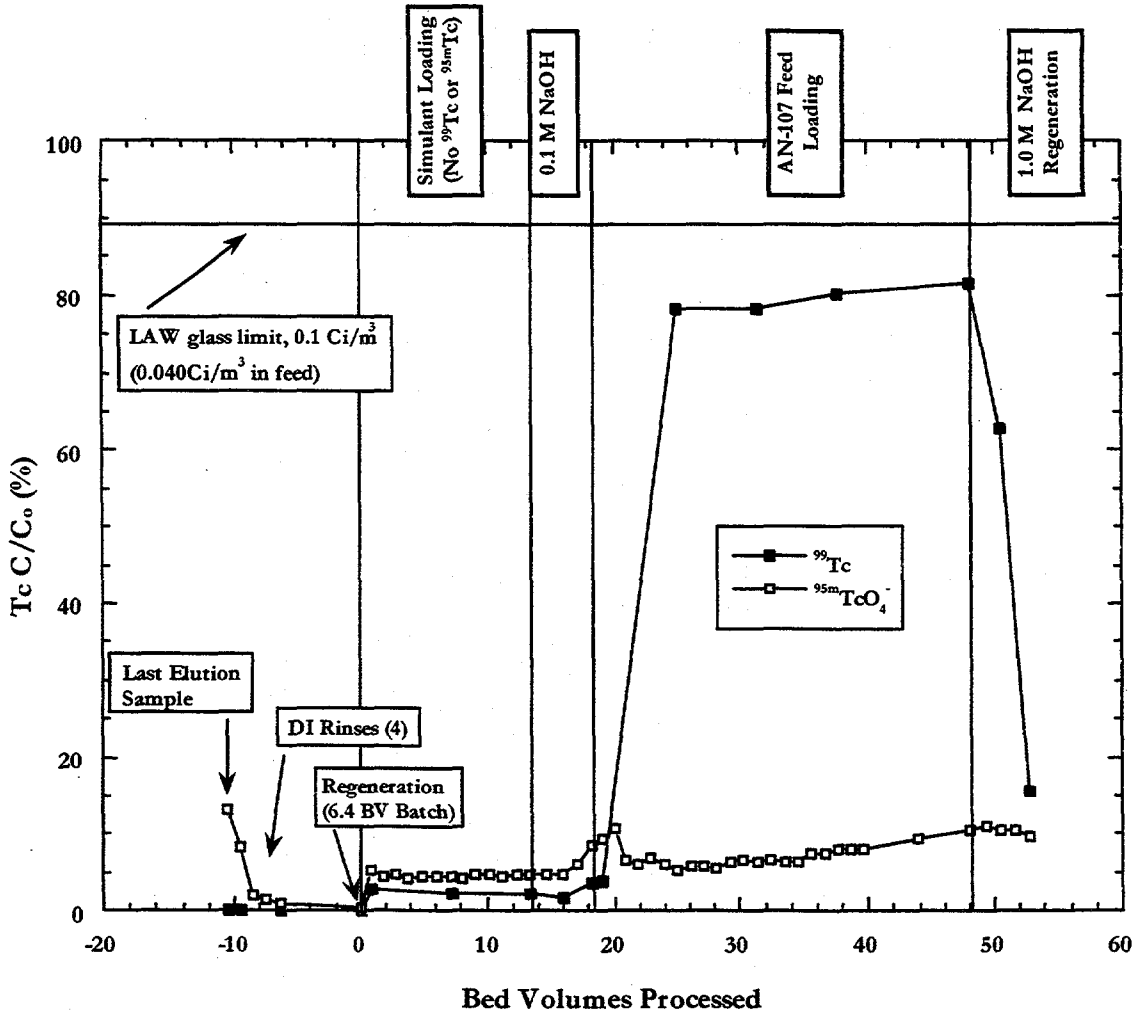

Figure 3.8. Reloading of Eluted Column with Simulant and Actual Feed

An immediate high ^{99}Tc breakthrough (58%) was also observed during Phase IA testing by Hassan and McCabe (1997) for similar feed and the same resin, and was attributed to the presence of non-pertechnetate technetium. Comparison with the data from that report must be made with caution, however, as the feed was prepared differently and virgin SL-639 resin was used for that study.

If it is assumed that the resin retains none of the non-pertechnetate, the fraction of non-pertechnetate Tc in the AN-107 may be calculated using the data in Figure 3.7. The initial ^{99}Tc breakthrough is 78%. Subtracting the contribution from the column bleed (2.5%), the non-pertechnetate fraction is calculated to be about 75.5%, leaving 24.5% as TcO_4^-. This is close to the 29% estimated in Section 3.1, and certainly within the estimated error bars (± 10%). It is also consistent with a previous estimate (Kurath et al., 1999).

Bleed from the residual Tc on a loaded and eluted column results in immediate breakthrough of 2.5% to 5%. For AN-107, this is dwarfed by the breakthrough of non-pertechnetate ^{99}Tc. For other waste feeds and/or envelopes, the column bleed may represent a much larger fraction of the initial breakthrough.

The plot suggests that a freshly eluted bed of SL-639 will remove sufficient ^{99}Tc from AN-107 feed, under the test conditions, to meet the average ^{99}Tc limit in the LAW glass without blending or additional alternate pretreatment. The C_0 determined for this set of loading cycle tests, 45.0 µCi/L, is low relative to the value found for the first loading cycle test (57.1 µCi/L), although the same feed bottle was used. The first value found is consistent with three values found for this feed following Sr/TRU removal (54.7 µCi/L, 54.0 µCi/L, and 60.4 µCi/L, in sample DF-20, a duplicate of DF-20, and sample DF-21; Hallen, Bredt, Brooks and Jagoda, 2000).

The cause of the difference in the two C_0's is unknown. If it is due to inadvertent dilution of the feed after the first set of tests, the expected average limit in Figure 3.8 would be reduced from 89% to 70%, the C/C_0 values would remain the same (i.e., jump to 78% immediately on loading), and it would be concluded that SL-639 will not be able to remove sufficient Tc to meet the expected average limit in AN-107 feed. If the low C_0 value is due to an error in only the feed sample analysis, the average limit in Figure 3.8 would be reduced to 70%, and the C/C_0 values would be reduced as well. The curve would jump to only 62% and would climb to 64% over the 30 BV run. In that case it would be concluded that the SL-639 will be able to remove sufficient Tc from AN-107 to meet the average limit, but most likely for a limited volume of feed per load cycle.

In either case it appears that the ratio of ^{99}Tc to sodium in AN-107 waste is low enough that SL-639 will provide sufficient Tc decontamination that the LAW limit can be met. If it is not possible to meet the limit specifically for AN-107 feed, it is expected that more than enough ^{99}Tc can be removed from other envelopes so that the limit can be met on average. It has been demonstrated, for example, that more than enough ^{99}Tc can be removed to meet the expected limit for AW-101, an Envelope A waste (Blanchard et al., 2000). However, other Envelope C wastes should be tested to confirm this, and to determine the removal requirements for the other envelopes. Although AN-107 is believed to have the largest fraction of inextractable Tc due to its high concentrations of organic complexants and complexant fragments, wastes from Tanks 241-SY-101 and 241-SY-103 contain over 4 times more Tc, and also have large fractions of inextractable Tc (50% or more; Blanchard et al., 1997).

4.0 CONCLUSIONS AND RECOMENDATIONS

Batch contacts of various Tank AN-107 (Envelope C) waste feeds with SL-639 showed reasonable removal of TcO_4^- (65% - 87%), as measured by a $^{95m}TcO_4^-$ tracer, but showed low total Tc removal (11% - 22%), as measured by ^{99}Tc.

An attempt to load ^{99}Tc from AN-107 waste onto a 4.7 mL bed of SL-639 that had been partially loaded with ^{99}Tc in a previous test of AW-101 waste resulted in elution of ^{99}Tc from the column. Even after passing through a second, eluted, 4.7 mL bed, the minimum concentration of ^{99}Tc in the effluent samples was greater than 54.7 µCi/L (3.21 mg/L). An overall DF of 0.96 was obtained using the two 4.7 mL beds in series, indicating that the concentration of ^{99}Tc in the effluent was slightly higher than in the feed. The ^{99}Tc concentration in the effluent was 59.3 µCi/L (3.47 mg/L). The poor performance is attributed to the presence of a large fraction of non-pertechnetate Tc in the AN-107 feed and the partially loaded column used for the tests.

Pertechnetate, as measured by a $^{95m}TcO_4^-$ tracer, was effectively removed by SL-639 during the small column test, in sharp contrast to results for total Tc. The maximum TcO_4^- DFs for the first and second columns were 9.9 and 74, as determined from the C/C_0 of the tracer in the first samples taken from the columns (after 10 BV). The relatively low values reflect the partially loaded lead bed used for the test. The overall TcO_4^- DF, determined from the C/C_0 of $^{95m}TcO_4^-$ for a sample of the effluent composite, was 22.5. Pertechnetate was not as effectively removed from this feed with this bed as it was from Tank 241-AW-101 (Envelope A) waste using virgin resin.

The feed displacement (0.1 M NaOH) volume of 7.5 BV was sufficient to flush feed from the columns, as indicated by the disappearance of the dark brown color of the feed and a 1.5 - 2 order of magnitude drop in the concentration of most feed components (Na, Al, P, etc.). However, the feed displacement appeared to elute Tc from the column slightly after the 5th BV. For this reason it is recommended that the feed displacement be terminated with the 5th BV. This feed displacement volume is larger than the assumed flow sheet volume, but the test system has a total system holdup in the pumps, valves and tubing equal to 3 bed volumes of resin. A smaller holdup volume would likely allow a reduction in the required amount of feed displacement. A subsequent rinse of only the first column further reduced most feed components, but also continued to elute Tc slightly.

Elution of the technetium-loaded columns with DI water at 53.5°C was much faster than a previous elution using 0.5 M nitric acid at room temperature. The concentration of ^{99}Tc peaked in the 2nd BV at $C/C_0 = 27$. The tail off was still quite slow, however, with more than 20 BV required to reach $C/C_0 = 0.10$. When the 53.5°C DI water was allowed to stand in contact with the loaded resin, the concentration of Tc in the eluate samples increased when the elution was restarted. The longer the contact, the higher the concentration on restart. The tail off from these spikes was still quite slow, however. After 46 BV, C/C_0 for ^{99}Tc was less than 0.05. When the column had cooled to room temperature, a 4 BV DI water rinse decreased the C/C_0 for ^{99}Tc in the eluate samples by another order of magnitude, to 0.005.

The SL-639 was regenerated with 6.4 BV of 1.0 M NaOH. The regeneration effluent was collected in one batch, in which the ^{99}Tc concentration was found to be 0.14 µCi/L (0.008 mg/L).

When AN-107 waste feed simulant containing no Tc was flushed through the eluted SL-639 bed, residual ^{99}Tc was found to bleed from the resin ($C/C_0 = 2.5\%$ relative to actual AN-107 feed). When actual AN-107 waste feed was then run through the column, there was immediate high breakthrough

(C/C_0 = 78%) of ^{99}Tc. The 22% initial removal slightly exceeds the target of 20% for this envelope. Removal drops below 20% by the 20th bed volume.

Special analysis of the feed, and the $^{99m}TcO_4^-$ tracer results from the batch contacts and column tests, are all consistent with the presence of (29±10)% pertechnetate ^{99}Tc in this feed, with the remainder present as a non-pertechnetate form that is not removed by the SL-639 resin.

The average maximum allowed concentration in Envelope C feed to meet the ILAW glass limit for ^{99}Tc (0.1 Ci/m^3) is expected to be 40 uCi/L (2.3 mg/L). Small column testing indicates that SL-639 will either barely provide, or not provide, sufficient ^{99}Tc decontamination to meet the limit in glass made from AN-107 waste. However, it was previously shown that an Envelope A waste (AW-101) can be ^{99}Tc decontaminated to a greater extent than needed to meet the ILAW limit. It is expected that sufficient ^{99}Tc can be removed from AW-101 and AN-107 wastes so that the <u>average</u> ^{99}Tc concentration in the ILAW glass produced from AW-101 and AN-107 wastes is less than or equal to the ILAW limit.

It is expected that this will be true in general – it will be possible to exceed the ^{99}Tc removal requirements for Envelope A and B wastes, so that the average ^{99}Tc concentrations in the ILAW glasses produced from Envelopes A, B, and C are less than or equal to the ILAW limit. The extent of ^{99}Tc removal possible in Envelope C waste will probably determine the required ^{99}Tc removal in Envelopes A and B. The concentration of ^{99}Tc in two other Envelope C wastes (241-SY-101 and 241-SY-103) is approximately four times higher than in AN-107, and the fraction of non-pertechnetate ^{99}Tc is expected to be nearly as high. Therefore it is recommended that flowsheet testing (including small column ^{99}Tc decontamination tests) of these and other Envelope C wastes be performed to determine the extent of ^{99}Tc removal possible in these wastes, and therefore the extent of ^{99}Tc removal required for Envelopes A and B.

Fouling of the resin bed was not observed.

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APPENDIX A

Appendix A: Sample Identification

Sample ID	Description
N7-Tc-0	Initial feed sample
N7-Tc-L1 through L17	Loading samples from lead column
N7-Tc-P1 through P9	Loading samples from lag column
AN-107 Tc IX Effluent – 1	Effluent composite container
N7-Tc-PW1 through PW9	Feed displacement samples
N7-Tc-PR1 and PR2	DI water rinse samples
N7-Tc-E1-1 through E1-45	Lead column eluate samples
N7-Tc-ELU-COMP	Lead column eluate composite sample
N7-Tc-E1-R1 through R4	Lead column eluant rinse samples
N7-Tc-REG-01	Regeneration effluent composite container
N7-Tc-R01	Regeneration effluent composite sample
Tc-SL0	Actual feed sample, reloading test
Tc-SL2 through SL14	Simulant loading samples, reloading test
Tc-SW1 through SW4	Simulant displacement samples, reloading test
AN-Tc-L1 through L25	Actual feed loading samples, reloading test
AN-Tc-W1 through W4	Regeneration samples, reloading test

APPENDIX B

Appendix B: Data Sheets, Analytical Data, and Calculations

Batch Contacts: AN-107 Contacted with Superlig-639 (SL-639)

Performed 9/24/99 through 10/1/99, as per "BNFL AN-107/Tc Batch Contact Test Instructions", BNFL-TI-29953-050.
The AN-107 is an archive sample from Cs IX performed by Doug Hendrickson and Dean Kurath at 222-S in 1996.
Untreated samples, and samples treated by a permanganate precipitation were tested.
The treated samples are denoted with a "P."
The Kd's were determined for a Tc-95m pertechnetate tracer ONLY. Tc-99 Kd's were NOT determined.
The bed density was determined in a previous round of experiments,
published in BNW report BNFL - RPT - 009.

Shaker type: Back and forth
Shaker frequency: 180 cycles/min

ID Number	N7	N7-39	N7-39D	N7P	N7P-39	N7P-39D
Material	SL-639	SL-639	SL-639	SL-639	SL-639	SL-639
Solution	Arcv. AN-107	Arcv. AN-107	Arcv. AN-107	Treated Archv. AN-107	Treated Archv. AN-107	Treated Archv. AN-107
Description	**Untreated Control**	**PR = 100**	**PR=100**	**Treated Control**	**PR=100**	**PR=100**
Start time	13:15	13:15	13:15	13:15	13:15	13:15
Start date	9/27/99	9/27/99	9/27/99	9/27/99	9/27/99	9/27/99
Stop time	13:15	13:15	13:15	13:15	13:15	13:15
Stop date	10/1/99	10/1/99	10/1/99	10/1/99	10/1/99	10/1/99
Total time	96 hrs	96 hrs	96 hrs	96 hrs	96 hrs	96 hrs
Liq Sample Mass, g	5.4817	6.1650	6.1828	5.6039	6.3033	6.3010
Liq Sample Density, g/mL	1.202	1.202	1.202	1.224	1.224	1.224
Liq Volume, mL	4.560	5.129	5.144	4.578	5.150	5.148
Temp, ° C	**24°C**	**24°C**	**24°C**	**24°C**	**24°C**	**24°C**
F Factor	-	0.9605	0.9605	-	0.9605	0.9605
Resin Mass	0	0.0497	0.0509	0	0.0525	0.0521
Phase Ratio (mL/g)	Control	103	101	Control	98	99
Fltr'd Sample Mass	5.2611	5.7875	5.8464	5.3184	5.8370	5.9060
Fltr'd Sample Vol	4.377	4.815	4.864	4.345	4.769	4.825
Tc-95m Feed Count (counts)	7447	7447	7447	5568	5568	5568
Tc-95m Sample Counts (counts)	7447	2196	1424	5568	1975	1823
Tc-95m Kd Value	-	**257**	**445**	-	**186**	**211**
Density	-	0.5	0.5	-	0.5	0.5
Tc-95m Lambda Value	-	**128**	**223**	-	**93**	**106**
% Tc-95m remaining	**100.00%**	**29.49%**	**19.12%**	**100.00%**	**35.47%**	**32.74%**
% Tc-95m removed	**0.00%**	**70.51%**	**80.88%**	**0.00%**	**64.53%**	**67.26%**

Batch Contacts: AN-107 Contacted with Superlig-639 (SL-639)

Performed 11/8/99 through 11/12/99, as per "BNFL AN-107/Tc Batch Contact Test Instructions (Using BNFL AN-107 Sample)", BNFL-TI-29953-068.
The AN-107 is from the BNFL sample that has had Sr/TRU ppt'd and Cs removed
The Kd's were determined for a Tc-95m pertechnetate tracer and for Tc-99.

Shaker type:	Back and forth
Shaker frequency:	180 cycles/min

Sample ID	**N7B**	**N7B-39**	**N7B-39D**
Material	SL-639	SL-639	SL-639
Solution	BNFL AN-107	BNFL AN-107	BNFL AN-107
Description	**Control**	**PR = 100**	**PR=100**
Start time	14:25	14:25	14:25
Start date	11/8/99	11/8/99	11/8/99
Stop time	14:50	14:50	14:50
Stop date	11/12/99	11/12/99	11/12/99
Total time	96 hrs	96 hrs	96 hrs
Liq Sample Mass, g	6.1896	6.2036	6.2031
Liq Sample Density, g/mL	1.241	1.241	1.241
Liq Volume, mL	4.986	4.997	4.997
Temp, ° C	24°C - 25°C	24°C - 25°C	24°C - 25°C
F Factor	-	0.956	0.956
Resin Mass	0	0.0492	0.0493
Phase Ratio (mL/g)	Control	102	101
Count Sample Mass	5.9702	5.9108	5.9296
Count Sample Vol	4.809	4.761	4.777
Tc-95m (Pertechnetate)			
Tc-95m Counts (net)	121538	26120	52099
Tc-95m Count Time (sec)	721	1200	1200
Tc-95m Activity (counts/g/sec)	28	4	7
Kd Value	-	**708**	**303**
Density	-	0.5	0.5
Lambda Value	-	**354**	**151**
% Tc-95m remaining	**100.00%**	**13.04%**	**25.94%**
% Tc-95m removed	**0.00%**	**86.96%**	**74.06%**
Tc-99 (Total Tc)			
Tc-99 Feed Conc (ng/g)	3091	3091	3091
Tc-99 Sample Conc (ng/g)	3091	2402	2743
Tc-99 Kd	-	**30**	**13**
Density	-	0.5	0.5
Tc-99 Lambda	-	**15**	**7**
% Tc-99 remaining	**100.00%**	**77.71%**	**88.74%**
% Tc-99 removed	**0.00%**	**22.29%**	**11.26%**

TI Number	**29953-068**
Balance No.	**362-06-01-040**
Next Calib.	**2/1/00**

Testing of SL-639 with AN-107 feed for Tc removal.

Tc-95m tracer used to follow progress of various process steps.

Initial Loading with AN-107 Feed

Start Date and Time:	11/4/99	6:08
Bed volume col 1=	4.7	mL
Bed volume col 2=	4.7	mL
Empty Eff Btl w/ cap(w / hole) =	251.5	g
Bed volumes diverted to waste =		BV
Supernate Density =	1.2414	(g/ml)

Feed analyses: Tc-99, uCi/mL

Tracer: Tc-95m, activity determined at various times given in table.

Total feed vo 1.5 L

Feed processed: 0.8 L

Pump Speed: 60 (approx 30 mL/hr or 6 CV/hr)

The effluent volumes on which the bed volumes are based were determined by determining the total mass and calculating the volume of waste pumped through the columns at the time of sampling. For column #1 this includes the mass of the waste in the effluent bottle + the samples taken from column # 1 and # 2 prior to the sample of interest (the mass of the effluent bottle was recorded during each sample event). For column # 2 the effluent volume includes the mass of the waste in the effluent bottle + the samples taken from column #2 prior to the the sample of interest (samples taken from column # 1 did not go through column # 2.

The system holdup is estimated to be 2 BV to the sample valve for the 1st column, and 3 BV to the sample valve for the 2nd column. The effluent was diverted into the waste jug for 10 minutes with the pump set at 50 mL/hr (controller set at 99) at the start of the loading cycle to flush out the holdup volume. The mass of effluent from this step was not determined, but from the pump speed and the duration, it must have been approx 8.3 mL = 1.8 BV. So given the estimates for the holdup, the BV scale should be about right for the 1st column samples, and 1 BV too high for the 2nd column samples.

Vial	Sample date	sample time (hr:mm:s)	elapsed time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Count date	Count start time (*)	file name	count time min	gross counts	Net counts	back ground	net cts-bck	counts/g of sample/min	C/Co, Tc-95m	Eff Btl Mass (g)
N7-Tc-0	11/3/99	9:00:00	0.000	16.8343	30.0000	13.1657	10.6055	11/4/99	8:43:21	N7TcF02	3	91897	77109	0	77109	1952	1.000	251.5
N7-Tc-0				16.8343	30.0000	13.1657	10.6055	11/4/99	14:28:24	N7TcF03	3	91820	75880	0	75880	1921	1.000	-
N7-Tc-0 Co Count	11/5/99	-	-	16.6837	20.2082	3.5245	2.8391	11/4/99	18:57:26	N7TcF04	3	25321	20370	0	20370	1927		
N7-Tc-0 Co Count	11/5/99	-	-	16.6837	20.2082	3.5245	2.8391	11/4/99	19:02:20	N7TcF05	5	42118	33920	0	33920	1925		
N7-Tc-0 Co Count	11/5/99	-	-	16.6837	20.2082	3.5245	2.8391	11/4/99	19:09:02	N7TcF06	15	125342	102395	0	102395	1937		
average						3.5245					23		156685		156685	1933		
N7-Tc-0 Co Count	11/5/99	-	-	16.6837	20.2082	3.5245	2.8391	11/5/99	4:09:23	N7TcF07	3	25089	20264	0	20264	1916		
N7-Tc-0 Co Count	11/5/99	-	-	16.6837	20.2082	3.5245	2.8391	11/5/99	8:26:39	N7TcF09	5	40522	32635	0	32635	1852		
N7-Tc-0 Co Count	11/5/99	-	-	16.6837	20.2082	3.5245	2.8391	11/5/99	11:40:56	N7TcF10	10	80272	64497	0	64497	1830		
Start loading	11/4/99	6:08:00	0.000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
N7-Tc-L1	11/4/99	7:51:30	1.725	16.9392	19.1523	2.2131	1.7827	11/4/99	8:55:45	N7TcL01	3	3375	1292	0	1292	195	0.101	310.3
N7-Tc-L2	11/4/99	9:30:30	3.375	16.8328	19.3106	2.4778	1.9960	11/4/99	9:14:25	N7TcL02	3	4157	1990	0	1990	268	0.139	365.2
N7-Tc-L3	11/4/99	11:11:10	5.053	16.6814	18.3529	1.6715	1.3465	11/4/99	10:40:52	N7TcL03	3	3490	1782	0	1782	355	0.184	420.9
N7-Tc-L4	11/4/99	12:50:00	6.700	16.7175	18.8517	2.1342	1.7192	11/4/99	12:35:18	N7TcL04	3	4699	2579	0	2579	403	0.208	474.4
N7-Tc-L5	11/4/99	14:41:25	8.557	16.8918	19.1114	2.2196	1.7880	11/4/99	14:21:48	N7TcL05	3	5497	3110	0	3110	467	0.242	537.2
N7-Tc-L6	11/4/99	16:10:00	10.033	16.8846	19.1847	2.3001	1.8528	11/4/99	16:47:51	N7TcL06	3	6065	3642	0	3642	528	0.273	584.8
N7-Tc-L7	11/4/99	17:50:00	11.700	16.7720	18.9198	2.1478	1.7301	11/4/99	17:22:09	N7TcL07	3	6157	3613	0	3613	561	0.290	641.1
N7-Tc-L8	11/4/99	19:30:00	13.367	16.7638	19.0699	2.3061	1.8577	11/4/99	18:50:57	N7TcL08	3	7284	4900	0	4900	708	0.366	695.7
N7-Tc-L9	11/4/99	21:10:00	15.033	16.6923	19.0172	2.3249	1.8728	11/4/99	20:23:30	N7TcL09	3	7957	5351	0	5351	767	0.397	751.8
N7-Tc-L10	11/4/99	22:53:00	16.750	16.7480	19.1329	2.3849	1.9211	11/4/99	22:07:52	N7TcL10	3	8566	6033	0	6033	843	0.436	807.8
N7-Tc-L11	11/5/99	0:30:00	18.367	16.8182	19.1319	2.3137	1.8638	11/5/99	23:51:28	N7TcL11	3	8794	6078	0	6078	876	0.453	862.6
N7-Tc-L12	11/5/99	2:10:00	20.033	16.8375	19.1867	2.3492	1.8924	11/5/99	1:25:04	N7TcL12	3	9462	6664	0	6664	946	0.493	917.0
N7-Tc-L13	11/5/99	3:50:00	21.700	16.7527	19.0353	2.2826	1.8387	11/5/99	3:13:43	N7TcL13	3	9872	7138	0	7138	1042	0.544	974.0
N7-Tc-L14	11/5/99	5:30:00	23.367	16.7378	19.0125	2.2747	1.8324	11/5/99	4:51:05	N7TcL14	3	9716	6999	0	6999	1026	0.535	1028.4
N7-Tc-L15	11/5/99	7:10:00	25.033	16.8595	19.1426	2.2831	1.8391	11/5/99	7:01:30	N7TcL15	5	17343	13031	0	13031	1142	0.596	1085.4
N7-Tc-L16	11/5/99	8:50:00	26.700	16.6922	18.9687	2.2765	1.8338	11/5/99	8:11:07	N7TcL16	5	17823	12898	0	12898	1133	0.612	1139.4
N7-Tc-L17	11/5/99	10:12:45	28.079	16.8111	19.2257	2.4146	1.9451	11/5/99	10:43:47	N7TcL17	5	19042	14429	0	14429	1195	0.653	1185.5

Vial	Sample date	sample time (hr:mm:s)	elapsed time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Date of counting	Time of counting	file name	count time min	gross counts	Net counts	back ground	net cnts - bck	net counts/g of sample counted/min	C/Co,Tc-95m	Eff Btl Mass (g)
Start loading	11/4/99	6:08:00	0.000	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
N7-Tc-P1	11/4/99	7:55:30	1.792	16.5089	18.7718	2.2629	1.8229	11/4/99	9:03:42	N7TcP01	3	2124	177	0	177	26	0.013	310.3
N7-Tc-P2	11/4/99	11:14:20	5.106	16.7324	18.9435	2.2111	1.7811	11/4/99	10:46:26	N7TcP02	3	2328	367	0	305	46	0.024	420.9
N7-Tc-P3	11/4/99	14:45:25	8.624	16.7078	19.0026	2.2948	1.8486	11/4/99	14:16:53	N7TcP03	3	2520	503	0	503	73	0.038	537.2
N7-Tc-P4	11/4/99	17:54:00	11.767	16.7380	18.9557	2.2177	1.7865	11/4/99	17:27:00	N7TcP04	3	2784	719	0	719	108	0.056	641.1
N7-Tc-P5	11/4/99	21:14:00	15.100	16.7480	18.9428	2.1948	1.7680	11/4/99	20:32:32	N7TcP05	3	3219	1291	0	1291	196	0.101	751.8
N7-Tc-P6	11/4/99	0:34:00	18.433	16.8595	19.0628	2.2033	1.7749	11/05/99	23:56:33	N7TcP06	3	3806	1529	0	1529	231	0.120	862.6
N7-Tc-P7	11/5/99	3:54	21.767	16.8470	19.1258	2.2788	1.8357	11/05/99	3:20:05	N7TcP07	3	4340	1935	0	1935	283	0.146	974
N7-Tc-P8	11/5/99	7:10:00	25.033	16.8338	19.1559	2.3221	1.8705	11/05/99	8:42:55	N7TcP08	5	8232	4580	0	4580	394	0.204	1085.4
N7-Tc-P9	11/5/99	10:16:45	28.146	16.9904	19.1906	2.2002	1.7724	11/05/99	10:34:19	N7TcP09	5	8963	5229	0	5229	475	0.246	1185.5

Testing of SL-639

Tc-95m tracer used

Initial Loading with

Tc-95m Decay Calculator

Enter initial amount or activity and the elapsed (decay) time in days,

and the expected Final Amount is given.

Isotope	Half life (d)	Initial Amount (any unit)	Decay time (d)	Final Amount (same unit)
Tc-95m	61	218	127	51.48988389

Vial	Effluent Mass+ samples (g)	Eff Vol (mL)	Bed volumes	flow rate BV/hr	flow rate mL/hr	ICP-MS Tc-99 ng/mL	ICP-MS Tc-99 C/Co	Notes
N7-Tc-0	0	0.0	0.0	-	-	3340		
N7-Tc-0	-	-	-	-	-	-	-	
N7-Tc-0 Co Count								
N7-Tc-0 Co Count								
N7-Tc-0 Co Count								
average								
N7-Tc-0 Co Count								
N7-Tc-0 Co Count								
N7-Tc-0 Co Count								
Start loading	-	-	-	-	-	-	-	Start loading, feed at top of 1st column.
N7-Tc-L1	61.0	49.1	10.5	6.1	28.5	4792	1.435	Max Tc-95m DF = 9.932575 Max Tc-99 DF = 0.69702
N7-Tc-L2	120.7	97.2	20.7	6.2	29.1			
N7-Tc-L3	178.0	143.4	30.5	5.9	27.5	4544	1.360	
N7-Tc-L4	235.9	190.0	40.4	6.0	28.3			
N7-Tc-L5	300.9	242.4	51.6	6.0	28.2	4630	1.386	
N7-Tc-L6	353.1	284.4	60.5	6.1	28.5			
N7-Tc-L7	411.5	331.5	70.5	6.0	28.2	4320	1.293	
N7-Tc-L8	470.7	379.1	80.7	6.1	28.6			
N7-Tc-L9	529.1	426.2	90.7	6.0	28.2	4407	1.319	
N7-Tc-L10	589.7	475.0	101.1	6.0	28.4			
N7-Tc-L11	646.8	521.0	110.9	6.1	28.5	4072	1.219	
N7-Tc-L12	705.7	568.5	121.0	6.1	28.5			
N7-Tc-L13	765.0	616.2	131.1	6.1	28.7	4121	1.234	
N7-Tc-L14	824.0	663.7	141.2	6.1	28.5			
N7-Tc-L15	885.6	713.4	151.8	6.3	29.8	4084	1.223	
N7-Tc-L16	946.2	762.2	162.2	6.2	29.3			
N7-Tc-L17	994.8	801.3	170.5	6.0	28.3	4221	1.264	

Vial	Effluent Mass+ sample (g)	Eff Vol (mL)	Bed volumes	flow rate BV/hr	flow rate mL/hr	ICP-MS Tc-99	C/Co Tc-99	
Start loading	-	-	-	-	-	-	-	*Start loading, feed at top of 1st column.*
N7-Tc-P1	61.1	49.2	10.5	5.8	27.5	3265	0.978	Max Tc-95m DF = 74.13366 Max Tc-99 DF = 1.02301
N7-Tc-P2	173.9	140.1	29.8	5.8	27.4			
N7-Tc-P3	292.5	235.6	50.1	5.8	27.2	3215	0.963	
N7-Tc-P4	398.6	321.1	68.3	5.8	27.2			
N7-Tc-P5	511.5	412.0	87.7	5.8	27.3	4072	1.219	
N7-Tc-P6	624.5	503.0	107.0	5.8	27.3			
N7-Tc-P7	738.2	594.6	126.5	5.8	27.5	3824	1.145	
N7-Tc-P8	851.9	686.2	146.0	6.0	28.0			
N7-Tc-P9	954.2	768.6	163.5	5.6	26.5	4233	1.267	

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q
66																	
67	**Effluent Composite Analysis**																
68	Vial	Sample date	sample time (hr:mm:s)	elapsed time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Date of counting	Time of counting	file name	count time min	gross counts	Net counts	back ground	net cnts - bck	net counts/g of sample/min
69																	
70	N7-Tc-0 Co Count	11/5/99	-	-	16.6837	20.2082	3.5245	2.8391	11/10/99	0:03:12	N7TcF18	5	76896	62778	0	62778	3562
71																	
72	N7-Tc-EFF-COMP	11/9/99	N/A	N/A	16.8556	23.5769	6.7213	5.4143	11/09/99	22:56:10	N7TcEFF	10	22588	10659	0	10659	159
73																	
74																	
75	**Feed Displacement: 0.1 M NaOH**																
76																	
77		Start Date and Time:		11/5/99	10:40	AM											
78		*Density of 0.1 M NaOH @ 25*		1.001	*g/mL*												
79		Pump Speed:		31 (approx 13.8 mL/hr or 3 CV/hr)													
80	Vial/Bottle	Sample date	sample start time (hr:mm:s)	sample finish time	Sampling time (hr)	mass of vial + cap	mass of vial + cap + sample	Sample Mass	Sample Vol	Eff Vol (mL)	Flow Rate, mL/hr	Eff Vol (BV)	Flow Rate, BV/hr	Date of counting	Time of counting	file name	count time, min
81	N7-Tc-0 Co Count					16.6837	20.2082	3.5245	2.8391	0.000	0.000	0.00	0.00	11/5/99	11:40:56	N7TcF10	10
82																	
83	N7-Tc-PW1	11/5/99	10:40	11:00	0.333	16.7635	22.5024	5.7389	4.6229	4.62	13.87	0.98	2.95	11/05/99	10:52:28	N7TcPW01	5
84	N7-Tc-PW2	11/5/99	11:00	11:20	0.333	16.8011	22.5076	5.7065	4.5968	9.22	13.79	1.96	2.93	11/05/99	10:59:12	N7TcPW02	5
85	N7-Tc-PW3	11/5/99	11:20	11:40	0.333	16.7784	22.4221	5.6437	4.5462	13.77	13.64	2.93	2.90	11/05/99	11:05:57	N7TcPW03	5
86	N7-Tc-PW4	11/5/99	11:40	12:00	0.333	16.9824	22.1754	5.1930	5.1878	18.95	15.56	4.03	3.31	11/05/99	11:14:56	N7TcPW04	5
87	N7-Tc-PW5	11/5/99	12:00	12:20	0.333	17.0028	21.767	4.7642	4.7594	23.71	14.28	5.05	3.04	11/05/99	11:29:26	N7TcPW05	5
88	N7-Tc-PW6	11/5/99	12:20	12:40	0.333	16.7560	21.4153	4.6593	4.6546	28.37	13.96	6.04	2.97	11/05/99	11:55:17	N7TcPW06	5
89	N7-Tc-PW7	11/5/99	12:40	13:00	0.333	16.7200	21.288	4.5680	4.5634	32.93	13.69	7.01	2.91	11/05/99	12:11:35	N7TcPW07	5
90	N7-Tc-PW8	11/5/99	13:00	13:10	0.167	16.8542	18.9955	2.1413	2.1392	35.07	12.83	7.46	2.73	11/05/99	12:20:43	N7TcPW07	5
91				Total time	2.500			Avg vol:	4.7045				2.97				
92																	
93	**DI water rinse**																
94		start date and time		7/1/99	5:40												
95		Pump speed:		60 (approx 30 mL/hr or 6 CV/hr)													
96																	
97	Vial/Bottle	Sample date	sample start time (hr:mm:s)	sample finish time	Sampling time (hr)	mass of vial + cap	mass of vial + cap + sample	Sample Mass	Sample Vol	Eff Vol (mL)	Flow Rate, mL/hr	Eff Vol (BV)	Flow Rate, BV/hr	Date of counting	Time of counting	file name	count time, min
98	*N7-Tc-0 Co Count*					16.6837	20.2082	3.5245	2.8391	0.000	0.000	0.00	0.00	11/8/99	7:29:58	N7TcF11	20
99																	
100	N7-Tc-PR1	11/8/99	8:20	8:40	0.333	16.8211	21.7137	4.8926	4.8926	4.89	14.68	1.04	3.12	11/8/99	7:55:07	N7TcPR01	10
101	N7-Tc-PR2	11/8/99	8:40	9:00	0.333	16.8990	21.6989	4.7999	4.7999	9.69	14.40	2.06	3.06	11/8/99	8:16:08	N7TcPR02	10

	A	R	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF
66																
67	**Effluent Composite**															
68	Vial	C/Co,Tc-95m	Eff Bll Mass (g)	Effluent Mass+ sample (g)	Eff Vol (mL)	Bed volumes	flow rate BV/hr	flow rate mL/hr	ICP-MS Tc-99	C/Co Tc-99	DF Tc-99	Activity, Tc-99 (uCi/L)	Max Tc-99 Activity in Vit Feed, Env C (mg/L)	Max Tc-99 Activity in Vit Feed, Env C (uCi/L)	Actual:Max Tc-99	
69																
70	N7-Tc-0 Co Count	1.000		N/A	N/A	N/A	N/A	N/A	3340							
71																
72	N7-Tc-EFF-COMP	0.045	N/A	N/A	768.6	163.5	N/A	N/A	3470	1.039	0.963	59.3	1.93	33	1.798	
73																
74																
75	**Feed Displacement**															
76																
77																
78																
79																
80	Vial/Bottle	back-ground	Tot Counts	Net Area	net cnts per g of sample per min	net cnts per mL of sample per min	C/Co, Tc-95m	Tc-99, ICP-MS	C/Co, Tc-99	Notes						
81	N7-Tc-0 Co Count	0	80272	64497	1830	2272	1.000	3340	1.000							
82																
83	N7-Tc-PW1	0	19102	12755	445	552	0.243	3985	1.193							
84	N7-Tc-PW2	0	17848	10961	384	477	0.210									
85	N7-Tc-PW3	0	17925	11955	424	526	0.232	3960	1.186							
86	N7-Tc-PW4	0	16235	11455	441	442	0.194			The color started to lighten with this sample. Density probably isn't correct.						
87	N7-Tc-PW5	0	16444	12674	532	533	0.234	1612	0.483	Density probably isn't correct.						
88	N7-Tc-PW6	0	23565	19630	843	843	0.371			Density probably isn't correct.						
89	N7-Tc-PW7	0	31501	26978	1181	1182	0.520	2482	0.743							
90	N7-Tc-PW8	0	16484	13418	1253	1255	0.552									
91										0.23						
92																
93	**DI water rinse**															
94																
95																
96																
97	Vial/Bottle	background	Total counts	Net Area	net cnts per g of sample per min	net cnts per mL of sample per min	C/Co, Tc-95m	Tc-99, ICP-MS	C/Co, Tc-99							
98	N7-Tc-0 Co Count	0	155695	126121	1789	2221	1.000	3340	1.000							
99																
100	N7-Tc-PR1	0	152759	133040	2719	2719	1.22	3140	0.940							
101	N7-Tc-PR2	0	157274	137656	2868	2868	1.29	3460	1.036							

104 Elution - 1st Column

106 Start Date and Time: 7/1/99 8:21

107 Pump speed (start): 11 (approx 5 mL/hr or 1 CV/hr)

108 eluant (water) density @ 24 °C = 0.9973 g/mL

	Vial/Bottle	Sample date	sample start time (hr:mm:s)	sample finish time	Sampling time (hr)	mass of vial + cap, g	mass of vial + cap + sample, g	Mass of sample, g	Vol of Sample (mL)	Eff Vol (mL)	Flow Rate, mL/hr	Eff Vol (BV)	Flow Rate, BV/hr	Date of counting	Time of counting	file name	count time, min	background	Total counts	Net Area	net counts/ g sample/ minute
109																					
110	N7-Tc-0 (after subsample)	11/8/99				16.8343	26.4687	9.6344	7.7609	0.000	0.000	0.00	0.00	11/8/99	13:59:41	N7TcF12	10	0	26789	19287	200
111	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/8/99	18:46:26	N7TcF13	10	0	77516	63516	1802
112	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/9/99	5:03:36	N7TcF14	10	0	76333	62313	1768
113	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/9/99	12:00:31	N7TcF15	10	0	78064	64165	1821
114	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/9/99	12:28:08	N7TcF16	10	0	77603	63783	1810
115	Avg																		77834	63974	1815
116	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/9/99	16:52:47	N7TcF17	10	0	77164	63256	1795
117	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/10/99	0:03:12	N7TcF18	10	0	76896	62778	1781
118	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/10/99	6:40:50	N7TcF19	10	0	76303	62175	1764
119	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/10/99	12:15:35	N7TcF20	10	0	76320	62328	1768
120	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/10/99	15:57:54	N7TcF21	10	0	75561	61111	1734
121																					
122																					
123																					
124																					
125	N7-Tc-E1-1	11/8/99	11:40	12:40	1.000	16.8486	21.4282	4.5796	4.5920	4.59	4.59	0.98	0.98	11/8/99	14:12:14	N7Tc1E01	10	0	172281	146317	3195
126	N7-Tc-E1-2		12:40	13:40	1.000	17.0436	21.2799	4.2363	4.2478	8.84	4.25	1.88	0.90	11/8/99	14:34:42	N7Tc1E02	10	0	629726	541732	12788
127	N7-Tc-E1-3		13:40	14:40	1.000	16.9196	21.2217	4.3021	4.3137	13.15	4.31	2.80	0.92	11/8/99	14:52:17	N7Tc1E03	10	0	279178	238832	5552
128	N7-Tc-E1-4		14:40	15:40	1.000	16.8117	21.9652	5.1535	5.1675	18.32	5.17	3.90	1.10	11/8/99	16:14:01	N7Tc1E04	10	0	93515	77819	1510
129	N7-Tc-E1-5		15:40	16:40	1.000	16.8757	22.0566	5.1809	5.1949	23.52	5.19	5.00	1.11	11/8/99	16:00:24	N7Tc1E05	10	0	45414	36107	697
130	N7-Tc-E1-6		16:40	17:42	1.033	16.8876	21.7988	4.9112	4.9245	28.44	4.77	6.05	1.01	11/8/99	17:22:01	N7Tc1E06	10	0	29247	22507	458
131	N7-Tc-E1-7		17:42	18:40	0.967	16.8872	21.3708	4.4836	4.4957	32.94	4.65	7.01	0.99	11/8/99	17:55:49	N7Tc1E07	10	0	21228	15478	345
132	N7-Tc-E1-8		18:40	19:40	1.000	16.7696	21.4545	4.6849	4.6976	37.63	4.70	8.01	1.00	11/8/99	19:00:07	N7Tc1E08	10	0	133823	117045	2498
133	N7-Tc-E1-9		19:40	20:40	1.000	16.9720	21.5504	4.5784	4.5908	42.22	4.59	8.98	0.98	11/8/99	19:50:51	N7Tc1E09	10	0	106590	92132	2012
134	N7-Tc-E1-10		20:40	21:40	1.000	16.6729	21.2653	4.5924	4.6048	46.83	4.60	9.96	0.98	11/8/99	20:51:58	N7Tc1E10	10	0	91146	78172	1702
135	N7-Tc-E1-11		21:40	22:40	1.000	16.9719	21.6197	4.6478	4.6604	51.49	4.66	10.96	0.99	11/8/99	21:53:13	N7Tc1E11	10	0	80414	69236	1490
136	N7-Tc-E1-12		22:40	23:40	1.000	16.8638	21.4881	4.6243	4.6368	56.13	4.64	11.94	0.99	11/8/99	22:52:53	N7Tc1E12	10	0	72041	61662	1333
137	N7-Tc-E1-13		23:40	0:40	1.000	16.8150	22.0033	5.1883	5.2023	61.33	5.20	13.05	1.11	11/8/99	23:50:37	N7Tc1E13	10	0	69079	58681	1131
138	N7-Tc-E1-14		0:40	1:40	1.000	16.8040	22.0092	5.2052	5.2193	66.55	5.22	14.16	1.11	11/9/99	0:57:54	N7Tc1E14	10	0	59581	49890	958
139	N7-Tc-E1-15		1:40	2:40	1.000	16.7210	21.9178	5.1968	5.2109	71.76	5.21	15.27	1.11	11/9/99	1:52:07	N7Tc1E15	10	0	52971	44259	852
140	N7-Tc-E1-16		2:40	3:40	1.000	16.8817	21.5296	4.6479	4.6605	76.42	4.66	16.26	0.99	11/9/99	2:53:57	N7Tc1E16	10	0	43867	36430	784
141	N7-Tc-E1-17		3:40	4:40	1.000	16.8507	21.4398	4.5891	4.6015	81.02	4.60	17.24	0.98	11/9/99	3:52:15	N7Tc1E17	10	0	44827	37263	812
142	N7-Tc-E1-18		4:40	5:40	1.000	16.7897	21.4293	4.6396	4.6522	85.67	4.65	18.23	0.99	11/9/99	4:51:07	N7Tc1E18	10	0	41602	34119	735
143	N7-Tc-E1-19		5:40	6:40	1.000	16.7591	21.3897	4.6306	4.6431	90.32	4.64	19.22	0.99	11/9/99	6:09:40	N7Tc1E19	10	0	38202	31589	682
144	N7-Tc-E1-20		6:40	7:40	1.000	16.7655	21.4015	4.6360	4.6486	94.96	4.65	20.21	0.99	11/9/99	6:52:20	N7Tc1E20	10	0	35223	28180	608
145	N7-Tc-E1-21		7:40	9:16	1.600	16.8337	23.7134	6.8797	6.8983	101.86	4.31	21.67	0.92	11/9/99	8:27:10	N7Tc1E21	10	0	44946	36784	535
146	N7-Tc-E1-22		9:16	10:15	0.983	16.7960	21.9685	5.1725	5.1865	107.05	5.27	22.78	1.12	11/9/99	9:31:23	N7Tc1E22	10	0	54096	45384	877
147	N7-Tc-E1-23		10:15	11:15	1.000	16.9126	21.9445	5.0319	5.0455	112.10	5.05	23.85	1.07	11/9/99	10:28:15	N7Tc1E23	10	0	53772	45592	906
148	N7-Tc-E1-24		11:15	12:15	1.000	16.8091	21.9428	5.1337	5.1476	117.24	5.15	24.95	1.10	11/9/99	11:26:05	N7Tc1E24	10	0	46550	38381	748
149	N7-Tc-E1-25		12:15	13:15	1.000	16.7787	21.9141	5.1354	5.1493	122.39	5.15	26.04	1.10	11/9/99	12:53:29	N7Tc1E25	10	0	39936	32820	639
150	N7-Tc-E1-26		13:15	14:15	1.000	16.9013	22.0555	5.1542	5.1682	127.56	5.17	27.14	1.10	11/9/99	13:25:03	N7Tc1E26	10	0	34988	27908	541
151	N7-Tc-E1-27		14:15	15:15	1.000	16.8385	21.9684	5.1299	5.1438	132.70	5.14	28.23	1.09	11/9/99	14:24:59	N7Tc1E27	10	0	31356	25028	488
152	N7-Tc-E1-28		15:15	16:15	1.000	16.6474	21.2679	4.6205	4.6330	137.34	4.63	29.22	0.99	11/9/99	15:25:02	N7Tc1E28	10	0	26318	20420	442
153	N7-Tc-E1-29		16:15	17:16	1.017	16.9074	21.6447	4.7373	4.7501	142.09	4.67	30.23	0.99	11/9/99	16:40:06	N7Tc1E29	10	0	25474	19889	420
154	N7-Tc-E1-30		17:16	18:16	1.000	16.8451	21.4857	4.6406	4.6532	146.74	4.65	31.22	0.99	11/9/99	18:28:41	N7Tc1E30	10	0	24065	18590	401
155	N7-Tc-E1-31		18:16	19:16	1.000	16.9046	21.4945	4.5899	4.6023	151.34	4.60	32.20	0.98	11/9/99	19:24:15	N7Tc1E31	10	0	22757	16960	370
156	N7-Tc-E1-32		19:16	20:16	1.000	16.6994	21.2915	4.5921	4.6045	155.95	4.60	33.18	0.98	11/9/99	20:42:29	N7Tc1E32	10	0	21388	15821	345
157	N7-Tc-E1-33		20:16	21:16	1.000	16.9159	21.4926	4.5767	4.5891	160.54	4.59	34.16	0.98	11/9/99	21:27:05	N7Tc1E33	10	0	20740	15897	347
158	N7-Tc-E1-34		21:16	22:16	1.000	16.8189	21.4004	4.5815	4.5939	165.13	4.59	35.13	0.98	11/9/99	22:26:25	N7Tc1E34	10	0	19398	14253	311
159	N7-Tc-E1-35		22:16	23:16	1.000	16.8807	21.3941	4.5134	4.5256	169.66	4.53	36.10	0.96	11/9/99	22:42:52	N7Tc1E35	10	0	18912	14033	311
160	N7-Tc-E1-36		23:16	0:16	1.000	16.8993	21.4691	4.5698	4.5822	174.24	4.58	37.07	0.97	11/9/99	23:30:57	N7Tc1E36	10	0	18300	13109	287
161	N7-Tc-E1-37		7:26	8:30	1.067	16.9958	22.5897	5.5939	5.6090	179.85	5.26	38.27	1.12	11/10/99	7:40:40	N7Tc1E37	10	0	33597	26930	481
162	N7-Tc-E1-38		8:30	9:31	1.017	16.6397	21.4578	4.8181	4.8311	184.68	4.75	39.29	1.01	11/10/99	10:56:21	N7Tc1E38	10	0	46749	38606	801
163	N7-Tc-E1-39		9:31	10:30	0.983	16.8811	21.4052	4.5241	4.5363	189.21	4.61	40.26	0.98	11/10/99	11:10:12	N7Tc1E39	10	0	31916	25432	562
164	N7-Tc-E1-40		10:30	11:32	1.0333	16.8536	21.5546	4.7010	4.7137	193.93	4.56	41.26	0.97	11/10/99	11:26:06	N7Tc1E40	10	0	27205	21180	451
165	N7-Tc-E1-41		11:32	12:30	0.9667	16.6922	21.1718	4.4796	4.4917	198.42	4.65	42.22	0.99	11/10/99	11:41:58	N7Tc1E41	10	0	23163	17303	386
166	N7-Tc-E1-42		12:30	13:30	1.0000	16.7997	21.3284	4.5287	4.5410	202.96	4.54	43.18	0.97	11/10/99	12:38:30	N7Tc1E42	10	0	22196	16343	361
167	N7-Tc-E1-43		13:30	14:30	1.0000	16.8361	21.3815	4.5454	4.5577	207.52	4.56	44.15	0.97	11/10/99	13:39:06	N7Tc1E43	10	0	20144	14828	326
168	N7-Tc-E1-44		14:30	15:30	1.0000	16.9431	21.4916	4.5485	4.5608	212.08	4.56	45.12	0.97	11/10/99	14:39:41	N7Tc1E44	10	0	18768	13755	302
169	N7-Tc-E1-45		15:30	16:30	1.0000	16.9425	21.653	4.7105	4.7233	216.80	4.72	46.13	1.00	11/10/99	15:45:05	N7Tc1E45	10	0	18553	13444	285
170					45.667		Total:	216.2174	216.8028		213.87		45.50								
171							Avg:	4.805	4.818		4.753		1.01								

	A	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH
104	Elution - 1st Colur													
105														
106														
107														
108														
109	Vial/Bottle	net cnts per mL of sample per min	C/Co, Tc-95m	Tc-99, ICP-MS	C/Co, Tc-99	Comments								
110	N7-Tc-0 (after subsample)	249	1.000	3340		Tc-95mm counted in back right sample position - farthest from detector.								
111	N7-Tc-0 (small subsample)	2237	1.000			Tc-95m counting location move to rear first (left) position								
112	N7-Tc-0 (small subsample)	2195	1.000			Rear position								
113	N7-Tc-0 (small subsample)	2260	1.000			Rear position								
114	N7-Tc-0 (small subsample)	2247	1.000			Rear position								
115	Avg	2253	1.000											
116	N7-Tc-0 (small subsample)	2228	1.000											
117	N7-Tc-0 (small subsample)	2211	1.000											
118	N7-Tc-0 (small subsample)	2190	1.000											
119	N7-Tc-0 (small subsample)	2195	1.000											
120	N7-Tc-0 (small subsample)	2152	1.000											
121														
122														
123														
124														
125	N7-Tc-E1-1	3186	12.822	32100	9.61078	Tc-95m counted in back right sample position - farthest from detector. Same with subsequent samples.								
126	N7-Tc-E1-2	12753	51.318	90700	27.1557									
127	N7-Tc-E1-3	5537	22.279	29800	8.92216									
128	N7-Tc-E1-4	1506	6.060	7330	2.19461	Pump speed increased to 12								
129	N7-Tc-E1-5	695	2.797											
130	N7-Tc-E1-6	457	1.839			Pump speed decreased to 11								
131	N7-Tc-E1-7	344	1.385											
132	N7-Tc-E1-8	2492	1.114	1110	0.33234	Tc-95m counting location move to rear first (left) position								
133	N7-Tc-E1-9	2007	0.897											
134	N7-Tc-E1-10	1698	0.759											
135	N7-Tc-E1-11	1486	0.664											
136	N7-Tc-E1-12	1330	0.594	633	0.18952									
137	N7-Tc-E1-13	1128	0.504											
138	N7-Tc-E1-14	956	0.436											
139	N7-Tc-E1-15	849	0.387											
140	N7-Tc-E1-16	782	0.356	447	0.13383									
141	N7-Tc-E1-17	810	0.369											
142	N7-Tc-E1-18	733	0.334											
143	N7-Tc-E1-19	680	0.310											
144	N7-Tc-E1-20	606	0.276	364	0.10898									
145	N7-Tc-E1-21	533	0.243											
146	N7-Tc-E1-22	875	0.388											
147	N7-Tc-E1-23	904	0.401	452	0.13533									
148	N7-Tc-E1-24	746	0.331											
149	N7-Tc-E1-25	637	0.283											
150	N7-Tc-E1-26	540	0.240											
151	N7-Tc-E1-27	487	0.216	255	0.07635									
152	N7-Tc-E1-28	441	0.198											
153	N7-Tc-E1-29	419	0.188											
154	N7-Tc-E1-30	400	0.179											
155	N7-Tc-E1-31	369	0.165	214	0.06407									
156	N7-Tc-E1-32	344	0.154											
157	N7-Tc-E1-33	346	0.157											
158	N7-Tc-E1-34	310	0.140											
159	N7-Tc-E1-35	310	0.140	167	0.05									
160	N7-Tc-E1-36	286	0.129											
161	N7-Tc-E1-37	480	0.219											
162	N7-Tc-E1-38	799	0.364	439	0.13144									
163	N7-Tc-E1-39	561	0.255											
164	N7-Tc-E1-40	449	0.205											
165	N7-Tc-E1-41	385	0.175											
166	N7-Tc-E1-42	360	0.164	180	0.05389									
167	N7-Tc-E1-43	325	0.148											
168	N7-Tc-E1-44	302	0.140											
169	N7-Tc-E1-45	285	0.132	160	0.0479	2.736 uCi/L Tc-99								
170														
171														

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
173															
174	**Col 1 Elution Composite Analysis**														
175	Vial	Sample date	sample time (hr:mm:s)	elapsed time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Date of counting	Time of counting	file name	count time min	gross counts	Net counts	back ground
176															
177	A1-Tc-0	11/5/99	N/A	N/A	16.6837	20.2082	3.5245	2.8391	12/06/99	10:18:18	N7TcF23	10	61080	44774	0
178	N7-Tc-ELU-COMP	12/6/99	N/A	N/A	N/A	N/A	21.7332	21.7920	12/06/99	9:29:28	N7TcECP1	10	1022766	900920	0
179	Background	12/6/99	N/A	N/A	N/A	N/A	N/A	N/A	12/06/99	9:51:13	N7BK05	10	7330	-345	0
180															
181															
182	**Elution rinse DI water**														
183		Start Date and Time:		7/2/99	15:57										
184		Pump Speed:		60 (approx 30 mL/hr or 6 CV/hr)											
185	Vial/Bottle	Sample Date	sample start time (hr:mm:s)	sample finish time	Sampling time (hr)	mass of vial + cap	mass of vial + cap + sample	Mass of sample	Vol of Sample	Eff Vol (mL)	Flow Rate, mL/hr	Eff Vol (BV)	Flow Rate, BV/hr	Date of counting	Time of counting
186	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/11/99	8:22:01
187															
188	N7-Tc-E1-R1	11/11/99	8:35	8:45	0.167	16.6771	21.3946	4.7175	4.7175	4.72	28.31	1.00	6.02	11/11/99	8:03:33
189	N7-Tc-E1-R2	11/11/99	8:45	8:55	0.167	16.8547	21.6324	4.7777	4.7777	9.50	28.67	2.02	6.10	11/11/99	8:36:52
190	N7-Tc-E1-R3	11/11/99	8:55	9:05	0.167	16.6664	21.4399	4.7735	4.7735	14.27	28.64	3.04	6.09	11/11/99	8:53:47
191	N7-Tc-E1-R4	11/11/99	9:05	9:15	0.167	16.8795	21.8548	4.9753	4.9753	19.24	29.85	4.09	6.35	11/11/99	9:11:51
192					0.667										
193	**Regeneration - 1.0 M NaOH**														
194		Start Date and Time:		7/9/99	14:55										
195		Pump Speed:		60 (approx 30 mL/hr or 6 CV/hr)											
196		Regenerant (1.0 M NaOH) density @ 25 °C =					1.025	g/mL							
197	Vial/Bottle	Sample Date	sample start time (hr:mm:s)	sample finish time	Sampling time (hr)	mass of vial + cap	mass of vial + cap + sample	Mass of sample	Vol of Sample	Eff Vol (mL)	Flow Rate, mL/hr	Eff Vol (BV)	Flow Rate, BV/hr	Date of counting	Time of counting
198	N7-Tc-0 (small subsample)	11/8/99				16.6837	20.2082	3.5245	2.8391					11/11/99	8:22:01
199															
200	N7-Tc Reg-1	11/11/99	9:15:00	10:15:00	1.000	40.0211	70.9410	30.9199	30.1658	30.1658	30.17	6.42	6.42	-	-
201	N7-Tc-R01	11/11/99	-	-	-	16.8182	21.9803	5.1621	5.0362	5.04	-	-	-	11/11/99	10:09:11
202															
203															

	A	P	Q	R	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD
173																
174	**Col 1 Elution Comp**															
175	Vial	net cnts - bck	net counts per g per min	net cnts per mL of sample per min	C/Co,Tc-95m	Eff Btl Mass (g)	Effluent Mass+ sample (g)	Eff Vol (mL)	Eff Vol (BV)	flow rate BV/hr	flow rate mL/hr	ICP-MS Tc-99	C/Co ICP-MS	Comp DF, Tc-99	Comp DF, Tc-95m	
176																
177	A1-Tc-0	44774	1270	1577	1.000	N/A	N/A	N/A	N/A	N/A	N/A	3340			N/A	
178	N7-Tc-ELU-COMP	900920	4145	4134	2.621	N/A	216.2174	216.8028	46.1283	1.0101	4.7475	3555	1.064		N/A	
179	Background	0	0	0	N/A	N/A	N/A	N/A	N/A	N/A	N/A				N/A	
180																
181																
182	**Elution rinse DI wa**															
183																
184																
185	Vial/Bottle	file name	count time, min	background	Total counts	Net Area	net cnts per g of sample per min	net cnts per mL of sample per min	C/Co, Tc-95m	Tc-99, ICP-MS	C/Co, Tc-99					
186	N7-Tc-0 (small subsample)	N7TcF22	10	0	75298	61435	1743	2164	1	3340						
187																
188	N7-Tc-E1-R1	N7Tc1ER1	10	0	12733	8275	175	175	0.081	94.6	0.028					
189	N7-Tc-E1-R2	N7Tc1ER2	10	0	5727	2055	43	43	0.020							
190	N7-Tc-E1-R3	N7Tc1ER3	10	0	5366	1541	32	32	0.015							
191	N7-Tc-E1-R4	N7Tc1ER4	10	0	4346	887	18	18	0.008	15.3	0.005	0.26163	uCi/L Tc-99			
192																
193	**Regeneration - 1.0**															
194																
195																
196																
197	Vial/Bottle	file name	count time, min	background	Total counts	Net Area	*net cnts per* g of sample per min	net cnts per *mL of* sample per min	C/Co, Tc-95m	Tc-99, ICP-MS	C/Co, Tc-99	Notes				
198	N7-Tc-0 (small subsample)	N7TcF22	10	0	75298	61435	1743	2164	1	2690	1					
199																
200	N7-Tc Reg-1	-	-	-	-	-	-	-	-	-	-	Total Regenerate - all collected in one batch				
201	N7-Tc-R01	N7TcR01B	300	0	115961	7874	5	5	0.002	8.2	0.003	Sample of Total Regenerate				
202																
203										0.1402	uCi/L Tc-99					

Loading of Eluted Column with AN-107 Simulant

Start Date and Time: 12/9/99 9:41 AM
Bed volume col 1= 4.7 mL
Bed volume col 2= 4.7 mL
All effuent collected, in 1 BV aliquots

Supernate Density = 1.243 (g/ml)
Feed analyses: Tc-99, uCi/mL
Tracer: Tc-95m, activity determined at various times given in table.

Pump Speed: 64 (approx 30 mL/hr or 6 CV/hr)

Vial	Sample date	sample start time (hr:mm:s)	sample finish time (hr:mm:s)	sampling time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Count date	Count start time	file name	count time min	gross counts	Net counts	back ground	net cts-bck	counts/mL of sample/min
Tc-SL0	12/9/99	N/A	N/A	N/A	16.4848	22.7992	6.3144	5.0865	12/9/99	10:18:00	TcSL0	10	166231	136680	0	136680	2687
Tc-SL1	12/9/99				17.0377	22.2546	5.2169	4.1970	12/9/99	10:10:00	TcSL1	10	39004	28954	0	28954	690
Tc-SL2	12/9/99	10:29:15	10:39:15	0.167	17.0533	22.2470	5.1937	4.1784	12/9/99	10:55:00	TcSL2	10	13331	5683	0	5683	136
Tc-SL3	12/9/99	10:40:00	10:50:05	0.168	17.0072	22.7555	5.7483	4.6245	12/9/99	11:10:00	TcSL3	10	13337	5598	0	5598	121
Tc-SL4	12/9/99	10:50:40	10:59:43	0.151	16.8111	22.2482	5.4371	4.3742	12/9/99	11:20:00	TcSL4	10	12368	5318	0	5318	122
Tc-SL5	12/9/99	11:00:20	11:10:20	0.167	16.7353	22.8820	6.1467	4.9451	12/9/99	11:25:00	TcSL5	10	13369	5456	0	5456	110
Tc-SL6	12/9/99	11:10:50	11:20:53	0.168	17.0145	23.2207	6.2062	4.9929	12/9/99	11:35:00	TcSL6	10	13143	5844	0	5844	117
Tc-SL7	12/9/99	11:23:30	11:33:40	0.169	16.7025	23.6279	6.9254	5.5715	12/9/99	11:45:00	TcSL7	10	14203	6510	0	6510	117
Tc-SL8	12/9/99	11:35:00	11:45:00	0.167	16.4979	22.7164	6.2185	5.0028	12/9/99	12:00:00	TcSL8	10	13460	5977	0	5977	119
Tc-SL9	12/9/99	11:46:30	11:55:00	0.142	16.5647	21.8141	5.2494	4.2232	12/9/99	12:10:00	TcSL9	10	12796	4681	0	4681	111
Tc-SL10	12/9/99	11:56:00	12:06:10	0.169	16.5717	22.7988	6.2271	5.0097	12/9/99	12:20:00	TcSL10	10	13555	6101	0	6101	122
Tc-SL11	12/9/99	12:07:00	12:17:00	0.167	16.6618	22.8419	6.1801	4.9719	12/9/99	12:30:00	TcSL11	10	13553	6134	0	6134	123
Tc-SL12	12/9/99	12:18:00	12:28:00	0.167	16.5349	22.6868	6.1519	4.9492	12/9/99	12:40:00	TcSL12	10	13556	5899	0	5899	119
Tc-SL13	12/9/99	12:30:00	12:40:00	0.167	16.5921	22.7490	6.1569	4.9533	12/9/99	12:50:00	TcSL13	10	13455	6311	0	6311	127
Tc-SL14	12/9/99	12:42:00	12:52:00	0.167	16.6758	22.9310	6.2552	5.0323	12/9/99	13:15:00	TcSL14	10	13641	6328	0	6328	126
				2.1336													

Caustic Wash: 0.1 M NaOH

Start Date and Time: 12/9/99 1:04 PM
Density of 0.1 M NaOH @ 25 1.001 g/mL
Pump Speed: 31 (approx 13.8 mL/hr or 3 CV/hr)

Vial	Sample date	sample start time (hr:mm:s)	sample finish time (hr:mm:s)	sampling time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Count date	Count start time	file name	count time min	gross counts	Net counts	back ground	net cts-bck	counts/mL of sample/min
Tc-SL0	12/9/99	N/A	N/A	N/A	16.4848	22.7992	6.3144	5.0865	12/9/99	10:18:00	TcSL0	10	166231	136680	0	136680	2687
Tc-SW1	12/9/99	13:04:00	13:24:00	0.333	16.7292	23.0572	6.3280	6.3217	12/9/99	13:30:00	TcSW1	10	13661	6233	0	6233	122
Tc-SW2	12/9/99	13:25:00	13:45:00	0.333	16.6319	22.6485	6.0166	6.0106	12/9/99	13:55:00	TcSW2	10	13868	6055	0	6055	125
Tc-SW3	12/9/99	13:45:30	14:05:30	0.333	16.9031	22.4793	5.5762	5.5706	12/9/99	14:10:00	TcSW3	10	15025	7395	0	7395	165
Tc-SW4	12/10/99	8:48:00	9:08:15	0.337	16.8767	22.0208	5.1441	5.1390	12/10/99	9:20:00	TcSW4	10	17230	9371	0	9371	226
			Total:	1.338													

	A	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH
207	**Loading of Eluted**																
208																	
209																	
210																	
211																	
212																	
213																	
214																	
215																	
216																	
217																	
218																	
219																	
220	Vial	C/Co, Tc-95m	Eff Btl Mass (g)	Effluent Mass (g)	Eff Vol (mL)	Eff Vol (BV)	flow rate BV/hr	flow rate mL/hr	ICP-MS, Tc-99, - bkg ng/g	ICP-MS, Tc-99 ng/mL	C/Co Tc-99	Notes					
221																	
222	Tc-SL0	1.000	N/A	N/A	N/A	N/A	N/A	N/A	2118.1	2629.409	1.000						
223																	
224	Tc-SL1	0.257	N/A	N/A	N/A	N/A	N/A	N/A	-		-	Sample of 1 M NaOH flush. DO NOT include in BT graph. Yellowish sample					
225	Tc-SL2	0.051	N/A	5.1937	4.178	0.88901	5.33408	25.07015	56.55	70.29165	0.027	1st simulant loading sample.			Max Tc-95m DF =	3.8950894	
226	Tc-SL3	0.045	N/A	10.9420	8.803	1.87296	5.85487	27.51791									
227	Tc-SL4	0.045	N/A	16.3791	13.177	2.80363	6.17023	29.00006									
228	Tc-SL5	0.041	N/A	22.5258	18.122	3.85577	6.31283	29.67031									
229	Tc-SL6	0.044	N/A	28.7320	23.115	4.91809	6.34223	29.80848									
230	Tc-SL7	0.043	N/A	35.6574	28.687	6.10352	6.99598	32.8811									
231	Tc-SL8	0.044	N/A	41.8759	33.689	7.16795	6.38657	30.01689	44.1	54.8163	0.021						
232	Tc-SL9	0.041	N/A	47.1253	37.913	8.0665	6.34268	29.81061									
233	Tc-SL10	0.045	N/A	53.3524	42.922	9.1324	6.29056	29.56565									
234	Tc-SL11	0.046	N/A	59.5325	47.894	10.1903	6.34714	29.83154									
235	Tc-SL12	0.044	N/A	65.6844	52.843	11.2433	6.31817	29.69541									
236	Tc-SL13	0.047	N/A	71.8413	57.797	12.2972	6.32331	29.71955									
237	Tc-SL14	0.047	N/A	78.0965	62.829	13.3679	6.42427	30.19405	49	60.907	0.023						
238							81.4	382.8									
239							6.3	29.4									
240																	
241	**Caustic Wash: 0.1**																
242																	
243																	
244																	
245																	
246																	
247	Vial	C/Co, Tc-95m	Eff Btl Mass (g)	Effluent Mass (g)	Eff Vol (mL)	Eff Vol (BV)	flow rate BV/hr	flow rate mL/hr	ICP-MS Tc-99	ICP-MS, Tc-99 ng/mL	C/Co Tc-99	Notes					
248																	
249	Tc-SL0	1.000	N/A	N/A	N/A	N/A	N/A	N/A	2118.1	2629.409	1.000						
250																	
251	Tc-SW1	0.046	N/A	6.3280	6.322	1.34504	4.03511	18.96503	-		-						
252	Tc-SW2	0.047	N/A	12.3446	12.332	2.62389	3.83655	18.03177	45.9	45.9459	0.017						
253	Tc-SW3	0.061	N/A	17.9208	17.903	3.80913	3.55572	16.71189									
254	Tc-SW4	0.084	N/A	23.0649	23.042	4.90252	3.23969	15.22655	90.5	90.5905	0.034						
255						Total:	14.6671	68.93524									
256						Avg:	3.66677	17.23381									
257																	

Continued Loading with Actual AN-107 Feed

Start Date and Time: 12/10/99 9:10 AM
Bed volume col 1= 4.7 mL
Bed volume col 2= 4.7 mL
All effuent collected, in 1 BV aliquots

Supernate Density = 1.2414 (g/ml)
Feed analyses: Tc-99, uCi/mL
Tracer: Tc-95m, activity determined at various times given in table.

Pump Speed: 64 (approx 30 mL/hr or 6 CV/hr)

Vial	Sample date	sample start time (hr:mm:s)	sample finish time (hr:mm:s)	sampling time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Count date	Count start time	file name	count time min	gross counts	Net counts	back ground	net cts-bck	counts/g of sample/min
Tc-SL0	12/9/99	N/A	N/A	N/A	16.4848	22.7992	6.3144	5.0865	12/9/99	10:18:00	TcSL0	10	166231	136680	0	136680	2165
AN-Tc-L1	12/10/99	9:12:15	9:22:15	0.167	16.6698	21.6565	4.9867	4.0170	12/10/99	9:43:00	ANTCL1	10	17594	9936	0	9936	199
AN-Tc-L2	12/10/99	9:23:00	9:33:00	0.167	16.6598	21.5723	4.9125	3.9572	12/10/99	9:55:00	ANTCL2	10	19874	11365	0	11365	231
AN-Tc-L3	12/10/99	9:34:00	9:44:00	0.167	16.9686	22.4686	5.5000	4.4305	12/10/99	10:00:00	ANTCL3	10	21455	7950	0	7950	145
AN-Tc-L4	12/10/99	9:45:00	9:55:00	0.167	16.5874	22.4614	5.8740	4.7318	12/10/99	10:15:00	ANTCL4	10	23277	7673	0	7673	131
AN-Tc-L5	12/10/99	9:56:00	10:06:00	0.167	16.5911	22.5398	5.9487	4.7919	12/10/99	10:25:00	ANTCL5	10	23849	8776	0	8776	148
AN-Tc-L6	12/10/99	10:09:00	10:19:00	0.167	16.7706	22.8325	6.0619	4.8831	12/10/99	10:35:00	ANTCL6	10	24188	7905	0	7905	130
AN-Tc-L7	12/10/99	10:23:00	10:33:00	0.167	16.5288	22.5879	6.0591	4.8809	12/10/99	10:45:00	ANTCL7	10	24363	6779	0	6779	112
AN-Tc-L8	12/10/99	10:36:00	10:46:00	0.167	16.5742	22.7088	6.1346	4.9417	12/10/99	11:00:00	ANTCL8	10	24359	7791	0	7791	127
AN-Tc-L9	12/10/99	10:48:00	10:58:00	0.167	16.6431	22.7375	6.0944	4.9093	12/10/99	11:10:00	ANTCL9	10	24098	7593	0	7593	125
AN-Tc-L10	12/10/99	11:01:00	11:11:00	0.167	16.5140	22.6015	6.0875	4.9037	12/10/99	11:20:00	ANTCL10	10	24262	7318	0	7318	120
AN-Tc-L11	12/10/99	11:14:00	11:24:00	0.167	16.6233	22.7157	6.0924	4.9077	12/10/99	11:35:00	ANTCL11	10	24941	8236	0	8236	135
AN-Tc-L12	12/10/99	11:25:00	11:36:00	0.183	16.5386	23.1702	6.6316	5.3420	12/10/99	11:45:00	ANTCL12	10	26518	9428	0	9428	142
AN-Tc-L13	12/10/99	11:40:00	11:50:00	0.167	16.5080	22.5980	6.0900	4.9058	12/10/99	11:55:00	ANTCL13	10	25242	8244	0	8244	135
AN-Tc-L14	12/10/99	11:51:00	12:01:00	0.167	16.5954	22.7166	6.1212	4.9309	12/10/99	12:10:00	ANTCL14	10	25197	8786	0	8786	144
AN-Tc-L15	12/10/99	12:03:00	12:13:00	0.167	16.5417	22.5983	6.0566	4.8788	12/10/99	12:25:00	ANTCL15	10	25264	8413	0	8413	139
AN-Tc-L16	12/10/99	12:15:00	12:25:00	0.167	16.4691	22.6033	6.1342	4.9414	12/10/99	12:35:00	ANTCL16	10	25965	8436	0	8436	138
AN-Tc-L17	12/10/99	12:27:00	12:37:00	0.167	16.5475	22.6860	6.1385	4.9448	12/10/99	12:50:00	ANTCL17	10	26204	9703	0	9703	158
AN-Tc-L18	12/10/99	12:39:00	12:49:00	0.167	16.5981	22.6742	6.0761	4.8946	12/10/99	13:00:00	ANTCL18	10	26340	9626	0	9626	158
AN-Tc-L19	12/10/99	12:51:00	13:01:00	0.167	16.5879	22.6565	6.0686	4.8885	12/10/99	13:10:00	ANTCL19	10	26953	10258	0	10258	169
AN-Tc-L20	12/10/99	13:03:00	13:13:00	0.167	16.7663	22.8349	6.0686	4.8885	12/10/99	13:25:00	ANTCL20	10	27050	10492	0	10492	173
AN-Tc-L21	12/10/99	13:15:00	13:25:00	0.167	16.5813	22.6799	6.0986	4.9127	12/10/99	13:35:00	ANTCL21	10	27463	10520	0	10520	172
AN-Tc-L22	12/10/99	13:28:00	13:58:00	0.500	16.5107	34.5693	18.0586	14.5470	12/10/99	?	ANTCL22	10	?	?	0		
AN-Tc-L23	12/10/99	14:00:00	14:10:00	0.167	16.6099	22.6673	6.0574	4.8795	12/10/99	14:15:00	ANTCL23	10	29034	12100	0	12100	200
AN-Tc-L24	12/10/99	14:13:00	14:43:00	0.500	16.6678	34.8488	18.1810	14.6456	12/10/99	N.P.	N.P.	N.P.	N.P.	N.P.	0		
AN-Tc-L25	12/10/99	14:45:00	14:55:00	0.167	16.5659	22.6586	6.0927	4.9079	12/10/99	15:00:00	ANTCL25	10	31257	13663	0	13663	224
			Total:	4.850													

Regeneration: 1.0 M NaOH

Start Date and Time: 12/10/99 1:04 PM
1.0 M NaOH density @ 25 °C = 1.025 g/mL
Pump Speed: 31 (approx 13.8 mL/hr or 3 CV/hr)

Vial	Sample date	sample start time (hr:mm:s)	sample finish time (hr:mm:s)	elapsed time (hr)	sample vial + cap (tare) g	mass of sample + vial + cap g	sample mass counted g	sample volume counted mL	Count date	Count start time	file name	count time min	gross counts	Net counts	back ground	net cts-bck	counts/g of sample/min
Tc-SL0	12/9/99	N/A	N/A	N/A	16.4848	22.7992	6.3144	5.0865	12/9/99	10:18:00	TcSL0	10	166231	136680	0	136680	2165
AN-Tc-W1	12/10/99	15:00:00	15:20:00	0.333	16.6823	22.6890	6.0067	5.8602	12/10/99	15:25:00	ANTcW1	10	31009	14178	0	14178	236
AN-Tc-W2	12/10/99	15:21:00	15:41:00	0.333	16.5453	22.4528	5.9075	5.7634	12/10/99	15:45:00	ANTcW2	10	29543	13113	0	13113	222
AN-Tc-W3	12/10/99	15:42:00	16:02:00	0.333	16.7639	22.1466	5.3827	5.2514	12/10/99	16:05:00	ANTcW3	10	24818	12055	0	12055	224
AN-Tc-W4	12/10/99	16:03:00	16:23:00	0.333	16.5277	21.5960	5.0683	4.9447	12/10/99	16:30:00	ANTcW4	10	20074	10546	0	10546	208
			Total:	1.333													

	A	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH
258	**Continued_Loadin**																
259																	
260																	
261																	
262																	
263																	
264																	
265																	
266																	
267																	
268																	
269																	
270																	
271	Vial	C/Co, Tc-95m	Eff Btl Mass	Effluent Mass	Eff Vol	Eff Vol	flow rate	flow rate	ICP-MS	ICP-MS, Tc-99	C/Co	Notes					
272			(g)	(g)	(mL)	(BV)	BV/hr	mL/hr	Tc-99	ng/mL	Tc-99						
273	Tc-SL0	1.000	N/A	N/A	N/A	N/A	N/A	N/A	2118.1	2629.409	1.000						
274																	
275	AN-Tc-L1	0.092	N/A	4.9867	4.017	0.85468	5.12808	24.10198	83.3	103.4086	0.039	Clear and colorless- no AN-107 feed in this sample.					
276	AN-Tc-L2	0.107	N/A	9.8992	7.974	1.69664	5.05178	23.74335				Trace of color - AN-107 feed begins emerging with this sample.					
277	AN-Tc-L3	0.067	N/A	15.3992	12.405	2.6393	5.65593	26.58289									
278	AN-Tc-L4	0.060	N/A	21.2732	17.136	3.64606	6.04054	28.39053									
279	AN-Tc-L5	0.068	N/A	27.2219	21.928	4.66561	6.11736	28.75157									
280	AN-Tc-L6	0.060	N/A	33.2838	26.812	5.70458	6.23376	29.2987									
281	AN-Tc-L7	0.052	N/A	39.3429	31.692	6.74306	6.23089	29.28516	1658.1	2058.365	0.783	Max Tc-95m DF =		19.3470773			
282	AN-Tc-L8	0.059	N/A	45.4775	36.634	7.79448	6.30853	29.65007									
283	AN-Tc-L9	0.058	N/A	51.5719	41.543	8.83901	6.26719	29.45578									
284	AN-Tc-L10	0.056	N/A	57.6594	46.447	9.88236	6.26009	29.42243									
285	AN-Tc-L11	0.062	N/A	63.7518	51.355	10.9265	6.26513	29.44611									
286	AN-Tc-L12	0.066	N/A	70.3834	56.697	12.0631	6.19965	29.13836									
287	AN-Tc-L13	0.063	N/A	76.4734	61.603	13.1069	6.26266	29.43451	1658.1	2058.365	0.783						
288	AN-Tc-L14	0.066	N/A	82.5946	66.533	14.156	6.29475	29.58531									
289	AN-Tc-L15	0.064	N/A	88.6512	71.412	15.1941	6.22831	29.27308									
290	AN-Tc-L16	0.064	N/A	94.7854	76.354	16.2455	6.30811	29.64814									
291	AN-Tc-L17	0.073	N/A	100.9239	81.298	17.2975	6.31254	29.66892									
292	AN-Tc-L18	0.073	N/A	107.0000	86.193	18.3389	6.24837	29.36733									
293	AN-Tc-L19	0.078	N/A	113.0686	91.082	19.379	6.24065	29.33108	1698.1	2108.021	0.802						
294	AN-Tc-L20	0.080	N/A	119.1372	95.970	20.4192	6.24065	29.33108									
295	AN-Tc-L21	0.080	N/A	125.2358	100.883	21.4644	6.27151	29.47608									
296	AN-Tc-L22		N/A	143.2944	115.430	24.5595	6.1902	29.09393									
297	AN-Tc-L23	0.092	N/A	149.3518	120.309	25.5977	6.22914	29.27695									
298	AN-Tc-L24		N/A	167.5328	134.955	28.7138	6.23215	29.29112									
299	AN-Tc-L25	0.104	N/A	173.6255	139.863	29.758	6.26544	29.44756	1728.1	2145.263	0.816						
300						Total:	153.083	719.492									
301						Avg:	6.12334	28.77968									
302																	
303	**Regeneration: 1.0**																
304																	
305																	
306																	
307																	
308																	
309	Vial	C/Co, Tc-95m	Eff Btl Mass	Effluent Mass	Eff Vol	Eff Vol	flow rate	flow rate	ICP-MS	ICP-MS, Tc-99	C/Co	Notes					
310			(g)	(g)	(mL)	(BV)	BV/hr	mL/hr	Tc-99	ng/mL	Tc-99						
311	Tc-SL0	1.000	N/A	N/A	N/A	N/A	N/A	N/A	2118.1	2629.409	1.000						
312																	
313	AN-Tc-W1	0.109	N/A	6.0067	5.8602	1.2469	3.7406	17.5806	-		-						
314	AN-Tc-W2	0.103	N/A	11.9142	11.6236	2.4731	3.6788	17.2902	1608.1	1648.303	0.627						
315	AN-Tc-W3	0.103	N/A	17.2969	16.8750	3.5904	3.3520	15.7542									
316	AN-Tc-W4	0.096	N/A	22.3652	21.8197	4.6425	3.1562	14.8340	399.6	409.59	0.156						
317						Total:	13.9275	65.4591									
318						Avg:	3.48187	16.36478									

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

RT HALLEN
Date 9/3/99
Route
File 11-041
Copy

Project: # 29953
Client: ~~K.~~ Brooks

ONLY DATA FOR MN-21 & MN-22 (Tc IX#1 & Tc IX#2). SEE LAST PAGE.

Archive AN-107

ACL Number(s): 99-2255 through 99-2260

Client ID: "Mn-23" through "Mn-28"

ASR Number: 5457

Total Samples: 6

original final data report

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: DR Sanders

Analysis Date (Filename): 08-04-99 (A0538)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

8-13-99
Reviewed by

8-17-99
Concur

Post-It® Fax Note 7671	Date	# of pages ▶
To Dave Blanchard	From Rich Hallen	
Co./Dept.	Co.	
Phone #	Phone #	
Fax # 372-2156	Fax #	

8/13/99

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ... ICPAES Data Report

Six radioactive aqueous samples, Mn-23 through Mn-28 (ACL# 99-2255 through 99-2260), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. All samples were prepared using plastic labware. Approximately 5ml aliquots of sample solution were weighed, processed and diluted to a final volume of 20ml. All liquid samples were caustic, salt solutions prior to processing. Sample Mn-28 (ACL# 99-2260) was filtered after being diluted to 20 ml using 0.45 um membrane filter.

All results reported are in µg/g including liquid samples as requested by client. All results have been corrected for preparation and analytical dilution. Volumes and weights are recorded on bench sheets (included with raw data, etc.). Analytes of interest include Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Ti, U, and Zn. No changes have been made relative to the preliminary report provided earlier to the client.

All quality control checks met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Aqueous samples) All results were within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

(Aqueous samples) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

(Aqueous samples) All analytes of interest in the blank spike were recovered within tolerance limit of 80% to 120%.

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ... ICPAES Data Report

Matrix Spiked Sample:
(Aqueous samples)

All analytes of interest in the matrix-spike were recovered within tolerance limit of 75% to 125% except for Ba and Pb. Matrix-spike recovery for barium in sample Mn-22 (ACL# 99-2259) was only about 5%. Lead recovery in the same sample was about 41%. Low recovery for the two analytes may be caused by moderate to high concentration of sulfate in the sample. Post-spike and blank-spike recovery for all analytes of interest was recovered within tolerance limit of 75% to 125%.

Quality Control Check Standards:

Concentration of all analytes of interest was within tolerance limit of ± 10% accuracy in the standards: QC_MCVA and QC_MCVB. Calibration Blank (ICP98.0) concentration was less than two times IDL.

High Calibration Standard Check:

Verification of the high-end calibration accuracy for all analytes of interest except lanthanum and uranium was within ± 5% tolerance. The high-end calibration accuracy for lanthanum was somewhat low, about –6.9% and uranium was also low by about –5.7%. This will cause results for lanthanum and uranium concentration to appear lower than what is actually present in the samples. Lanthanum and uranium concentration measurements were all below EQL (and below MRQ).

Process Blank:
(Aqueous samples)

All analytes of interest were within tolerance limit of ≤ EQL or < 5% of sample concentration.

Laboratory Control Standard (LCS):
(Aqueous samples)

No LCS was prepared for PNL-ALO-128 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%. See attached ICPAES data results.

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ... ICPAES Data Report

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	3.2 99-2255-PB @1 Process Blank 8/4/99 ug/g	16.3 99-2255 @5 Mn-23 8/4/99 ug/g	16.4 99-2256 @5 Mn-31 8/4/99 ug/g	16.3 99-2257 @5 Mn-32 8/4/99 ug/g	15.9 99-2258 @5 Mn-21 8/4/99 ug/g	
0.015	Ag	–	–	–	–	–	
0.060	Al	–	119	105	106	133	
0.080	As	–	–	–	–	–	
0.050	B	–	13.8	18.4	13.8	18.5	
0.010	Ba	–	[0.33]	–	–	[1.3]	
0.010	Be	–	–	–	–	–	
0.100	Bi	–	–	–	–	–	
0.100	Ca	–	130	129	129	227	
0.015	Cd	–	23.2	22.6	22.6	27.0	
0.100	Ce	–	–	–	–	[9.7]	
0.025	Co	–	[1.8]	[1.8]	[1.8]	[2.0]	
0.020	Cr	–	35.7	36.9	36.1	58.6	
0.015	Cu	[0.13]	11.5	14.1	12.7	12.8	
0.050	Dy	–	–	–	–	–	
0.100	Eu	–	–	–	–	–	
0.025	Fe	[0.13]	8.00	7.58	5.82	445	
2.000	K	–	604	584	592	685	
0.025	La	–	[0.77]	[0.71]	[0.74]	9.46	
0.005	Li	–	[0.34]	[0.25]	[0.26]	[0.25]	
0.100	Mg	–	–	–	–	–	
0.005	Mn	–	1.13	[0.48]	[0.50]	44.1	
0.030	Mo	–	13.2	12.8	12.9	15.0	
0.100	Na	[0.75]	84,600	84,200	86,200	104,000	5.754 M
0.100	Nd	–	[2.6]	[2.9]	[2.9]	29.0	
0.030	Ni	–	189	184	185	217	
0.100	P	–	166	162	163	184	
0.060	Pb	–	69.5	68.9	67.4	134	
0.300	Pd	–	–	–	[4.9]	[16]	
0.300	Rh	–	–	–	–	[4.8]	
0.075	Ru	–	13.0	12.5	12.7	14.8	
0.050	Sb	–	–	–	–	–	
0.050	Se	–	[1.0]	[0.94]	[1.0]	[1.00]	
0.100	Si	–	38.2	43.8	34.4	41.9	
1.000	Sn	–	–	–	–	–	
0.005	Sr	–	115	171	159	1.14	
0.500	Te	–	–	–	–	–	
0.800	Th	–	–	–	–	–	
0.005	Ti	–	–	–	–	0.943	
0.250	Tl	–	–	–	–	–	
2.000	U	–	[40]	[38]	[41]	[48]	
0.015	V	–	–	–	–	–	
0.500	W	–	[60]	[59]	[58]	[70]	
0.010	Y	–	[0.44]	[0.59]	[0.56]	2.81	
0.020	Zn	[0.18]	5.19	5.48	5.39	9.36	
0.025	Zr	–	[1.1]	[1.2]	[0.94]	14.3	

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	16.3 99-2259 @5 Mn-22 8/4/99 ug/g	16.3 99-2259-DUP @5 Mn-22 8/4/99 ug/g	80.5 99-2260 @25 Mn-26 8/4/99 ug/g			
0.015	Ag	–	–	–	–	–	
0.060	Al	105	107	121	–	–	
0.080	As	–	–	–	–	–	
0.050	B	13.1	13.4	[15]	–	–	
0.010	Ba	–	–	[1.9]	–	–	
0.010	Be	–	–	–	–	–	
0.100	Bi	–	–	–	–	–	
0.100	Ca	126	130	220	–	–	
0.015	Cd	22.8	23.4	22.6	–	–	
0.100	Ce	–	–	[15]	–	–	
0.025	Co	[1.7]	[1.8]	–	–	–	
0.020	Cr	34.4	35.2	84.2	–	–	
0.015	Cu	11.1	11.3	12.1	–	–	
0.050	Dy	–	–	–	–	–	
0.100	Eu	–	–	–	–	–	
0.025	Fe	4.32	4.40	887	–	–	
2.000	K	597	614	[600]	–	–	
0.025	La	[0.63]	[0.67]	[11]	–	–	
0.005	Li	[0.21]	[0.23]	[0.60]	–	–	
0.100	Mg	–	–	–	–	–	
0.005	Mn	3.73	3.84	2,610	–	–	
0.030	Mo	12.8	13.3	[12]	–	–	
0.100	Na	90,700	85,100 4.75E 5	85,200	–	–	
0.100	Nd	[1.9]	[2.0]	[38]	–	–	
0.030	Ni	185	191	188	–	–	
0.100	P	162	166	136	–	–	
0.060	Pb	67.2	69.1	140	–	–	
0.300	Pd	–	–	–	–	–	
0.300	Rh	–	–	–	–	–	
0.075	Ru	12.6	13.0	[13]	–	–	
0.050	Sb	–	–	–	–	–	
0.050	Se	[0.86]	[1.00]	[4.8]	–	–	
0.100	Si	29.5	30.4	[44]	–	–	
1.000	Sn	–	–	–	–	–	
0.005	Sr	110	114	4,770	–	–	
0.500	Te	–	–	–	–	–	
0.800	Th	–	–	–	–	–	
0.005	Ti	–	–	[1.3]	–	–	
0.250	Tl	–	–	–	–	–	
2.000	U	[36]	[39]	–	–	–	
0.015	V	–	–	–	–	–	
0.500	W	[59]	[60]	[47]	–	–	
0.010	Y	[0.32]	[0.31]	[3.6]	–	–	
0.020	Zn	5.28	5.74	[8.4]	–	–	
0.025	Zr	[0.83]	[0.72]	–	–	–	

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "–" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # 5457

WP# W51304

File: L:\radchem\hydroxide\asr5457
Analysis Date: [redacted]
Print Date: 8/4/99

Hydroxide and Alkalinity Determination
Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: [signature] 8/2/99
Reviewer: [signature] 8/4/99

Titrant	Molarity
HCl	0.2034

Std. & Spike	Molarity
NaOH	0.1018

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	OH 1st Equivalence Point pH	Found millimoles base	Molarity base	millimole RPD
99-2258	Mn-21		0.100	0.1271	1.271	1	11.558	0.423	10.624	0.086	0.86	
99-2258	Mn-21	Replicate	0.200	0.2547	1.274	2	11.759	0.855	10.750	0.174	0.87	1.06%
99-2259	Mn-22		0.200	0.2485	1.243	3	11.702	0.723	10.67	0.147	0.74	
99-2259	Mn-22	Replicate	0.200	0.2494	1.247	4	11.707	0.724	10.699	0.147	0.74	0.14%
QC Data:												
Reag. Blk.			5.00			1	6.143				OH % Recovery, Acc	
Standard 1	0.1018 N NaOH		5.000	5.0036	1.001	2	12.026	2.492	7.918	0.5069	99.6%	Std 1
Standard 2	0.1018 N NaOH		5.000	5.0196	1.004	3	11.89	2.486	7.578	0.5057	99.3%	Std 2
99-2258MS	Mn-21 + 2mL 0.1N NaOH		0.100	0.1256	1.256	5	11.846	1.338	10.611	0.272	91.2%	MS
99-2259MS	Mn-22 + 2mL 0.1N NaOH		0.100	0.1229	1.229	6	11.702	1.292	10.640	0.263	92.9%	MS

Buffer	VWR Lot #	CMS#	Expire Date
10	981659-24	144109	Jul-00
4	981583-24	144107	Jun-00
7	981894-24	144108	Aug-00

Performance checks

Balance # 360--01-06-037

		Vol.	Wt.
Pipet #	H30762	5.00	4.944
Pipet #	2734494	0.500	0.496
Pipet #	120737	0.100	0.1013
Pipet #	120737	0.200	0.1992

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Date 8/12/99
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Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # 5457
WP# W51304

File: L:\radchem\hydroxide\asr5457
Analysis Date: [redacted]
Print Date: 8/4/99

Hydroxide and Alkalinity Determination
Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: [signature] 8/4/99
Reviewer: [signature] 8/4/99

Titrant	Molarity
HCl	0.2034

RPG #	Sample ID		Sample Vol. (mL)	CO3 2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD	HCO3 3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
99-2258	Mn-21	0	0.100	0.859	7.658	0.089	0.887		1.314	4.427	0.093	0.93	
99-2258	Mn-22	Replicate	0.200	1.720	7.793	0.176	0.880	0.81%	2.617	4.526	0.182	0.91	1.4%
99-2259	Mn-22	0	0.200	1.460	7.670	0.150	0.750		2.210	4.573	0.153	0.76	
99-2259	Mn-22	Replicate	0.200	1.454	7.808	0.148	0.742	0.95%	2.211	4.581	0.154	0.77	0.9%
racy							CO3 % Recovered						
Standard 1	0.1018 N NaOH		5.000	2.559	4.054	0.01363	0.003	sample					
Standard 2	0.1018 N NaOH		5.000	2.566	3.879	0.01627	0.003	sample			HCO3 % recovered		
99-2258MS	Mn-21 + 2mL 0.1N NaOH		0.100	1.825	7.723	0.09906	112.1%	sample	2.324	4.573	0.1015	110.5%	sample
99-2259MS	Mn-22 + 2mL 0.1N NaOH		0.100	1.715	7.7	0.08604	115.3%	sample	2.146	4.495	0.0877	114.4%	sample

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0..100-mL of sample for each matrix spike.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

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Chem Rec_51a

Prep date: 4/18/99

Preparation of Standardized 0.2 M HCl

WP# K51300

Standardized 0.1021 M NaOH will be re-checked and then used to standardized the ~ 0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37 --prep.date 2-25-98 for original data) and re-verified against NIST SRM84] Potassium Acid Phthalate KHC8H404 (KAP) = 204.23 g/mole -- Barcode # 52232 --- (see below verification check).
The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.
Prepared 1- liters of ~0.2 M HCl by diluting 100 mL of 1.029M HCl (Chemrec_10) to 0.5 L with DI. H2O.
20 mL aliquots of 0.2 M HCl were were neutralized to the phenopthalen endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipitting error is estimated to be < 1% @ 1s. Thus total error is < 3 % for the measurements

NaOH Molarity veification

Verification Test #	Wt. of KAP	Vol. of 0.1021M NaOH to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.80894	38.95	0.1017	
2	0.80582	38.84	0.1016	
3	0.96233	46.12	0.1022	
		Ave=	0.1018	0.0003
			re-certified value	

Titration Id.	aliquot of sample	Vol. of 0.1018M NaOH to neutralize	Molarity of Acid in Sample	Molarity Error +/- @ 1 s
1	20.00	39.88	0.2030	
2	20.00	39.92	0.2032	
3	20.00	40.04	0.2038	
		Ave Molarity HCl =	[illegible]	

Analyst/Date 8/4/99

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

00-0292Tc oxidation

2/16/2000

Client : K. Brooks

Cognizant Scientist: D. K. Atkins Date : 2/16/00

Concur : L R Greenwood Date : 2/17/00

Measured Activities (μCi/ml) with 1-σ error

ALO ID Client ID	No oxidation Tc-99 Error %	Cerric oxidation Tc-99 Error %	Percentage Tc+7*	KMnO4 oxidation Tc-99 Error %	Percentage Tc+7*
00-0292 MR-01	1.65E-2 4%	5.27E-2 4%	31%	5.60E-2 4%	29%
00-0292 duplicate MR-01	1.60E-2 4%	5.04E-2 4%	32%	5.09E-2 4%	31%
RPD	3%	4%		10%	
00-0293 MR-02	1.69E-2 4%	5.60E-2 4%	30%	5.69E-2 4%	30%
00-0294 MR-03	1.74E-2 4%	4.35E-2 4%	40%	4.35E-2 4%	40%
Blank	<5.6E-5	<5.9E-5		<5.65E-5	
Blank Spike	101%	93%		98%	
Sample Spike	108%	109%		104%	

*Tc+7 percentage in the as-received sample.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

00-0292Tc oxidation

2/16/2000

Client : K. Brooks

Discussion

Absolute values of the Tc+7 and total technetium are reported. Technetium speciation is also reported as a percentage of Tc+7 in the sample. In order to measure these values, sample sets were prepared in both non-oxidizing and oxidizing environments. In all analysis cases, a 0.1-mL aliquot was diluted to 0.5 mL (DI water or 8M HNO3, depending on oxidizing or non-oxidizing conditions) and a 0.05-mL aliquot was processed and purified for Tc analysis. The Tc spike added to the reagent spike and matrix spike was the pertechnetate form.

Non-oxidizing conditions were provided by diluting sample aliquots in deionized water. Procedure PNL-ALO-432 was run with no added sodium dichromate (oxidizer). The only Tc that passes through the cation exchange column and is extracted as the tetraphenylarsonium pertechnetate is the Tc already in the +7 state. Under these conditions, the blank spike and matrix spike recovered well, the blank shows no evidence of contamination, and the precision measured by the sample duplicates is within the error of the method.

The cerric ammonium nitrate oxidation method, provided by Norm Schroeder from LANL, was used on one sample set. In this method, the sample aliquot was contacted with 8 M HNO3 and 0.1M cerric ammonium nitrate in 0.15M HNO3. The sample was heated. As the characteristic color of the cerric was lost, additional cerric was added and heating continued. The sample was reduced in volume to near dryness and 2-mL concentrated HNO3 was added. The sample was again reduced to near dryness and another 2-mL concentrated HNO3 was added. After a final volume reduction, 3-mL DI water was added and the sample was processed through the standard operating procedure PNL-ALO-432.

The potassium permanganate oxidation is similar to the cerric oxidation and was used on another sample set. A saturated solution of KMnO4 was added to the sample aliquot and allowed to react. As the purple color was lost, additional KMnO4 was added. The sample was allowed to sit over the weekend in contact with KMnO4. The sample was then heated to low volume, 1 drop of H2O2 added to dissolve MnO2 and the sample evaporated to near dryness. A 3-mL portion of DI water was added to each sample and the separation proceeded according to PNL-ALO-432.

In both oxidation cases, the blanks show no indication of cross-contamination. The reagent spikes and matrix spikes indicate no losses of Tc as a result of the vigorous oxidation processes. Precision was good, well below the threshold of 20% relative percent difference (RPD). The Tc concentration values of the 2 different oxidation techniques agree to within 6%.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

00-1072Tc oxidation

2/17/2000

Client : D. Blanchard

Cognizant Scientist: D. K. Fiskum Date : 2/17/00

Concur : L R Greenwood Date : 2/17/00

Measured Activities (μCi/ml) with 1-σ error

ALO ID Client ID	No oxidation Tc-99 Error %	Cerric oxidation Tc-99 Error %	Percentage Tc+7*	KMnO4 oxidation Tc-99 Error %	Percentage Tc+7*
00-1072 N7-Tc-F01	1.23E-2 4%	3.49E-2 4%	35%	3.73E-2 4%	33%
00-1072 duplicate N7-Tc-F01	1.36E-2 4%	3.77E-2 4%	36%	3.99E-2 4%	34%
RPD	10%	8%		7%	
Blank	<5.6E-5	<5.9E-5		<5.65E-5	
Blank Spike	101%	93%		98%	
Sample Spike	100%	99%		95%	

*Tc+7 percentage in the as-received sample.

Client : D. Blanchard

Discussion

Absolute values of the Tc+7 and total technetium are reported. Technetium speciation is also reported as a percentage of Tc+7 in the sample. In order to measure these values, sample sets were prepared in both non-oxidizing and oxidizing environments. In all analysis cases, a 0.1-mL aliquot was diluted to 0.5 mL (DI water or 8M HNO3, depending on oxidizing or non-oxidizing conditions) and a 0.05-mL aliquot was processed and purified for Tc analysis. The Tc spike added to the reagent spike and matrix spike was the pertechnetate form.

Non-oxidizing conditions were provided by diluting sample aliquots in deionized water. Procedure PNL-ALO-432 was run with no added sodium dichromate (oxidizer). The only Tc that passes through the cation exchange column and is extracted as the tetraphenylarsonium pertechnetate is the Tc already in the +7 state. Under these conditions, the blank spike and matrix spike recovered well, the blank shows no evidence of contamination, and the precision measured by the sample duplicates is within the error of the method.

The cerric ammonium nitrate oxidation method, provided by Norm Schroeder from LANL, was used on one sample set. In this method, the sample aliquot was contacted with 8 M HNO3 and 0.1M cerric ammonium nitrate in 0.15M HNO3. The sample was heated. As the characteristic color of the cerric was lost, additional cerric was added and heating continued. The sample was reduced in volume to near dryness and 2-mL concentrated HNO3 was added. The sample was again reduced to near dryness and another 2-mL concentrated HNO3 was added. After a final volume reduction, 3-mL DI water was added and the sample was processed through the standard operating procedure PNL-ALO-432.

The potassium permanganate oxidation is similar to the cerric oxidation and was used on another sample set. A saturated solution of KMnO4 was added to the sample aliquot and allowed to react. As the purple color was lost, additional KMnO4 was added. The sample was allowed to sit over the weekend in contact with KMnO4. The sample was then heated to low volume, 1 drop of H2O2 added to dissolve MnO2 and the sample evaporated to near dryness. A 3-mL portion of DI water was added to each sample and the separation proceeded according to PNL-ALO-432.

In both oxidation cases, the blanks show no indication of cross-contamination. The reagent spikes and matrix spikes indicate no losses of Tc as a result of the vigorous oxidation processes. Precision was good, well below the threshold of 20% relative percent difference (RPD). The Tc concentration values of the 2 different oxidation techniques agree to within 7%.

Battelle
Pacific Northwest Laboratories

Project Number

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329/4 File
Mike Urie

Date December 22, 1999

To Dave Blanchard

From Tom Farmer Orrill Thomas Farmer III

Subject ICP/MS Analysis of Submitted Samples
(ACL #00-0626 through 00-0667) ASR 5028

Pursuant to your request, the 46 samples that you submitted for analysis were analyzed by ICPMS for ^{99}Tc. The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc standard was used to generate the calibration curve and an independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank. The samples were diluted an extra 10x from the dilutions received. The results include your dilutions and are reported in ng analyte/ g (ppb) of original sample ± one standard deviation.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc. The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses..

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624.

Blanchard Tc-99 Analysis

December 22, 1999

Results are reported in ng analyte /g (ppb) of original sample.

Sample ID	ICP/MS Number	Tc-99 ng/g	±	1SD
1%HNO3	9c21a1	<0.015		
1%HNO3	9c21a30	<0.015		
1%HNO3	9c21a51	0.025	±	0.009
1%HNO3	9c22a1	<0.029		
1%HNO3	9c22a6	<0.029		
1%HNO3	9c22a27	<0.029		
00-00626 Blk1	9c21a8	1.6	±	0.3
00-00626 Blk2	9c21a9	1.2	±	0.1
00-00626 Blk3	9c21a10	0.80	±	0.15
00-00626	9c21a12	2590	±	130
00-00626DUP	9c21a13	2790	±	50
00-00627	9c21a14	3860	±	70
00-00628	9c21a15	3660	±	190
00-00629	9c21a16	3730	±	160
00-00630	9c21a17	3480	±	90
00-00631	9c21a18	3550	±	100
00-00632	9c21a19	3280	±	100
00-00633	9c21a20	3320	±	210
00-00634	9c21a21	3290	±	100
00-00635	9c21a23	3400	±	50
00-00636	9c21a43	2630	±	220
00-00637	9c21a25	2590	±	40
00-00637 + spike	9c21a29	7570	±	180
Spike Recovery		**110%**		
00-00638	9c21a26	3280	±	140
00-00639	9c21a27	3080	±	210
00-00640	9c21a28	3410	±	20
00-00641	9c21a34	3210	±	40
00-00642	9c21a35	3190	±	80
00-00643	9c21a36	1610	±	70
00-00644	9c21a37	2480	±	130
00-00645	9c21a38	3140	±	50
00-00646	9c21a39	3460	±	30
00-00647	9c22a8	32100	±	1150
00-00648	9c22a9	90700	±	1250
00-00649	9c22a10	29800	±	900
00-00650	9c21a44	7330	±	120
00-00651	9c21a45	1110	±	60
00-00652	9c21a46	633	±	53
00-00653	9c22a11	447	±	39
00-00654	9c22a12	364	±	18
00-00655	9c22a13	452	±	28
00-00656	9c22a14	255	±	0.7
00-00657	9c22a15	214	±	19
00-00658	9c22a16	167	±	5
00-00659	9c22a17	439	±	17
00-00660	9c22a18	180	±	12
00-00660 + spike	9c22a26	3570	±	30
Spike Recovery		98%		
00-00661	9c22a19	160	±	20
00-00662	9c22a29	94.6	±	3.2
00-00663	9c22a30	15.3	±	0.9
00-00664	9c22a31	8.2	±	1.0

DATA REVIEW

Reviewed by: O. J. Farmer

Date: 30 Dec 99 Pages: 1 of 2

Blanchard Tc-99 Analysis

December 22, 1999

Results are reported in ng analyte /g (ppb) of original sample.

Sample ID	ICP/MS Number	Tc-99 ng/g	±	1SD
00-00665	9c22a23	2490	±	110
00-00666	9c22a24	1960	±	20
00-00666DUP	9c22a25	1910	±	80
00-00667	9c21a49	2210	±	60
00-00667 + spike	9c21a50	5760	±	110
Spike Recovery		**112%**		
CCV results are reported in ng/ml (ppb)				
5ppb Tc-99	9c21a4	4.56	±	0.22
5ppb Tc-99	9c21a22	5.22	±	0.18
5ppb Tc-99	9c21a53	4.97	±	0.02
5ppb Tc-99	9c22a4	4.83	±	0.15
5ppb Tc-99	9c22a28	4.74	±	0.10
100ppb Co	9c21a7	0.028	±	0.0002
100ppb Co	9c22a7	0.031	±	0.009

DATA REVIEW

Reviewed by: O. J. Farmer III

Date: 30 Dec 99 Pages: 2 of 2

Blanchard Tc-99 Analysis

December 22, 1999

Results are reported in ng analyte /g (ppb) of original sample.

Sample ID	Client ID	ICP/MS Number	Tc-99 ng/g	±	1SD	Sltn Density	Tc-99 ng/mL
1%HNO3		9c21a1	<0.015				
1%HNO3		9c21a30	<0.015				
1%HNO3		9c21a51	0.025	±	0.009		
1%HNO3		9c22a1	<0.029				
1%HNO3		9c22a6	<0.029				
1%HNO3		9c22a27	<0.029				
00-00626 Blk1	Process Blank	9c21a8	1.6	±	0.3		
00-00626 Blk2	Process Blank	9c21a9	1.2	±	0.1		
00-00626 Blk3	Process Blank	9c21a10	0.80	±	0.15		
00-00626	N7-Tc-0	9c21a12	2590	±	130	1.2414	3215
00-00626DUP	N7-Tc-L0	9c21a13	2790	±	50	1.2414	3464
00-00627	N7-Tc-L1	9c21a14	3860	±	70	1.2414	4792
00-00628	N7-Tc-L3	9c21a15	3660	±	190	1.2414	4544
00-00629	N7-Tc-L5	9c21a16	3730	±	160	1.2414	4630
00-00630	N7-Tc-L7	9c21a17	3480	±	90	1.2414	4320
00-00631	N7-Tc-L9	9c21a18	3550	±	100	1.2414	4407
00-00632	N7-Tc-L11	9c21a19	3280	±	100	1.2414	4072
00-00633	N7-Tc-L13	9c21a20	3320	±	210	1.2414	4121
00-00634	N7-Tc-L15	9c21a21	3290	±	100	1.2414	4084
00-00635	N7-Tc-L17	9c21a23	3400	±	50	1.2414	4221
00-00636	N7-Tc-P1	9c21a43	2630	±	220	1.2414	3265
00-00637	N7-Tc-P3	9c21a25	2590	±	40	1.2414	3215
00-00637 + spike	N7-Tc-P3	9c21a29	7570	±	180	1.2414	9397
Spike Recovery			**110%**				
00-00638	N7-Tc-P5	9c21a26	3280	±	140	1.2414	4072
00-00639	N7-Tc-P7	9c21a27	3080	±	210	1.2414	3824
00-00640	N7-Tc-P9	9c21a28	3410	±	20	1.2414	4233
00-00641	N7-Tc-PW1	9c21a34	3210	±	40	1.2414	3985
00-00642	N7-Tc-PW3	9c21a35	3190	±	80	1.2414	3960
00-00643	N7-Tc-PW5	9c21a36	1610	±	70	1.001	1612
00-00644	N7-Tc-PW7	9c21a37	2480	±	130	1.001	2482
00-00645	N7-Tc-PR1	9c21a38	3140	±	50	1	3140
00-00646	N7-Tc-PR2	9c21a39	3460	±	30	1	3460
00-00647	N7-Tc-E1-1	9c22a8	32100	±	1150	1	32100
00-00648	N7-Tc-E1-2	9c22a9	90700	±	1250	1	90700
00-00649	N7-Tc-E1-3	9c22a10	29800	±	900	1	29800
00-00650	N7-Tc-E1-4	9c21a44	7330	±	120	1	7330
00-00651	N7-Tc-E1-8	9c21a45	1110	±	60	1	1110
00-00652	N7-Tc-E1-12	9c21a46	633	±	53	1	633
00-00653	N7-Tc-E1-16	9c22a11	447	±	39	1	447
00-00654	N7-Tc-E1-20	9c22a12	364	±	18	1	364
00-00655	N7-Tc-E1-23	9c22a13	452	±	28	1	452
00-00656	N7-Tc-E1-27	9c22a14	255	±	0.7	1	255
00-00657	N7-Tc-E1-31	9c22a15	214	±	19	1	214
00-00658	N7-Tc-E1-35	9c22a16	167	±	5	1	167
00-00659	N7-Tc-E1-38	9c22a17	439	±	17	1	439
00-00660	N7-Tc-E1-42	9c22a18	180	±	12	1	180
00-00660 + spike	N7-Tc-E1-42	9c22a26	3570	±	30	1	3570
Spike Recovery			**98%**				
00-00661	N7-Tc-E1-45	9c22a19	160	±	20	1	160
00-00662	N7-Tc-E1-R1	9c22a29	94.6	±	3.2	1	95
00-00663	N7-Tc-E1-R4	9c22a30	15.3	±	0.9	1	15
00-00664	N7-Tc-RO	9c22a31	8.2	±	1.0	1	8

Blanchard Tc-99 Analysis

December 22, 1999

Results are reported in ng analyte /g (ppb) of original sample.

Sample ID		ICP/MS Number	Tc-99 ng/g	±	1SD		
00-00665	N7BF	9c22a23	2490	±	110	1.2414	3091
00-00666	N7B-38F	9c22a24	1960	±	20	1.2414	2433
00-00666DUP	N7B-38F	9c22a25	1910	±	80	1.2414	2371
00-00667	N7B-38DF	9c21a49	2210	±	60	1.2414	2743
00-00667 + spike	N7B-38DF	9c21a50	5760	±	110	1.2414	7150
Spike Recovery			**112%**				
CCV results are reported in ng/ml (ppb)							
5ppb Tc-99		9c21a4	4.56	±	0.22		
5ppb Tc-99		9c21a22	5.22	±	0.18		
5ppb Tc-99		9c21a53	4.97	±	0.02		
5ppb Tc-99		9c22a4	4.83	±	0.15		
5ppb Tc-99		9c22a28	4.74	±	0.10		
100ppb Co		9c21a7	0.028	±	0.0002		
100ppb Co		9c22a7	0.031	±	0.009		

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: D. Kurath

ACL Number(s): 00-0380

Client ID: "N7-Tc-eff-comp"

ASR Number: 5582

Total Samples: 1

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename): 11-11-99 (A0554) & 11-17-99 (A0556)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

1-13-00
Reviewed by

1-14-00
Concur

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

One radioactive aqueous sample, **N7-Tc-Eff-Comp** (ACL# 00-0380) was analyzed by ICPAES after the sample was dilute 10-fold using 2% nitric acid by the Sample Receiving and Preparation Laboratory (SRPL). Sodium was the only analyte requested. The sample was analyzed on two different days unintentionally. An analytical dilution of 5-fold and 25-fold was made due to the high concentration of sodium present in the sample. Results are calculated as shown below and reported as molarity of sodium (mols per liter). Preliminary results were sent by e-mail to the client earlier. The previously reported results were 4.97 M Na and 4.80 M Na, which is 0.01 M Na lower than results reported below. The difference is due to decimal value rounding.

Sodium concentration, as measured by ICP, was corrected for calibration drift by multiplying the ratio of a single element sodium standard ("true" value) to the concentration of the standard as measured by ICP.

Sodium molarity is calculated as follows:

[Na] = $(\mu g/ml)_{ICP}$ * $\{(\mu g/ml\ std)_{TRUE} / (\mu g/ml\ std)_{MEASURED}\}$ * 1.0E-03 (conversion factor to convert ug/ml to g/liter) / gram formula wt of sodium (22.9898 g/L) = mols Na

First analysis (11-11-99):

[Na] = 119000 µg/ml * [(500 µg/ml "True")/(520 µg/ml "measured")] * 1.0E-03) / 22.9898 g/L = **4.98** M Na

Second analysis (11-17-99):

[Na] = 106000 µg-Na/ml * [(1000 µg/ml"True")/(963 µg/ml "measured")] * 1.0E-03 / 22.9898 g/L = **4.79** M Na

Average [Na]= **4.88** M ± 0.09 M Na

Measurement results reported have been corrected for preparation and analytical dilution. Weights and volumes used have been recorded on bench sheets and are included with this report.

Quality control check-standard results for sodium met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:
(Aqueous sample)

All results for sodium in the diluted sample was within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):
(Aqueous sample)

All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):
(Aqueous sample)

All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):
(Aqueous sample)

All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:
(Aqueous sample)

None.

Matrix Spiked Sample:
(Aqueous sample)

None.

Quality Control Check Standards (aqueous sample):

Concentration for all analytes of interest is within tolerance limit of ± 10% accuracy except for a slightly high recovery for sodium in the standard QC_MCVA. One of five measurement results for sodium was 12%. The other four measurement results were within tolerance. Sodium was also measured using a single element standard that was measured about the same time as the sample and was within acceptable tolerance limits.

High Calibration Standard Check (aqueous sample):

Verification of the high-end calibration concentration for all analytes of interest is within tolerance of ± 5% accuracy.

Process Blank:
(Aqueous sample)

None.

Laboratory Control Standard (LCS):
(Aqueous sample)

None.

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	250.0		50.0		1.0			
	ALO#=	00-0380 @25		00-0380 @5		Na $ 500 PPM			
	Client ID=	N7-Tc-eff-comp		N7-Tc-eff-comp					
Det. Limit	Run Date=	11/11/99		11/11/99		11/11/99			
(ug/mL)	(Analyte)	ug/mL		ug/mL		ug/mL			
0.025	Ag	--	=========	--	=========	--	=========	--	=========
0.060	Al	2460		2360		--		--	
0.100	As	--	=========	--	=========	--	=========	--	=========
0.050	B	--		[16]		--		--	
0.010	Ba	--	=========	--	=========	--	=========	--	=========
0.010	Be	--		--		--		--	
0.100	Bi	--		--		--		--	
0.250	Ca	140-		159		--		--	
0.015	Cd	[28]	=========	27.1	=========	--	=========	--	=========
0.200	Ce	--		--		--		--	
0.050	Co	--		--		--		--	
0.020	Cr	[47]	=========	44.5	=========	--	=========	--	=========
0.025	Cu	[6.8]		12.8		--		--	
0.050	Dy	--		--		--		--	
0.100	Eu	--		--		--		--	
0.025	Fe	[6.5]		[8.6]		--		--	
2.000	K	[700]		[740]		--		--	
0.050	La	--		--		--		--	
0.030	Li	--		--		--		--	
0.100	Mg	--		--		--		--	
0.050	Mn	--		--		--		--	
0.050	Mo	[16]		[17]		--		--	
0.150	Na	119000		>50000		520		--	
0.100	Nd	--		--		--		--	
0.030	Ni	224		216		--		--	
0.100	P	308		300		--		--	
0.050	Pb	[56]	=========	60	=========	--	=========	--	=========
0.750	Pd	--		--		--		--	
0.300	Rh	--		--		--		--	
1.100	Ru	--		--		--		--	
0.500	Sb	--		--		--		--	
0.050	Se	--	=========	--	=========	--	=========	--	=========
0.500	Si	--		--		--		--	
0.750	Sn	--		--		--		--	
0.015	Sr	149		141		--		--	
1.500	Te	--		--		--		--	
1.000	Th	--		--		--		--	
0.025	Ti	--		--		--		--	
0.500	Tl	--		--		--		--	
2.000	U	--		--		--		--	
0.050	V	--		--		--		--	
2.000	W	--		--		--		--	
0.050	Y	--		--		--		--	
0.050	Zn	--		[9.3]		--		--	
0.050	Zr	--		[3.1]		--		--	

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= (Analyte)	250.0 / 00-0380 @25 / N7-Tc-Eff-Comp / 11/17/99 ug/mL		50.0 / 00-0380 @5 / N7-Tc-Eff-Comp / 11/17/99 ug/mL		1.0 / Na $1000 PPM / 11/17/99 ug/mL			
0.025	Ag	--	=========	--	=========	--	=========	--	=========
0.060	Al	2420		2400		--		--	
0.100	As	--	=========	--	=========	--	=========	--	=========
0.050	B	--		[16]		--		--	
0.010	Ba	--	=========	--	=========	--	=========	--	=========
0.010	Be	--		--		--		--	
0.100	Bi	--		--		--		--	
0.250	Ca	[150]		172		--		--	
0.015	Cd	[28]	=========	27.8	=========	--	=========	--	=========
0.200	Ce	--		--		--		--	
0.050	Co	--		--		--		--	
0.020	Cr	[46]	=========	45.1	=========	--	=========	--	=========
0.025	Cu	[8.6]		14.0		--		--	
0.050	Dy	--		--		--		--	
0.100	Eu	--		--		--		--	
0.025	Fe	[7.6]		[10.0]		--		--	
2.000	K	[730]		[740]		--		--	
0.050	La	--		--		--		--	
0.030	Li	--		--		--		--	
0.100	Mg	--		--		--		--	
0.050	Mn	--		--		--		--	
0.050	Mo	[17]		[17]		--		--	
0.150	Na	106000		>55000		963		--	
0.100	Nd	--		--		--		--	
0.030	Ni	226		220		--		--	
0.100	P	302		307		--		--	
0.050	Pb	[60]	=========	61.4	=========	--	=========	--	=========
0.750	Pd	--		--		--		--	
0.300	Rh	--		--		--		--	
1.100	Ru	--		--		--		--	
0.500	Sb	--		--		--		--	
0.050	Se	--	=========	--	=========	--	=========	--	=========
0.500	Si	--		--		--		--	
0.750	Sn	--		--		--		--	
0.015	Sr	137		134		--		--	
1.500	Te	--		--		--		--	
1.000	Th	--		--		--		--	
0.025	Ti	--		--		--		--	
0.500	Tl	--		--		--		--	
2.000	U	--		--		--		--	
0.050	V	--		--		--		--	
2.000	W	--		--		--		--	
0.050	Y	--		--		--		--	
0.050	Zn	--		[8.8]		--		--	
0.050	Zr	--		[3.0]		--		--	

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # **5628**
Client: D. Blanchard
WP# W48414

File: L:\radchem\hydroxide\asr5628
Analysis Date: **1/5/2000**
Print Date: 1/7/00

Governing Procedures: **RPG-CMC-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

Equip # **WB76843** Lab Loc. 525

Analyst: [signature] 1-7-00
Reviewer: [signature] 1-7-00

Titrant	Molarity
HCl	**0.2034**
pH 7.0 reading =	6.91

Std. & Spike	Molarity
NaOH	**0.1018**

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-0641	**N7-Tc-PW1**		0.200	0.2476	1.238	18	11.877	0.637	10.815	0.130	0.65	
00-0641	**N7-Tc-PW1**	Replicate	0.200	0.2497	1.249	19	11.719	0.657	10.660	0.134	0.67	3.09%
00-0642	**N7-Tc-PW3**		0.200	0.2486	1.243	20	11.689	0.65	10.643	0.132	0.66	
00-0642	**N7-Tc-PW3**	Replicate	0.200	0.2479	1.240	21	11.620	0.633	10.627	0.129	0.64	2.65%
00-0643	**N7-Tc-PW5**		0.300	0.3221	1.074	22	11.566	0.594	10.462	0.121	0.40	
00-0643	**N7-Tc-PW5**	Replicate	0.500	0.5388	1.078	23	11.877	1.042	10.571	0.212	0.42	5.12%
00-0644	**N7-Tc-PW7**		0.500	0.5079	1.016	24	10.632	0.321	7.073	0.065	0.13	
00-0644	**N7-Tc-PW7**	Replicate	0.500	0.5045	1.009	25	10.578	0.328	4.340	0.067	0.13	2.16%
00-0645	**N7-Tc-PR1**		0.500	0.5051	1.010	26	10.175	0.270	7.119	0.055	0.11	
00-0645	**N7-Tc-PR1**	Replicate	1.000	1.0069	1.007	27	11.154	0.437	10.087	0.089	0.09	21.08%
00-0646	**N7-Tc-PR2**		0.500	0.5029	1.006	28	10.906	0.254	7.102	0.052	0.10	
00-0646	**N7-Tc-PR2**	Replicate	1.000	1.0105	1.011	29	11.105	0.439	9.723	0.089	0.09	14.57%
00-0664	**N7-Tc-R01**		0.500	0.5211	1.042	30	11.556	2.142	6.777	0.436	0.87	
00-0664	**N7-Tc-R01**	Replicate	0.500	0.5204	1.041	31	11.609	2.042	7.363	0.415	0.83	4.78%
QC Data:												
Reag. Blk.1			5.00			1	3.284					
Reag. Blk.2			5.00			14	4.328			**OH % Recovery**		
Standard 1	0.1018 N NaOH		5.000	5.034	1.007	2	11.426	2.428	10.457	0.4939	**97.0%**	Std 1
Standard 2	0.1018 N NaOH		5.000	5.0464	1.009	3	12.088	2.391	10.601	0.4863	**95.5%**	Std 2

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

ASR # **5628**

WP# W48414

File: L:\radchem\hydroxide\asr5628
Analysis Date: **01/05/2000**
Print Date: 1/6/00

Governing Procedures: **RPG-CMC-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator

Equip # **WB76843** Lab Loc. 525

Analyst: 1-6-2000
Reviewer: Beamwood 1-6-00

Titrant	Molarity
HCl	**0.2034**
pH 7.0 reading =	6.91

Std. & Spike	Molarity
NaOH	**0.1018**

RPG #	Sample ID	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	OH 1st Equivalence Point Titrant Vol. (mL)	OH 1st Equivalence Point pH	Found millimoles base	Molarity base	millimole RPD
Standard 3	0.1018 N NaOH	5.000	5.0329	1.007	35	11.698	2.422	7.544	0.4926	**96.8%**	Std 3
00-0641MS	00-0641 + 2mL 0.1N NaOH	0.100	0.1221	1.221	32	11.670	1.256	10.347	0.255	**93.2%**	MS
00-0643MS	00-0643 + 2mL 0.1N NaOH	0.300	0.3191	1.064	33	11.701	1.536	10.179	0.312	**92.5%**	MS
00-0664MS	00-0664 + 2mL 0.1N NaOH	0.500	0.5219	1.044	34	11.742	2.922	10.001	0.594	**82.9%**	MS

Buffer	Fisher Lot #	CMS#	Expire Date
10	SB115-500	179557	May-01
4	SB101-500	179554	May-01
7	SB107-500	179555	May-01

Performance checks using Balance # 360--01-06-037

Pipet #	Vol.	Wt.
H30762	5.00	4.9796
223382	0.300	0.2971
223382	0.100	0.0992
12218	0.500	0.5029

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
0

ASR # 5628
WP# W48414

File: L:\radchem\hydroxide\asr5628
Analysis Date: 01/05/2000
Print Date: 1/6/00

Governing Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
Operation of Brinkman 636 Auto-Titrator
Equip # WB76843

Analyst: 1-6-2000
Reviewer: 1-6-00

Titrant	Molarity
HCl	0.2034

RPG #		Sample Vol. (mL	CO3 2nd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	olarity base	illimole RPD	HCO3 3rd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-0641	0	0.200	1.538	7.813	0.183	0.916		2.365	4.500	0.168	0.84	
00-0641	Replica	0.200	1.569	7.577	0.186	0.928	1.21%	2.308	4.871	0.150	0.75	11.2%
00-0642	0	0.200	1.541	7.584	0.181	0.906		2.297	4.667	0.154	0.77	
00-0642	Replica	0.200	1.544	7.515	0.185	0.926	2.22%	2.298	4.650	0.153	0.77	0.3%
00-0643	0	0.300	0.890	6.977	0.060	0.201		1.147	3.924	0.052	0.17	
00-0643	Replica	0.500	1.491	7.410	0.091	0.183	9.41%	1.838	4.753	0.071	0.14	20.98%
00-0644	0	0.500	0.352	3.771	0.006	0.013						
00-0644	Replica	0.500										
00-0645	0	0.500	0.297	4.058	0.005	0.011						
00-0645	Replica	1.000	0.534	6.917	0.020	0.020	56.95%	0.588	3.637	0.011	0.01	
00-0646	0	0.500	0.275	3.998	0.004	0.009						
00-0646	Replica	1.000	0.509	7.256	0.014	0.014	50.00%	0.556	3.451	0.010	0.01	
00-0664	0	0.500	2.193	3.643	0.010	0.021						
00-0664	Replica	0.500	2.097	3.784	0.011	0.022	7.55%					
						CO3 % Recovered				HCO3 % recovered		
Standard 1		5.000	2.598	7.926	0.03458	6.8%	sample	2.735	3.909	0.0279	5.5%	
Standard 2		5.000	2.588	7.826	0.04007	7.9%	sample	2.738	4.144	0.0305	6.0%	

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
0

ASR # 5628
WP# W48414

File: L:\radchem\hydroxide\asr5628
Analysis Date: 01/05/2000
Print Date: 1/6/00

Governing Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
Operation of Brinkman 636 Auto-Titrator
Equip # WB76843

Analyst: [signature] 1-6-2000
Reviewer: [signature] 1-6-00

Titrant	Molarity
HCl	0.2034

RPG #		Sample Vol. (mL	CO3 2nd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	olarity base	illimole RPD	HCO3 3rd Equivalence Point Titrant ol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
Standard 3		5.000	2.479	3.718	0.01159	**2.3%**	sample					
00-0641MS		0.100	1.759	7.125	0.10231	**111%**	sample	2.162	4.281	0.0820	**103%**	sample
00-0643MS		0.300	1.848	7.007	0.06346	**110%**	sample	2.089	4.212	0.0490	**104%**	sample
00-0664MS		0.500	3.116	7.423	0.03946	**366%**	sample	3.276	3.352	0.0325		sample

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0.100-mL of sample for each matrix spike.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

Chem Rec_51a

Prep date: 04/18/1999

Preparation of Standardized 0.2 M HCl

WP# K51300

Standardized O.1021 M NaOH will be re-checked and then used to standardized the ~ 0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37 --prep.date 2-25-98 for original data) and re-verified against NIST SRM84j Potassium Acid Phthalate KHC8H404 (KAP) = 204.23 g/mole -- Barcode # 52232 --- (see below verification check).
The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.
Prepared 1- liters of ~0.2 M HCl by diluting 100 mL of 1.029M HCl (Chemrec_10) to 0.5 L with DI. H2O.
20 mL aliquots of 0.2 M HCl were were neutralized to the phenopthalien endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipitting error is estimated to be < 1% @ 1s. Thus total error is < 3 % for the measurements

NaOH Molarity veification

Verification Test #	Wt. of KAP	Vol. of **0.1021M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.80894	38.95	0.1017	
2	0.80582	38.84	0.1016	
3	0.96233	46.12	0.1022	
		Ave=	**0.1018**	0.0003
			re-certified value	

Titration Id.	aliquot of sample	Vol. of **0.1018M NaOH** to neutralize	**Molarity of Acid** in Sample	Molarity Error +/- @ 1 s
1	20.00	39.88	0.2030	
2	20.00	39.92	0.2032	
3	20.00	40.04	0.2038	
		Ave Molarity HCl =	**0.2034**	0.00042

Verification Ck on 11/12/99

1	10.00	20.10	0.2047	
2	10.00	20.05	0.2041	
3	10.00	20.00	0.2036	
		Ave Molarity HCl =	**0.2041**	0.00051

This value checks very well with original.

Analyst/Date 1-6-2000

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: D. Blanchard

ACL Number(s): 00-0615 to 00-0619
&
00-0626 to 00-0664

Client ID: "N7-Tc-Elu-Comp" to "C1-Cs-E2-Composite"
&
"N7-Tc-0" to "N7-Tc-R01"

ASR Number: 5626 & 5628

Total Samples: 13

Procedure: PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICP-AES).

Analyst: D.R. Sanders

Analysis Date (Filename): 12-16-99 (A0570) & 12-17-99 (A0571)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Jerry Wagner 12-29-99
Reviewed by

MD Thun 1-4-00
Concur

12/29/99

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Five radioactive liquid samples, N7-Tc-Elu-Comp to C1-Cs-E2-Composite (ACL# 00-0615 to 00-0619), were analyzed by ICPAES after each sample was diluted using dilute nitric acid by the Sample Receiving and Preparation Laboratory (SRPL). Final sample volume was 25ml (weighed). Concentration was adjusted for dilution using the weight of final solution divided by sample weight.

Eight radioactive liquid samples, N7-Tc-0 to N7-Tc-R01 (ACL# 00-0626 to 00-0664), were analyzed by ICPAES after samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. Approximately 3ml of sample (weighed) was digested and diluted to a final volume of approximately 10ml (weighed). Concentration was adjusted for dilution from processing using the weight of final solution divided by sample weight.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in μg/ml for liquid samples. Weights have been recorded on bench sheets and included with this report.

Liquid samples contained low to high concentrations of sodium (<0.1% to about 10%). All other analytes measured were typically much lower in concentration.

Quality control check-standard results met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Aqueous samples) All results for analytes of interest were within tolerance limit of ≤ 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

(Aqueous samples) All analytes of interest were recovered within tolerance limit of ≤ 20% relative percent difference (RPD).

Post-Spiked Samples (Group A):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:
(Aqueous samples) None required or prepared.

Matrix Spiked Sample:
(Aqueous samples) None required or prepared.

Quality Control Check Standards (aqueous samples):
Concentration of all analytes was within tolerance limit of ± 10% accuracy in the standards: QC_MCVA, QC_MCVB, ICP98.0 and QC_SSTMCV except as follows. Tin and palladium was low by as much as 23% in QC_MCVB check standard measurements. Single element standards of tin at 2 µg/ml and palladium 2 µg/ml measured were well within the tolerance limit thus confirming calibration check for these two analytes.

High Calibration Standard Check (aqueous samples):
Verification of the high-end calibration concentration for all analytes is within tolerance of ± 5% accuracy.

Process Blank:
(Aqueous samples) All analytes are within tolerance limit of ≤ EQL or < 5% of sample concentration.

Laboratory Control Standard (LCS):
(Aqueous samples) No LCS was prepared for PNL-ALO-128 acid digested samples.

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	1.0	5.2	5.2	10.3	10.3
	ALO#=	00-0615-DB	00-0615	00-0615-DUP	00-0616	00-0617
	Client ID=	Dilution Blank	N7-Tc-Elu-Comp	N7-Tc-Elu-Comp	A1-Cs-E2-Comp	A1R-Cs-E-Comp
Det. Limit	Run Date=	12/16/99	12/16/99	12/16/99	12/16/99	12/16/99
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
0.025	Ag	--	--	--	--	--
0.060	Al	--	[0.47]	[0.44]	[1.3]	[6.1]
0.250	As	--	--	--	--	--
0.050	B	--	10.5	10.5	7.78	7.42
0.010	Ba	--	[0.11]	[0.096]	[0.23]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[1.9]	--	[3.3]	[2.9]
0.015	Cd	--	--	--	[0.34]	[0.23]
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	6.54	3.25
0.025	Cu	--	--	--	12.7	7.13
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	12.8	8.09
2.000	K	--	--	--	[190]	[210]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	--	105	105	698	1,150
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.044]	[0.18]	--	[2.8]	5.69
0.100	P	--	--	--	--	--
0.100	Pb	--	--	--	12.8	[9.9]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	[23]	[22]	[6.5]	[6.3]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	6.20	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	10.3	10.4	1.0	15.1	14.6
	ALO#=	00-0618	00-0619	00-0626-BLK1	00-0626 @5	00-0641 @5
	Client ID=	C1-Cs-E1-Composite	C1-Cs-E2-Composite	Process Blank-1	N7-Tc-0	N7-Tc-PW1
Det. Limit	Run Date=	12/16/99	12/16/99	12/16/99	12/16/99	12/16/99
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
0.025	Ag	--	--	--	--	--
0.060	Al	[3.6]	[4.6]	--	2,070	1,920
0.250	As	--	--	--	--	--
0.050	B	12.5	13.8	--	25.4	33.5
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[5.3]	[0.34]	146	141
0.015	Cd	[0.50]	[0.59]	--	24.2	22.8
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	[1.8]	[1.8]
0.020	Cr	5.25	5.29	--	37.2	35.3
0.025	Cu	20.3	39.3	--	11.9	11.3
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	7.23	7.63	--	7.35	7.00
2.000	K	--	--	--	641	620
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	[1.1]	[1.0]
0.050	Mo	--	--	--	14.1	13.5
0.150	Na	920	1,630	--	99,600	90,500
0.100	Nd	--	--	--	[2.1]	[2.7]
0.030	Ni	67.5	36.7	[0.047]	181	174
0.100	P	--	--	--	266	245
0.100	Pb	[7.7]	[10]	--	52.2	50.7
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[15]	[39]	--	[65]	85.7
1.500	Sn	--	--	--	--	--
0.015	Sr	[0.84]	[0.49]	--	115	110
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	[87]	[170]	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	[66]	[63]
0.050	Y	--	--	--	[1.0]	[0.98]
0.050	Zn	[0.84]	5.40	[0.13]	[5.7]	[5.8]
0.050	Zr	--	--	--	[2.1]	[1.1]

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
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3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

	Multiplier=	14.7	16.4	3.4	3.4	3.8
	ALO#=	00-0642 @5	00-0643 @5	00-0644	00-0645	00-0646
	Client ID=	N7-Tc-PW3	N7-Tc-PW5	N7-Tc-PW7	N7-Tc-PR1	N7-Tc-PR2
Det. Limit	Run Date=	12/16/99	12/16/99	12/16/99	12/16/99	12/16/99
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
0.025	Ag	--	--	--	--	--
0.060	Al	1,870	398	21.1	13.5	8.37
0.250	As	--	--	--	--	--
0.050	B	34.1	26.8	19.3	18.7	22.4
0.010	Ba	--	--	[0.041]	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	134	[31]	[3.0]	[2.2]	[1.4]
0.015	Cd	22.1	4.72	[0.24]	[0.14]	--
0.200	Ce	--	--	--	--	--
0.050	Co	[1.8]	--	--	--	--
0.020	Cr	34.5	7.59	[0.39]	1.17	[0.69]
0.025	Cu	10.9	[2.3]	[0.16]	[0.14]	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	6.81	[1.6]	[0.44]	[0.25]	[0.81]
2.000	K	613	[100]	[14]	[9.9]	[7.6]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	[0.95]	--	--	[0.24]	--
0.050	Mo	13.2	[2.7]	--	--	--
0.150	Na	91,500	31,100	3,420	2,920	2,740
0.100	Nd	[2.7]	--	--	--	--
0.030	Ni	168	38.2	2.22	1.63	[0.66]
0.100	P	245	73.5	5.49	4.63	[1.8]
0.100	Pb	49.1	[11]	[0.71]	--	--
0.750	Pd	--	--	[2.9]	[2.9]	[3.0]
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	90.1	142	107	123	155
1.500	Sn	--	--	--	--	--
0.015	Sr	106	24.0	1.31	[0.34]	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	[61]	--	--	--	--
0.050	Y	[0.93]	--	--	--	--
0.050	Zn	[5.2]	[1.6]	[0.45]	[0.43]	[0.35]
0.050	Zr	[2.1]	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

Det. Limit (ug/mL)	Multiplier= / ALO#= / Client ID= / Run Date= / (Analyte)	16.9 / 00-0664 @5 / N7-Tc-R01 / 12/16/99 / ug/mL				
0.025	Ag	--	--	--	--	--
0.060	Al	11.2	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	34.7	--	--	--	--
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.44]	--	--	--	--
2.000	K	--	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	19,200	--	--	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	--	--	--	--	--
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	310	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	[0.38]	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # **5626**
Client: D. Blanchard
WP# **W48409**

File: L:\radchem\hydroxide\asr5526
Analysis Date: **1/4/2000**
Print Date: 1/7/00

Hydroxide and Alkalinity Determination
Governing Procedures: **RPG-CMC-228:** Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates and Operation of Brinkman 636 Auto-Titrator
Equip # **WB76843**
Lab Loc. 525

Analyst: 1-7-00
Reviewer: 1-7-00

Titrant	Molarity
HCl	**0.2034**
pH 7.0 reading =	6.92

Std. & Spike	Molarity
NaOH	**0.1018**

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Titrator Routine #	Initial pH reading	1st Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-00615	**N7-TC-Elu-comp**		2.000	2.0262	1.013	12	9.371	0.023	6.579	0.005	0.002	
00-00615	**N7-TC-Elu-comp**	Dup.	5.000	5.0335	1.007	13	9.954	0.057	7.463	0.012	0.002	0.87%
QC Data:												
Reag. Blk.1			5.00			1	3.284					
Reag. Blk.2			5.00			14	4.328			**OH % Recovery**		
Standard 1	0.1018 N NaOH		5.000	5.034	1.007	2	11.426	2.428	10.457	0.4939	**97.0%**	
Standard 2	0.1018 N NaOH		5.000	5.0464	1.009	3	12.088	2.391	10.601	0.4863	**95.5%**	
00-0615MS	00-0615 + 2mL 0.1N NaOH		2.000	1.9957	0.998	17	11.783	0.956	7.753	0.194	**93.2%**	MS

Buffer	Fisher Lot #	CMS#	Expire Date
10	SB115-500	179557	May-01
4	SB101-500	179554	May-01
7	SB107-500	179555	May-01

Performance checks

Balance # 360--01-06-037

		Vol.	Wt.
Pipet #	H30762	5.00	5.0087
Pipet #	H30762	0.30	0.2971
Pipet #	307880	1.00	1.0091

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # 5626
WP# W48409

File: L:\radchem\hydroxide\asr5526
Analysis Date: 01/04/2000
Print Date: 01/06/2000

Hydroxide and Alkalinity Determination
Governing Procedures: RPG-CMC-228: Determination of Hydroxyl (OH-) and Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: [illegible] 1-6-2000
Reviewer: [illegible] 1-6-00

Titrant	Molarity
HCl	0.2034

RPG #	Sample ID		Sample Vol. (mL)	2nd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD	3rd Equivalence Point Titrant Vol. (mL)	pH	Found millimoles base	Molarity base	millimole RPD
00-00615	N7-TC-Elu-comp	0	2.000	0.033	4.812	0.002	0.001						
00-00615	N7-TC-Elu-comp	Dup.	5.000	0.087	4.435	0.006	0.001	18.18%					
							CO3 % Recovered						
Std 1	Standard 1		5.000	2.598	7.926	0.03458	6.8%	sample	2.735	3.909	0.0279	5.5%	
Std 2	Standard 2		5.000	2.588	7.826	0.04007	7.9%	sample	2.738	4.144	0.0305	6.0%	
00-0615MS	00-0615 + 2mL 0.1N NaOH		2.000	1.033	3.884	0.01566	7.7%	sample					

Matrix spike recovery is calculated as follows:
Spike = 2.00 mL 0.1018 N NaOH was added to the 0..100-mL of sample for each matrix spilke.
SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH.
meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client: D Blanchard

Charge Code/Project: W48409/29953-75%
W48414/29953-25%

ACL Numbers: 00-0615 to 00-0619, 00-0626

ASR Number: 5626 and 5628

Analyst: MJ Steele

Analysis Date: December 18-20, 1999

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

Final Results:

Lab ID	Sample ID	(1) F ug/ml	Cl ug/ml	NO2 ug/ml	Br ug/ml	NO3 ug/ml	PO4 ug/ml	SO4 ug/ml	C2O4 ug/ml
00-00615	N7-Tc-Elu-Comp	< 0.3	15	8	< 0.3	19	< 0.5	1	< 0.5
00-00615 MS	N7-Tc-Elu-Comp MS Rec	94%	OvrRng	101%	103%	103%	103%	103%	101%
00-00616	A1-Cs-E2-Comp	< 100	< 100	< 200	< 100	31,400	< 200	< 200	< 200
00-00616	A1-Cs-E2-Comp Dup	< 100	< 100	< 200	< 100	31,600	< 200	< 200	< 200
	RSD	n/a	n/a	n/a	n/a	1%	n/a	n/a	n/a
00-00617	A1R-Cs-E-Comp	< 100	< 100	< 200	< 100	26,500	< 200	< 200	< 200
00-00618	C1-Cs-E1-Composite	< 100	< 100	< 200	< 100	24,500	< 200	< 200	< 200
00-00619	C1-Cs-E2-Composite	< 100	< 100	< 200	< 100	26,300	< 200	< 200	< 200
00-00626	N7-Tc-0	3,200	480	27,900	< 250	110,000	1,100	3,800	1,400
00-00626 MS	N7-Tc-0 MS Rec	112%	105%	122%	108%	OvrRng	111%	114%	111%

RPD = Relative Percent Difference (between sample and duplicate/replicate)
MS Rec = Matrix Spike Standard % recovery
(1) Fluoride is most likely formate or acetate due to slight peak shift and broadening

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquid samples were diluted at the IC workstation up to 4,000-fold to ensure that all anions were within the calibration range.

Q.C. Comments:

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, the sample, A1-Cs-E2-Comp (00-0616), was split at the IC workstation and analyzed as two samples (replicate). For anions above the estimated quantitation level (i.e., the lowest calibration standard), the Relative Percent Difference (RPD) is will within the acceptance criteria of 20%.

Matrix Spike: Two matrix spikes were prepared from samples submitted. The recoveries for all anions were within the 75% to 125% recovery acceptance criteria for those anions measured within the calibration range.

Blank Spike: No blank spikes were analyzed within the analytical runs.

System Blank/Processing Blanks: Twelve system blanks were process during the analysis of the samples. No anions were detected in the system blanks above the estimate quantitation level.

Quality Control Calibration Verification Check Standards: Seven mid-range verification standards were analyzed throughout the analysis runs. Except for only two oxalate values, the reported results for all anions of interest were recovered within the acceptance criteria of ±10% for the verification standard.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Analyst: M J Steele **Date** 1/13/00

Approval: M W Ellis **Date** 1/14/00

Archive Information:

Files: ASR 5626 5628 Blanchard.doc	ASR 5606 5626 5642.xls

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

Client: D Blanchard

Charge Code/Project: W48409/29953-75%
W48414/29953-25%

ACL Numbers: 00-0615 to 00-0619, 00-0626

ASR Number: 5626 and 5628

Analyst: MJ Steele

Analysis Date: December 18-20, 1999

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

ADDENDUM 1

The MS results reported in the original report are included in the table below.

Lab ID	Sample ID	F µg/ml	Cl µg/ml	NO2 µg/ml	Br µg/ml	NO3 µg/ml	PO4 µg/ml	SO4 µg/ml	C2O4 µg/ml
00-00615 MS	N7-Tc-Elu-Comp MS Rec	94%	OvrRng	101%	103%	103%	103%	103%	101%
00-00626 MS	N7-Tc-0 MS Rec	112%	105%	122%	108%	OvrRng	111%	114%	111%

The chloride for the 00-00615 spike and the nitrate for the 00-0626 spike were over range (OvrRng). The contribution of the sample for the chloride and nitrate were significantly higher than the contribution from the standard spike solution, and the sample was not diluted sufficiently enough to bring the sample plus spike contribution within the calibrations range. Using the over range chloride and nitrate results to calculate the spike recovery, the chloride spike recovery is 69% and the nitrate is 125%.

The QC protocol requires one matrix spike per analytical run for each sample "matrix type". Both samples 00-00615 and 00-00626 are aqueous solutions and as such constitute a single matrix type. The spike recovery is used to ascertain the "estimated" accuracy of the analytical measurement for the matrix type. Although the chloride and nitrate were over range one MS, the other MS (on a similar matrix) is used to ascertain the estimated accuracy.

Also, both samples from which the matrix spikes were generated were analyzed at more than one dilution. Sample 00-00615 produced chloride results of 13.0 µg/ml at 40-fold dilution and 14.9 µg/ml at 10-fold dilutions, and sample 00-00626 produced nitrate results of 113,600 µg/ml and 110,300 µg/ml at 40,000-fold and 10,000-fold dilutions, respectively. Based on this good agreement between serial dilutions, bias due to matrix effect is considered negligible.

Approval: _____ **Date** 5-22-00

Archive Information:

Files: ASR 5626 5628 Blanchard Addendum.doc	ASR 5606 5626 5642.xls

File: 00-0615.xls

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Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

12/22/1999

Client : Blanchard/Kurath

Cognizant Scientist: LR Greenwood Date : 12/22/99

Concur : T Trang-le Date : 12/28/99

Procedure: PNL-ALO-450 *Gamma Energy Analysis*

Measured Activities (uCi/g) with 1-σ error

ALO ID Client ID	Tc-95m* Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0615 N7-Tc-Elu-Comp	4.44E-1 2%	<2.E-4	<3.E-4	<9.E-5	<7.E-4	<7.E-4
00-0615 Dup N7-Tc-Elu-Comp	4.55E-1 2%	<2.E-4	<3.E-4	<1.E-4	<7.E-4	<7.E-4
00-0616 A1-Cs-E2-Comp	<6.E-2	3.99E-2 7%	2.38E+2 2%	<7.E-3	<2.E-1	<2.E-1
00-0617 A1R-Cs-E-Comp	<5.E-2	2.39E-2 8%	1.45E+2 2%	<6.E-3	<9.E-2	<9.E-2
00-0618 C1-Cs-E1-Composite	<3.E-1	<2.E-2	4.67E+2 2%	<4.E-2	<6.E-1	<6.E-1
00-0619 C1-Cs-E2-Composite	<4.E-1	<3.E-2	7.05E+2 2%	<4.E-2	<7.E-1	<7.E-1

* Tc-95m values are reported as of December 17, 1999 at 8:00 am PST.

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

1/18/2000

Client : Blanchard/Kurath

Cognizant Scientist: L R Greenwood Date : 1/18/00

Concur : T Trang-le Date : 1/18/2000

Procedure: PNL-ALO-420/421

Measured Activities (uCi/g) with 1-σ error

ALO ID Client ID	Alpha Error +/-	Sr-90 Error +/-
00-0615 N7-Tc-Elu-Comp	5.73E-6 43%	8.70E-4 5%
00-0616 A1-Cs-E2-Comp	<2.E-5	2.40E-1 4%
00-0617 A1R-Cs-E-Comp	1.62E-5 38%	8.43E-2 3%
00-0618 C1-Cs-E1-Composite	1.20E-4 10%	4.10E-2 8%
00-0619 C1-Cs-E2-Composite	9.86E-5 11%	5.60E-2 9%
00-0619 DUP C1-Cs-E2-Composite		1.41E-1 6%
RPD		86%
Matrix Spike	98%	105%
Blank Spike	108%	103%
Blank	<2.E-5	<7.E-5

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client:	D. Blanchard	**Charge Code:**	75%/25% W48409/W48414
		Project:	29953
ACL Numbers:	00-0615 to 00-0619	**ASR Number:**	5626
Analyst:	MJ Steele	**Analysis Date:**	January 7, 2000

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"

M&TE: Carbon System (WA92040); Balance (360-06-01-023).

Final Results:

Lab Number	Sample ID	Vol ml	TOC µg C/ml	TOC RPD %
00-0615	N7-Tc-Elu-Comp	0.3000	<60	
00-0615 Dup	N7-Tc-Elu-Comp Rep	1.0000	<20	n/a
00-0615 Spike	N7-Tc-Elu-Comp MS	1.0000	104%	
00-0616	A1-Cs-E2-Comp	0.3000	80	
00-0616 Dup	A1-Cs-E2-Comp Rep	1.0000	<20	n/a
00-0617	A1R-Cs-E-Comp	1.0000	<20	
00-0617 Dup	A1R-Cs-E-Comp Rep	1.0000	<20	n/a
00-0618	C1-Cs-E1-Comp	1.0000	150	
00-0618 Dup	C1-Cs-E1-Comp Rep	1.0000	150	0
00-0619	C1-Cs-E2-Comp	1.0000	200	
00-0619 Dup	C1-Cs-E2-Comp Rep	1.0000	190	5

RPD = Relative Percent Difference (between sample and duplicate/replicate)
n/a = Not applicable; either sample or duplicate result is <5 times MDL

See last page for TIC.

The analysis of the subject samples submitted under ASR 5626 were performed by the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC. Per the ASR and since the samples were acidic, only TIC analyses were performed on these samples.

The table above shows the results, rounded to one to two significant figures. The result for A1-Cs-E2-Comp (i.e., 80 µg/ml) is only marginally above the method detection limit for the 0.3 ml sample size analyzed (i.e., 60 µg/ml) and should be considered qualitative. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank

Q.C. Comments:

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC).

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration standards were all within acceptance criteria, with the average recovery being 98.4% for TIC and 98.1% for TOC.

Calibration Blanks: The three calibration blanks run at the beginning and middle of the analysis run were acceptable, averaging 13.9 µgC TIC and 454.7 µgC TOC.

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, each sample was analyzed in replicate and the relative percent differences (RPD) between replicates are within the acceptance criteria of 20% for all values measured above the EQL (i.e., 5 times the method detection limit).

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from sample 00-0615 (N7-Tc-Elu-Comp) with the TOC spike recovery being 103.8%, well within the 75% to 125% recovery acceptance criteria.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.
- Some results may be reported as less than ("<") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by: _____ **Date** 1-11-00

Review/Approval by: _____ **Date** 1-17-00

Archive Information:

Files: ASR 5626 Blanchard.doc	ASR 5478 5626 Liq+Solids.xls

Note added to TOC/TIC report for N7-Tc-Elu-Comp.

Although TIC is not reported, it is assumed to be <20 ug C/mL, as is the TOC. Normally TIC is determined by adding acid to the sample in a sealed vial and observing the quantity of CO2 evolved. TOC is then subsequently determined by opening the vial, adding potassium persulfate and silver nitrate, resealing the vial, heating, and observing the quantity of CO2 evolved. For the N7-Tc-Elu-Comp sample, it was incorrectly assumed that the sample was already acidic, so the persulfate and silver nitrate were added to the vial, it was sealed and heated, and the evolved CO2 was measured. Any evolved CO2 would have come from both TOC and TIC in this case, so the observation of < 20 ug C/mL evolved indicates that there must have been < 20 ug C/mL of either.

As per conversation with Marilyn Steele, 2/23/00.

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Pacific Northwest Laboratories

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Project Number

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329/4 File
Mike Urie

Date December 21, 1999

To Dave Blanchard

From Tom Farmer Thomas Farmer

Subject ICP/MS Analysis of Submitted Samples
(ACL #00-0615 through 00-0619)

Pursuant to your request, the 7 samples that you submitted for analysis were analyzed by ICPMS for ^{99}Tc. The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc standard was used to generate the calibration curve and an independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank. The samples were diluted an extra 10x from the dilutions received. The results include your dilutions and are reported in ng analyte/ g (ppb) of original sample ± one standard deviation.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc. The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Semiquantitative Ru concentrations, corrected for sample dilution, are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624.

ASR 5626

Kurath/Blanchard Tc-99 Analysis

December 21, 1999

Results are reported in ng/g (ppb) of original sample.

Sample ID	Client Number	ICP/MS Number	Tc-99 ng/g ± 1SD	Ru-101/ Ru-102	†Ru-101 ng/g
1%HNO3		9c17a7	0.035±0.005		
1%HNO3		9c17a1	<0.024		
1%HNO3		9c17a18	<0.024		
00-00615DB	Dilution Blank	9c17a8	<1.0	1.0303	0.6
00-00615	N7-Tc-Elu-Comp	9c17a15	3600±170	0.7692	4
00-00615DUP	N7-Tc-Elu-Comp	9c17a16	3510±160	3.2727	8
00-00616	A1-Cs-E2-Comp	9c17a11	27±6	0.0242	0.8
00-00617	A1R-Cs-E-Comp	9c17a12	15±4	0.9706	1
00-00618	C1-Cs-E1-Comp	9c17a13	22±4	0.7927	6
00-00619	C1-Cs-E2-Comp	9c17a14	15±4	1.2948	16
00-00619 + spike	C1-Cs-E2-Comp	9c17a17	547±21		
Spike Recovery			**103%**		
CCV results are reported in ng/ml (ppb)					
5ppb Tc-99		9c17a5	4.72±0.15		
5ppb Tc-99		9c17a19	5.13±0.03		

†Based on response from indium

DATA REVIEW

Reviewed by: O.J. Farmer

Date: 30Dec99 Page 1 of 1

Battelle
Pacific Northwest Laboratories

Project Number

Internal Distribution

329/4 File
Mike Urie

Date February 2, 2000

To Dave Blanchard

From Tom Farmer O.J. Farmer 4 Feb 00

Subject ICP/MS Analysis of Submitted Samples
(ACL #00-00969 through 00-00981)

Pursuant to your request, the 15 samples that you submitted for analysis were analyzed by ICPMS for ^{99}Tc. The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc standard was used to generate the calibration curve and an independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank. The samples were diluted an extra 2x to 10x from the dilutions received. The results include your dilutions and are reported in ng analyte/ g (ppb) of the original sample ± one standard deviation.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc. The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Ru counts, corrected for sample dilution, are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624.

Blanchard Tc-99 Analysis

February 2, 2000

Results are reported in ng/g (ppb) of original sample.

Sample ID	Client ID	ICP/MS Number	Tc-99 ng/g	±	1SD	$^{101}Ru/^{102}Ru$ (*.541)	†^{101}Ru ng/ml
1%HNO3		00202a1	<0.014				
1%HNO3		00202a7	<0.018				
1%HNO3		00202a25	<0.020				
1%HNO3		00202a28	<0.021				
blank		00202a8	1.9	±	0.4	0.595	0.2
00-00969	Tc-SLO	00202a16	2120	±	9	1.192	5080
00-00970	Tc-SL2	00202a9	59.5	±	0.9	1.231	3
00-00970dup	Tc-SL2	00202a10	57.4	±	0.6	1.163	3
00-00971	Tc-SL8	00202a30	46	±	5	1.429	1
00-00972	Tc-SL14	00202a31	50.9	±	0.3	1.221	1
00-00973	Tc-SW2	00202a32	47.8	±	2.8	1.396	2
00-00974	Tc-SW4	00202a14	92.4	±	4.0	1.394	0.5
00-00975	AN-Tc-L1	00202a15	85.2	±	8.8	1.509	1
00-00975 + spike	AN-Tc-L1	00202a24	471	±	20		
Spike Recovery			**98%**				
00-00976	AN-Tc-L7	00202a17	1660	±	90	1.202	4800
00-00977	AN-Tc-L13	00202a18	1660	±	50	1.182	4800
00-00978	AN-Tc-L19	00202a19	1700	±	40	1.192	4800
00-00979	AN-Tc-L25	00202a20	1730	±	50	1.213	4900
00-00980	AN-Tc-W2	00202a21	1610	±	20	1.155	4300
00-00981	AN-Tc-W4	00202a22	403	±	4	1.170	920
00-00981 Duplicate	AN-Tc-W4	00202a23	400	±	3	1.192	950
CCV Results are reported in ng/ml (ppb)							
2ppb Tc-99 CCV		00202a5	2.00	±	0.04		
2ppb Tc-99 CCV		00202a27	1.94	±	0.01		
2ppb Tc-99 CCV		00202a33	1.89	±	0.08		
50ppb Co		00202a26	<0.020				

* Natural $^{101}Ru/^{102}Ru$ ratio.

†Based on response from indium

DATA REVIEW

Reviewed by: O.J. Farmer

Date: 4Feb00 Pages: 1 of 1

Estimation of Tc-99 and Nitrate
In Untreated and Treated Archive AN-107 Samples

Assumptions:

1) Specific activity of Tc-99 = 1.71E-2 Ci/g.
2) Tc-99 conc in archive samples same as found previously for this same material (Blanchard et al., 1997). This is reported as 55 uCi/L, or 3.2 mg/L.
3) Nitrate conc in archive samples same as found previously for this same material (Blanchard et al., 1997). This is reported as 2.2 M.

Nitrate to Tc-99 in untreated archive AN-107 sample:

Tc-99 conc = 3.2 mg/L = 3.2E-5 M
[Nitrate]:[Tc-99] = 2.2 M / 3.2E-5 M = 6.8E+4

Tc-99 in treated archive sample:

Reported mass dilution (mass of final over mass of initial) due to treatment is 1.1247 (Hallen et al., 2000). Reported densities of untreated and treated samples are 1.26 g/mL and 1.23 g/mL, respectively.

Vol. of untreated sample = (1.0000 kg) / (1.26 kg/L) = 0.7937 L
Vol. of treated sample = (1.1247 kg) / (1.23 kg/L) = 0.9144 L
Volume dilution = 0.9144 L / 0.7937 L = 1.152
Tc-99 conc in treated sample = (Tc-99 conc in untreated) / (Vol dil)
= 55 uCi/L/1.152 = 48 uCi/L = 2.8 mg/L = 2.8E-5 M

Nitrate to Tc-99 in treated archive AN-107 sample:

Treatment included addition to make sample 0.075 M in $Sr(NO_3)_2$ (Hallen et al., 2000).

Nitrate = Initial conc (2.2 M), diluted by 1.152, plus 2 * (0.075 M), diluted by 0.95
= 2.05 M
[Nitrate]:[Tc-99] = 2.05 M/ 2.8E-5 M = 7.32E+4

Non-Pertechnetate Fraction in BNFL AN-107 from Batch Contact Results

Average pertechnetate extraction: (87.0% + 74.1%) / 2 = 79.0% of total pertech

Unextracted pertechnetate: 100% - 79% = 21% of total pertech

Total pertechnetate = x%
Total non-pertechnetate = y%
Total Tc = x% + y% = 100%

x + y = 100%

Average total Tc extraction: (22.3% + 11.3%) / 2 = 16.8%

Average total Tc <u>un</u>extracted: 100% - 16.8% = 83.2%

Assume none of the non-pertechnetate is extracted.

Then total Tc <u>un</u>extracted = total non-pertech + unextracted pertech
= y% + 0.21x% = 83.2%
y% + 0.21(100 – y%) = 83.2%
y% = 78.7%

So total non-pertech = 78.7%.

Tc Limit in AN-107 (Env. C) Tc Removal Effluent

Assumptions:

1) Conc Na_2O in Env. C glass = 14 wt% (= 14 g Na_2O / 100 g glass), as per attached email message from Mike Johnson.
2) For max Tc-99 in glass, assume all Na comes from feed. If some Na added, multiply max Tc-99 value determined here by (Na conc after addition): (Na in Tc removal effluent).
3) Glass Density = 2.66 MT/m^3 (=2.66 g/mL)
4) Max Tc-99 activity in glass = 0.1 Ci/m^3 (=0.1 Ci / 1E6 mL = 1E-7 Ci/mL)
5) [Na] in Tc removal effluent = 4.8 M
6) Tc-99 specific activity = 1.71E-2 Ci/g = 1.71E-2 uCi/ug

Na Loading in Glass (g Na / mL glass):

(14 g Na_2O / 100 g glass) * (1 mole Na_2O / 62 g Na_2O) * (2 mole Na / mole Na_2O) * (23 g Na / mole Na) * (2.66 g glass / mL glass) = 0.276 g Na / mL glass

Max Tc:Na in Glass:

(1E-7 Ci Tc-99 / mL glass) / (0.276 g Na / mL glass) = 3.62E-7 Ci Tc-99 / g Na

Na in Tc Removal Effluent (g Na / mL feed):

(4.8 mole Na / L feed) * (23 g Na / mole Na) = 110.4 g Na / L feed

Max Tc in Tc Removal Effluent:

(3.62E-7 Ci Tc-99 / g Na) * (110.4 g Na / L feed) = 40.0 uCi Tc-99 / L feed
= 2.34 mg Tc-99 / L feed

Example: Na added to make vit feed 150 g Na / L feed

New maximum Tc:Na (before the additional Na is added, ie, in Tc removal effluent)
= (150 / 110.4) * 2.34 mg Tc-99 / L feed (before Na addition)
= 3.18 mg Tc-99 / L feed (before Na addition)

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