

Small Column Testing of SuperLig<sup>®</sup> 639  
for Removing <sup>99</sup>Tc from Hanford Tank  
Waste 241-AN-102 Supernate (Envelope  
C) Mixed with Tank 241-C-104 Solids  
(Envelope D) Wash and Permeate  
Solutions

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March 2004

ACCEPTED FOR  
WTP PROJECT USE

Test specification: TSP-W375-00-00028  
Test plan: CHG-TP-41500-013, Rev. 0 TP-RPP-WTP-013, Rev 0.  
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R&T focus area: Pretreatment  
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Battelle Pacific Northwest Division  
Richland, Washington, 99352

## COMPLETENESS OF TESTING

*This report describes the results of work and testing specified by Test Specification TSP-W375-00-00028, Rev 1. and Test Plans CHG-TP-41500-013, Rev. 0 and TP-RPP-WTP-013 Rev 0. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.*

**Approved:**



Gordon H. Beeman, Manager  
WTP R&T Support Project

  
Date

<b>History Sheet</b>			
Rev	Date	Reason for revision	Revised by
0	January 2003	New Document	IE Burgeson
1	February 2004	Noted higher uncertainty in the total <sup>99</sup> Tc values in Tables 3.1, 3.2, 3.3, 3.6, 3.8 and 3.9 as footnotes.	IE Burgeson

## Summary

The River Protection Project-Waste Treatment Plant (RPP-WTP) baseline process for  $^{99}\text{Tc}$  removal from Hanford tank waste is ion exchange. The current flowsheet includes the use of  $\text{TcO}_4^-$ -selective sorbent SuperLig<sup>®</sup> 639 (SL-639) for Tc removal from the aqueous waste fraction. This material has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT. The process testing was performed on a blended tank waste feed that was prepared by combining Tank 241-C-104 (C-104) high-level waste pretreatment stream solutions (filtrate, wash, and leach solutions) with Tank 241-AN-102 (AN-102) tank waste supernate. At the time of testing, this combination represented the baseline process flowsheet for the AN-102 tank waste supernate at the Waste Treatment Plant.

This report documents the results of dual-column testing of the SL-639 sorbent with waste from Hanford Tank AN-102 blended with C-104 filtrate, wash and leach solutions (AN-102/C-104) for Bechtel National, Inc. in accordance with Test Specification TSP-W375-00-00028, Rev.1 (Johnson 2000). Section 5 of the Research and Technology Plan (BNI 2002) identifies the research needs for Tc ion exchange process verification [Statement of Work (SOW) Ref Sec C.6 Std 2(a)(3)(ii) and Work Breakdown Structure (WBS) 02.08.04]. The Tc ion exchange process verification tests are also addressed in Scoping Statement B-47, which is included in Appendix C of the *Research and Technology Plan*. These studies are to verify design and operating parameters for plant-scale ion exchange systems. Test results will also be used to validate ion exchange models.

## Objectives

The Tc ion exchange test objectives were to

- demonstrate the effectiveness of all SL-639 process steps
- prepare loading breakthrough profiles and elution profiles with AN-102/C-104 blended feed
- prepare batch-distribution isotherms
- produce a final effluent that removed at least 25% of the technetium from the AN-102/C-104 blended feed within a process goal of 250 bed volumes (BVs) of feed.

## Conduct of Testing

This report documents the results of dual-column testing of the SL-639 sorbent with the blended waste AN-102/C-104 for Bechtel National, Inc. Two columns were arranged in series with each column containing a 5.0-mL resin bed,  $L/D^{(a)} = 2.1$ . These resin beds were utilized in two previous technetium-removal tests: AW-101 simulant and AP-101 tank waste feed. The AN-102 supernate was combined with permeate from the filtration, washing, and leaching of C-104 solids; this blend was evaporated to concentrate the sodium to  $\sim 4.8 \text{ M Na}$  and processed for Cs removal by ion exchange using SuperLig<sup>®</sup> 644 before removing technetium. Approximately 0.68 L of the prepared waste feed was processed for technetium removal; this volume correlates to  $\sim 136$  BVs of feed. All ion exchange process steps were

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(a)  $L/D$  = ratio of bed height to bed diameter.

tested, including resin-bed preparation, loading, feed displacement, water rinse, elution, and resin regeneration.

## Results and Performance Against Objectives

After processing 136 BVs of feed (680 mL), the  $^{99}\text{Tc}$  lead and lag column final breakthrough ratios,  $C/C_0$ , were 46% and 43%, respectively. The initial feed technetium concentration was 50% non-perpertechnetate, which would not have been retained by the SL-639 resin. The initial  $^{99}\text{Tc}$  decontamination factors (DFs) for the feed passing through the first column and the whole column system (derived from the  $^{99}\text{Tc}$  concentration in the first sample from each column) were 1.9 and 2.2, respectively, indicating that 47% and 55% of the technetium was removed from the initial lead and lag column samples. The final DF of the effluent was 1.8, as measured by the concentration of  $^{99}\text{Tc}$  present in the initial and processed feed. The  $^{99}\text{Tc}$  concentration in the effluent composite was 44.1  $\mu\text{Ci/L}$ , and the initial concentration was 79.5  $\mu\text{Ci/L}$ , providing an overall  $^{99}\text{Tc}$  removal of 44%, which meets the success criteria of  $\geq 25\%$  removal of technetium. Based upon the slight breakthrough of pertechnetate on the lag column, it is predicted that the total technetium removal after processing 250 BVs of feed will meet or exceed the 25% removal target.

The pertechnetate quantitation appears to have suffered from an unidentified interferent. There was no interference observed for the  $^{99}\text{Tc}$  quantitation reported above. The lead and lag column initial pertechnetate breakthrough ratios,  $C/C_0$ , were  $\sim 13\%$ . The final breakthrough ratios were 19% for the lead column and  $\sim 14\%$  for the lag column. The lag column pertechnetate values are scattered and demonstrate an uncertainty of approximately 3%  $C/C_0$ . The initial DFs for pertechnetate (derived from the  $^{99}\text{TcO}_4^-$   $C/C_0$  in the first sample from each column) were 11 for both the lead and lag columns. The concentration of  $\text{TcO}_4^-$  in the effluent composite was 5.45  $\mu\text{Ci/L}$ , and the initial concentration of  $\text{TcO}_4^-$  was 39.9  $\mu\text{Ci/L}$ . The effluent composite concentration demonstrates a pertechnetate removal of 86%, or an overall  $\text{TcO}_4^-$  DF of 7.3.

Both columns were monitored during loading; however, only the lead column was eluted (deionized [DI] water at 65°C). The peak  $^{99}\text{Tc}$  concentration was 48 times the  $^{99}\text{Tc}$  concentration in the feed and was found in the second BV. After this, the elution proceeded relatively quickly; 16 BVs of eluant were required for the  $^{99}\text{Tc}$  concentration to drop below  $C/C_0 = 0.01$ . The  $^{99}\text{Tc}$  concentration of the composited eluate was 180  $\mu\text{Ci/L}$ , which correlates to a  $C/C_0$  of 4.5.

The column distribution value,  $\lambda$ , approximately represents the effective capacity of the SL-639 resin when kinetics are not a significant factor and is the number of BVs processed when the concentration of pertechnetate reaches 50% of the feed concentration ( $C/C_0=0.5$ ). Based upon the pertechnetate batch-distribution measurements and resin-bed density ( $K_d \times$  resin bed density), it was expected that  $\sim 310$  BVs would be processed before reaching  $C/C_0=0.50$  for pertechnetate. However, since there was only a slight change in the pertechnetate breakthrough during the run, it was not possible to experimentally determine the column  $\lambda$  value. An extremely rough  $\lambda$  prediction, which was extrapolated from a change of 13% to 19%  $\text{TcO}_4^-$  breakthrough, resulted in a column  $\lambda$  value of  $\sim 500$  BVs. The batch-contact and column-performance data are summarized in Table S1.

**Table S1.** Summary of Observed and Predicted Column Performance for AN-102/C-104 Blended Feed

Flowrate, BV/h	Extrapolated $\text{TcO}_4^-$ <sup>(b)</sup> Column $\lambda$ Value	Composite Decontamination Factor <sup>(a)</sup>			
		$\text{TcO}_4^-$ (Pertechnetate)	$^{99}\text{Tc}$ (Total Tc)	$^{99}\text{TcO}_4^- K_d$ , mL/g	$\text{TcO}_4^- K_d$ $\lambda$ value
3.0	500	7.3	1.8	620	310
(a) The DF is determined by dividing the final effluent technetium concentration by the initial feed concentration.					
(b) Extrapolated from final pertechnetate breakthrough at 136 BVs (19%) to 50% $C/C_0$ at 500 BVs.					

## Quality Requirements

Battelle—Pacific Northwest Division (PNWD) implemented the RPP-WTP quality requirements in a quality assurance project plan (QAPjP) as approved by the RPP-WTP quality assurance (QA) organization. Activities prior to September 2001 (AN-102 homogenization, subsampling, feed blending, evaporation, Sr/TRU precipitation and Cs IX) were conducted in accordance with PNWD's quality assurance project plan, CHG-QAPjP, Rev.0, which invoked PNWD's Standards Based Management System (SBMS), compliant with DOE Order 414.1A Quality Assurance and 10 CFR 830, Energy/Nuclear Safety Management, Subpart A -- Quality Assurance Requirements. Due to a change in the contract QA requirements, activities from September 2001 on (all analytical data for ion exchange testing) were conducted in accordance with PNWD's quality assurance project plan, RPP-WTP-QAPjP, Rev.0, which invoked NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, Part 2.7. These quality requirements were implemented through PNWD's *Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP)*. The quality of the data gathered during the early experiments was not impacted by the change in requirements.

PNWD addressed verification activities by conducting an Independent Technical Review of the final data report in accordance with procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the Test Plan objectives. The review procedure is part of PNWD's WTPSP Manual.

## Issues

No WTP design or operational issues were identified.



## Terms, Symbols, and Abbreviations

AEA	alpha energy analysis
BNI	Bechtel National, Inc.
BV	bed volume
CUF	crossflow filtration system (cell unit filter)
DF	decontamination factor
DI	distilled and deionized
DOE	U.S. Department of Energy
GEA	gamma energy analysis
IC	ion chromatography
ICP-AES	inductively coupled plasma/atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
ID	inner diameter
KPA	kinetic phosphorescence analysis
$\lambda$	lambda; the number of BV processed at 50% breakthrough
L/D	ratio of bed height to bed diameter
LAW	low-activity waste
MRQ	minimum reportable quantity
OD	outer diameter
PNWD	Battelle—Pacific Northwest Division
QA	quality assurance
QAPjP	quality assurance project plan
RPD	relative percent difference
RPP	River Protection Project
SBMS	Standards Based Management System
SOW	Statement of Work
TC	total carbon
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic
WBS	Work Breakdown Structure

WTP	Waste Treatment Plant
WTPSP	Waste Treatment Plant Support Project Quality Assurance Requirements and Description

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# 1.0 Introduction

The U.S. Department of Energy's Hanford Site in southeastern Washington State contains 177 large underground storage tanks with approximately 55 million gallons of radioactive waste. Plans are underway to separate the waste into high-level and low-activity waste (LAW) streams. The primary unit processes of the Hanford waste-treatment flowsheet for LAW feed are cross-flow filtration, Sr/TRU<sup>(a)</sup> precipitation, cesium and technetium ion exchange, evaporation, and vitrification. After solids are removed, the LAW feed will be passed through ion exchange columns to remove cesium and technetium. The decontaminated LAW feed will contain the bulk of the waste mass and will be vitrified as a low-activity glass and stored at the Hanford Site. The cesium and technetium eluted from the ion exchange columns will be mixed with tank sludge, vitrified as high-level waste, and eventually stored at a federal repository.

This report describes the results of small-column testing of the SL-639 sorbent with 241-AN-102 tank waste supernate combined with wash and leach permeate from 241-C-104 solids. (The 241 prefix, which is common to all Hanford tanks, will not be used hereafter). Approximately 680 mL of the prepared tank waste feed was processed through dual small-scale ion exchange columns. This blended tank feed was processed for Sr/TRU removal, the solids were removed using vacuum filtration (Hallen et al. 2002), and the feed was processed for Cs removal by ion exchange using SuperLig<sup>®</sup>644 (Fiskum et al. 2002) before technetium ion exchange. The removal goal for the combined AN-102/C-104 permeate solution is to reduce the total Tc concentration by at least 25% after processing  $\geq 250$  bed volumes (BVs) of solution.<sup>(b)</sup>

The objectives of this work were to

- demonstrate the effectiveness of all SL-639 process steps, including loading, feed displacement, deionized (DI) water washing, elution, and resin regeneration
- investigate the potential for resin and/or column fouling
- demonstrate the <sup>99</sup>Tc decontamination of an Envelope C material (Tank AN-102 blended with permeate from the filtration, washing and leaching of C-104 solids)
- provide batch-distribution measurements on actual tank-waste feed.

This report discusses the experimental conditions for the column ion exchange test procedure. Work was performed in accordance with Scoping Statement B-47, the test specification TSP-W375-00-00028,<sup>(c)</sup> Rev.1, and test plans CHG-TP-41500-013,<sup>(d)</sup> Rev 0 and TP-RPP-WTP-013, Rev 0.<sup>(e)</sup> The results of the feed composition, the batch distribution, the AN-102/C-104 column test, the comparison of the AN-102/C-104 initial feed and effluent composite composition, and the technetium mass balance are presented.

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(a) TRU = transuranic

(b) ME Johnson. Interoffice Memo to SM Mackay, "Minimum Required Technetium Removal from Low Activity Waste Solutions", CCN#016099C, October 25, 2000. CH2M Hill Hanford Group, Inc. Richland, WA.

(c) Test Specification: "Tank 241-AN-102 and 241-AP-101 Ion Exchange," TSP-W375-00-00028, Rev. 1, December 11, 2000, ME Johnson, CH2MHill Hanford Group, Inc, Richland, WA.

(d) Test Plan: "Actual Waste Ion Exchange Testing for the RPP-WTP Project," CHG-TP-41500-013, Rev 0, January 24, 2000, DL Blanchard, Battelle—Pacific Northwest Division, Richland, WA.

(e) Test Plan: "AP-101 and AN-102/C-104 Actual Waste Ion Exchange Testing " TP-TPP-WTP-013, Rev 0, February 22, 2002, DL Blanchard, Battelle—Pacific Northwest Division, Richland, WA.

## 2.0 Experimental

This section describes the history, the preparation, and the processing of the AN-102/C-104 blended feed before technetium ion exchange. It also describes the preparation and history of the technetium resin beds as well as the technetium processing of the AN-102/C-104 blended feed.

### 2.1 AN-102 Sample History

Hanford site personnel grab sampled the sludge layer of Tank AN-102 in August 2000 and retrieved 30 samples from Riser 22 at six different tank waste heights. Battelle—Pacific Northwest Division received 27 of these samples. Each sample was contained in a 500-mL bottle and consisted of wet solids plus supernatant incidental to obtaining the wet solids. Eight of these samples, which were collected 193 cm from the bottom of the tank, were used to prepare a homogenized composite for the initial characterization and process testing of AN-102 as-received material (Urie et al. 2002a). After verification of the homogenization, 15 subsamples representing the entire composite sample were collected. The samples were identified as *AN-102-AR-A* through *O*. The slurry composite sub sample *AN-102 AR-E* was combined with the supernate decanted from five additional composite subsamples (*AN-102 AR- J, K, L, M, and N*), to form an AN-102 composite slurry consisting of approximately 2 wt% solids (Urie et al. 2002b).

### 2.2 C-104 Sample History

Battelle—Pacific Northwest Division received 14 sample jars of C-104 from Hanford's 222-S laboratory on March 3, 1999, identified as jars 16273 through 16286. Each of the as-received samples was used to prepare a homogenized composite for characterization and process testing. After verification of the homogenization, six composite subsamples representing the entire composite were collected. The samples were identified as *C104 Comp A, B, C, D, E and GL*. Because there was composited material remaining behind in the composite vessel (due to a flat bottom), the mixing vessel was rinsed with a total of 200 mL of 0.01 M NaOH and collected in two additional subsample jars: *C104 RIN 1* and *RIN 2*.

The composite samples *C104 Comp C, C104 Comp D, C104 RIN 1, and C104 RIN 2* were used in crossflow filtrations, water washing, and caustic leaching tests described by Brooks (Brooks et al. 2000). The material from the four subsample jars was dewatered using the crossflow filtration system (CUF). Part of the filtrate resulting from the dewatering step was set aside for blending (samples *C-104 Filtrate #1* and *C-104 Filtrate #2*). The dewatered C-104 sludge was washed with inhibited water (0.01 M NaOH), and part of the water wash permeates were set aside for blending (*C-104 Wash (H<sub>2</sub>O) Filtrate Composite*). After washing, the sludge was caustic leached using 3.0 M NaOH. The leach permeate was combined, and a portion was set aside for blending (*C-104 Caustic Leach Permeate*). The leached C-104 sludge was contacted with inhibited water at elevated temperature. A portion of the resulting caustic wash solution was set aside for blending (*C-104 Caustic Wash Composite*).

### 2.3 Preparation of AN-102/C-104 Blended Feed

The filtrate, leachate, and wash solutions from Tank C-104 processing were blended with the 2-wt% solids AN-102 feed to produce an AN-102/C-104 blended feed. Following the successful compatibility

testing, a majority of the AN-102 composite slurry containing 2-wt% solids (802 g) was combined with five solutions collected in the C-104 process testing (~1570 g) to produce approximately 2 L of AN-102/C-104 blended feed. The blended feed was homogenized, and six sub-samples were collected, representing the entire blended feed. The subsamples were identified as *AN-102 DF A, B, C, D, E* and *TRANS3*. Sample *AN-102 DF A* was used for physical testing and chemical and radioisotope characterization and is reported in Urie et al. (2002b).

The sodium concentration for the blended feed was approximately 3.2 M. To undergo further process testing, the remaining five subsamples of blended feed were concentrated by evaporation using gentle heat (50°C) and a nitrogen sparge. The resulting solution, approximately 1.0 L, had a sodium concentration of 5.3 M (Lumetta et al. 2001). However, the resulting material was unstable toward precipitation, as observed in sample aliquots set aside to monitor solution stability. The main precipitate products were identified as sodium (nitrate, nitrite, and oxalate) and aluminum (hydroxide) salts, which formed within one week and continued to form in the sample aliquots over the next several months. This solid formation was not observed in the bulk material.

## 2.4 Processing of AN-102/C-104 Blended Feed

The evaporated feed was processed for strontium and TRU removal and filtered (Hallen et al. 2002). Although aliquots of the evaporated sample were observed to form crystalline precipitates over time, there was no observed formation of significant solids in the bulk sample of the blended feed before Sr/TRU removal. Because there were insufficient solids, the processed AN-102/C-104 feed was filtered using vacuum filtration to remove solids rather than cross-flow filtration. After filtration, the blended feed was processed for cesium removal by ion exchange (Fiskum et al. 2002). The blended feed did not undergo significant dilution as a result of the Sr/TRU removal or cesium ion exchange processing. The blended feed was processed for technetium removal without further alteration; i.e., no technetium tracer was added to the feed.

## 2.5 SL-639 Resin and Bed Preparation

The SL-639 resin was supplied by IBC Technologies, Inc. from Batch # 010227CTC-9-23. The resin consists of a proprietary organic compound (ligand) attached to spherical styrene beads. SuperLig® 639 functions by extracting the sodium pertechnetate salt-pair from either acidic or basic solutions. The manufacturer reports the mean diameter of the beads ( $D_p$ ) as 0.5 mm. A separate aliquot of this batch of SL-639 resin was sieved to determine the particle-size distribution of the resin. The results are reported in Table 2.1.

A subsample of the as-received resin was pulled from the original 1-L container by scooping ~2-g increments of the resin while rotating and occasionally inverting the resin within the container. This material was *not* sieved. The resin was soaked in a 10-fold excess volume of DI water (14 g of resin soaked in 140 mL of water) for 2 h, the water was decanted, and the resin was soaked a second time in 1.0 M NaOH for 2 h and then decanted. No volume change was observed going from the dry-resin form to the wet-resin form. An aliquot of the resin was transferred to each of the two columns using the following approach. The resin (in 1.0 M NaOH) was transferred to a 10-mL graduated cylinder and allowed to settle overnight. The resin BV was adjusted to  $5.0 \pm 0.05$  mL, slurried, and quantitatively transferred to the ion exchange column.

**Table 2.1.** Sieving Data for SL-639 Batch Number 010227CTC-9-23

Sieve Number	Particle Size, $\mu\text{m}$	Mass of Resin, g	% of Total (mass basis)
Sieve 18	>1000	0.1345	1.35
Sieve 30	1000–600	2.6099	26.1
Sieve 40	600–425	5.9904	60.0
Sieve 50	425–300	1.1000	11.0
Sieve 70	300–212	0.1456	1.46
Sieve 100	212–150	0.0075	0.08
<b>Totals</b>		9.9879	100.0

The settled resin bed height was 3.1 cm, with a BV of 5.0 mL and a length-to-diameter ratio (L/D) of 2.1. The ratio of the column diameter ( $D_c$ ) to the diameter of the resin beads ( $D_p$ ) was approximately 20-30:1, which is a large enough  $D_c/D_p$  ratio to avoid wall effects during small-scale column tests. The resin beds were gently packed with approximately 0.7 cm of quartz glass wool to prevent the resin from floating during contact with the AN-102/C-104 blended feed.

Duplicate fractions of the as-received resin (0.5 g each) were dried in a vacuum oven at 50°C until the dry mass was constant (within  $\pm 0.0006$  g). This allowed a determination of the F factor (0.9931), which is the ratio of the mass of the dried exchanger to the mass of the wet exchanger. The bed density was determined by weighing 5.0 mL of exchanger in a 5-mL graduated cylinder. Based on an as-received bed density of 0.50 g/mL and an F factor of 0.993, the dry mass of SL-639 in each column is estimated to be 2.5 g. Table 2.2 summarizes these values.

**Table 2.2.** Summary of Resin and Column Parameters

Resin Parameter	Value	Column Parameter	Value
Batch ID	010227CTC-9-23	Resin Bed Height	3.1 cm
Resin F Factor	0.9931	Resin BV	5.0 mL
Resin Bulk Dry Density	0.50 g/mL	Mass Resin in Column	2.5 g
Particle Density	1.07 g/mL <sup>(a)</sup>	Column Diameter to Resin Diameter Ratio	20–30
Resin Diameter	460–670 $\mu\text{m}$ <sup>(b)</sup>	Column Length to Column Diameter Ratio	2.1
(a) Particle density represents the grams of dry solids divided by the volume of wet resin particles (“as-received resin”) determined with a volumetric flask.			
(b) Weight-percent-based resin diameter determined from sieve data.			

## 2.6 History of Technetium Processing with Resin Bed

After preparing the column resin beds as described in Section 2.5, the column system was tested using an AW-101 simulant. Approximately 2.7 L of <sup>99</sup>Tc-spiked AW-101 simulant were processed through the dual columns. Once in contact with the simulant feed (1.24 g/mL), the resin bed began to float. As a result of this observation, all subsequent testing of the column system was performed using a quartz glass wool plug between the resin bed and the feed plunger to mitigate floating. All steps

associated with the process removal of technetium were performed in the AW-101 simulant shake-down test: resin loading, feed displacement, water rinse, elution, and resin regeneration. In this particular case, both columns were eluted and regenerated. The system was taken to 50% loading of technetium on the lead column (2.7 L at 64  $\mu\text{Ci/L}$  technetium). Elution was performed for each column until the  $C/C_0$  was  $<0.01$ .

An actual tank waste run was then performed using tank waste supernate from AP-101 (Burgeson et al. 2002a). The column system remained in the same configuration during the AP-101 actual tank waste run; i.e., the lead column in the AW-101 run was the lead column in the AP-101 run. During processing of the AP-101 tank waste, the lead column only was rinsed, eluted, and regenerated. The system processed 220 BV of feed (1100 mL at 50  $\mu\text{Ci/L}$  technetium), resulting in a breakthrough of  $\sim 5\%$  on the lead column. The lead column was eluted until the  $C/C_0$  was  $<0.01$ .

Before processing the AN-102/C-104 blended feed, the lead column from the AP-101 column run was switched to the lag-column position. This lag column had not been eluted and exhibited a slight breakthrough (0.2%) while processing AP-101. Both the lead and lag columns were prepared for AN-102/C-104 loading by flushing with 5 BV (25 mL) of 0.25 M NaOH. This was done primarily to assure that the bed was sufficiently settled before starting the column run.

## 2.7 Batch-Distribution Measurement

Some of the AN-102/C-104 blended feed was used to determine technetium batch-distribution coefficients ( $K_d$  values) before running the column. The AN-102/C-104 blended feed was processed to remove cesium before using the material for technetium-batch-distribution measurements. The resin was contacted with AN-102/C-104 blended feed spiked with approximately 6  $\mu\text{Ci}$  of  $^{95\text{m}}\text{TcO}_4^-$ . The  $^{95\text{m}}\text{Tc}$  was used as a tracer for following the removal of pertechnetate ( $\text{TcO}_4^-$ ) from solution. The contribution of technetium from the tracer is very small relative to the amount of technetium already present in the supernate (mole ratio  $^{99}\text{Tc}$  to  $^{95\text{m}}\text{Tc} = 1.1 \times 10^6$ ); therefore, the added tracer is not expected to affect the batch-distribution values. The  $^{95\text{m}}\text{Tc}$  spiked AN-102/C-104 blended feed was subdivided into three aliquots. The concentration of technetium in the AN-102/C-104 blended feed was approximately 70  $\mu\text{Ci/L}$ . The total technetium concentration was increased in two aliquots to approximately 1100 and 3100  $\mu\text{Ci/L}$  using a stock solution of  $^{99}\text{Tc}$  (17 and 54  $\mu\text{L}$  of 808  $\mu\text{Ci/mL}$  stock solution).

The batch contacts were conducted at a phase ratio of approximately 100:1 (liquid volume to resin mass), using resin batch # 010227CTC-9-23. Six milliliters of AN-102/C-104 blended feed were contacted with 0.06 g of exchanger. The exchanger mass was determined to an accuracy of  $\pm 0.0001$  g. The waste volume was transferred by pipette, and the actual volume was determined from the mass difference ( $\pm 0.0002$  g accuracy) and the solution density. An orbital shaker was used to provide agitation for approximately 96 h. The temperature was not controlled, and it varied between approximately 24°C and 25°C over the course of the 4-day contacts. All batch-distribution experiments were conducted in duplicate.

After equilibration, the supernate was syringe filtered using a 0.45- $\mu\text{m}$  nylon filter. The total  $^{99}\text{Tc}$  concentration in the AN-102/C-104 blended feed was determined by inductively coupled plasma-mass spectroscopy (ICP-MS), which in turn was used to calculate the  $^{99}\text{Tc}$   $K_d$ s. The total  $\text{TcO}_4^-$  concentration

in the AN-102/C-104 blended feed was determined by radiochemical separation and beta counting, which was used to calculate the  $\text{TcO}_4^- K_{ds}$ .

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

$$K_d = \frac{(C_0 - C_1) * V}{C_1 * M * F} \quad (2.1)$$

where  $C_0$  and  $C_1$  = initial and final concentrations in solution, respectively, of the species of interest (i.e.,  $^{95m}\text{TcO}_4^-$ ,  $^{99}\text{TcO}_4^-$  and  $^{99}\text{Tc}$ )

$V$  = volume of the liquid sample (mL)

$M$  = mass of resin used for the contact (g)

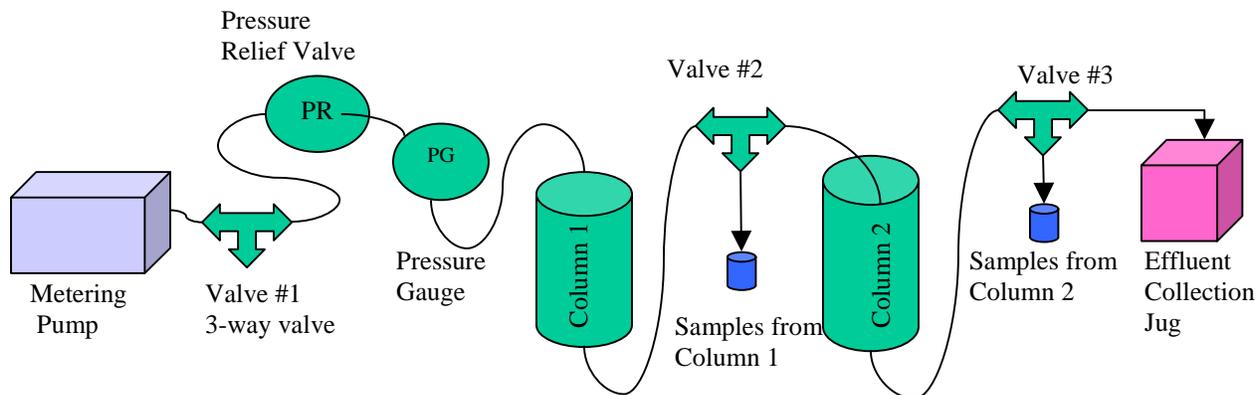
$F$  = mass of a sample of dried resin divided by its mass before drying

The resin was sampled for F-factor determination at the same time that resin samples for the batch contacts were measured to minimize mass changes due to changes in atmospheric humidity.

## 2.8 Tc-Removal Column System

Figure 2.1 shows a schematic of the column system for removing technetium. The system, which is mounted in a radiological fume hood, consists of two small columns containing the sorbent resin, a small metering pump, three 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 Spectra Chrom™ chromatography columns made of glass with adjustable plungers on the bottom and the top. The inside diameter of the columns is 1.5 cm, which corresponds to a volume of 1.77 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. An Accu® piston pump (SciLog, Middleton, Wisconsin) was used to deliver feed to the columns. The flowrate was controlled with a metering pump head (Fluid Metering, Inc., Oyster Bay, New York) and a pump stroke-rate controller (0.025 mL/stroke). The volume actually pumped through the system is determined using the effluent mass (including analytical samples) 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 system-apparatus volume was measured to be 20 mL (4 BV) with approximately 10 mL (2 BV) volume holdup from the feed line to the lead column.



**Figure 2.1.** Schematic Representation of Column System for Removing Technetium

## 2.9 Column Ion Exchange Test Procedure and Conditions

The experimental conditions for each process step of the column ion exchange test are shown in Table 2.3. The bed preparation, loading, and feed-displacement steps were conducted by passing the specified solution through both resin beds connected in series. Only the lead column was rinsed with DI water, eluted, and regenerated, as indicated in column one of the table.

**Table 2.3.** Experimental Conditions of AN-102/C-104 Blended Feed Small-Scale<sup>(a)</sup> Ion Exchange

Column	Process Step	Solution	Total Vol, mL/(BV)	Flowrate, ML/h/(BV/h)	Time, h (min)
Lead and Lag	Column Prep	0.25 <u>M</u> NaOH	15 (3)	15 (3)	1 (60)
Lead and Lag	Loading	AN-102/C-104 Feed	625–680 (132–136)	15 (3)	46 (2740)
Lead and Lag	Feed <sup>(b)</sup> Displacement	0.10 <u>M</u> NaOH	50 (~2 apparatus volumes)	15 (3)	5 (300)
Lead	Rinse	DI water	10 (2)	15 (3)	0.7 (40)
Lead	Elution	DI @ 65°C	132	5 (1)	~27 (1590)
Lead	Regeneration	0.25 <u>M</u> NaOH	~23 (4)	5 (1)	2 (120)

(a) The BV for each column was  $5.0 \pm 0.1$  mL.  
(b) 0.10 M NaOH is used to prevent the feed from precipitating, even though it has been demonstrated to slightly elute technetium from the loaded column (Blanchard et al. 2000a, 2000b; King 1999).

The resin beds were gently packed with approximately 0.7 cm of quartz glass wool to prevent the resin from floating during contact with the AN-102/C-104 blended feed. The adjustable plungers at the top of each column were lowered to be in contact with the glass wool. Both the resin-bed height and quartz glass-wool height were carefully monitored throughout the loading and elution process. There was no observed change in the height of either material, indicating that if the resin BV changed during the run, the change was less than 0.1 mL, or less than 2% of the total BV.

All steps of the column run were performed at ambient temperature, with the exception of the elution, which was performed at 65°C. Ambient temperature during this run ranged from 24 to 26°C. A recirculating water bath was used to circulate heated water through the column jacket during elution.

The sampling-and-analysis protocol is shown in Table 2.4. Samples were collected during loading to provide sample material for monitoring the ion exchange run as well as to provide sample material for the Tc monitoring system. The technetium monitor system requested sample volumes of 2 and 4 mL. To accommodate the Tc monitor-system needs and the column ion exchange needs, samples of either 3 mL or 5 mL were collected during loading.

The total technetium (<sup>99</sup>Tc) levels were determined by ICP-MS for all samples after the run was concluded. Pertechnetate was determined in all loading samples using TcO<sub>4</sub><sup>-</sup> data obtained by performing a chemical separation and beta scintillation count. Inductively coupled plasma-atomic emission spectrometry (ICP-AES) was used to determine sodium and other elemental concentrations for samples from the feed displacement and regeneration steps. Free hydroxide ion concentrations were determined by titration with standard hydrochloric acid solutions for the feed displacement and regeneration samples. Table 2.4 identifies the analyses performed on the samples collected during the ion exchange column run and the final sample composites.

**Table 2.4.** Sampling Interval and Analyses of Small-Scale Ion Exchange Column Process Stages

Process Step	Lead Column BV	Lag Column BV	Collected Volume, mL	Analyses <sup>(a)</sup>
Original Feed	--	--	--	ICP-MS, ICP-AES, OH <sup>-</sup> , IC <sup>(b)</sup> anions TOC <sup>(c)</sup> TIC, <sup>(d)</sup> rad chem., and TcO <sub>4</sub> <sup>-</sup>
<b>Column Preparation</b>				
Loading	Every 10 BV	Every 20 BV	3 or 5	ICP-MS, TcO <sub>4</sub> <sup>-</sup> , Tc monitoring sys.
Feed Displacement	--	Every 1 BV	5	ICP-AES, ICP-MS, OH <sup>-</sup>
DI water rinse	2 BV	--	10	ICP-AES, ICP-MS, OH <sup>-</sup>
Elution	Every 1 BV <sup>(a)</sup>	--	2.5/5 <sup>(a)</sup>	ICP-MS
Regeneration	--	2 BV	10	ICP-AES, ICP-MS, OH <sup>-</sup>
<b>Composite Samples</b>				
Effluent	--	1 composite	20	ICP-AES, TOC, TIC, IC, OH <sup>-</sup> , ICP-MS, U, Radchem <sup>(e)</sup>
Eluant	1 composite	--	20	ICP-AES, TIC, TOC, IC, OH <sup>-</sup> ICP-MS, U, Radchem <sup>(f)</sup>
(a) Initial samples (first 10 BV) were taken in ½ BV increments, resulting in 2.5-mL samples. (b) IC = ion chromatography (c) TOC = total organic carbon (d) TIC = total inorganic carbon (e) Radiochemical analyses were separation/alpha energy analysis (AEA), extended counting gamma energy analysis (GEA), <sup>90</sup> Sr separation, alpha counting, and pertechnetate speciation. (f) Radiochemical analyses were <sup>90</sup> Sr and GEA. -- Indicates data cell is not applicable				

### 3.0 Results and Discussion

This section provides the feed composition and the batch-distribution results for AN-102/C-104 blended feed. It also provides specific detail into each of the ion exchange processing steps and concludes with the Tc mass balance.

#### 3.1 Feed Composition

The composition of the AN-102/C-104 supernate used for the batch contacts and column ion exchange is shown in Table 3.1. Table 3.1 contains all of the analytes observed by each analytical method: ICP-AES, titration, ICP-MS, ion chromatography (IC), TIC/TOC, and GEA. The concentration of pertechnetate and <sup>90</sup>Sr was determined by radiochemical separation.

**Table 3.1.** Composition of AN-102/C-104 Blended Feed

Analyte	Analytical Result	Analytical Method	Analyte	Analytical Result	Analytical Method
Radiochemical Results in $\mu\text{Ci/mL}$					
<sup>99</sup> Tc <sup>(a)</sup>	7.95E-02	ICP-MS	<sup>241</sup> Am	1.04E-02	AEA
TcO <sub>4</sub> <sup>-</sup>	3.99E-02	Sep/Count	<sup>154</sup> Eu	2.20E-02	GEA
<sup>90</sup> Sr	1.52E+00	Sep/Count	<sup>155</sup> Eu	1.23E-02	GEA
Total $\alpha$	1.10E-02	Alpha count	<sup>60</sup> Co	3.41E-02	GEA
<sup>126</sup> Sn/Sb	4.34E-02	GEA	<sup>137</sup> Cs	1.40E-02	GEA
Inorganic Analysis Results in $\mu\text{g/mL}$					
Al	7,840	ICP-AES	Na	110,000	ICP-AES
B	86	ICP-AES	Nd	[3.6]	ICP-AES
Ca	149	ICP-AES	Ni	145	ICP-AES
Cd	24.2	ICP-AES	P	562	ICP-AES
Co	[1.90]	ICP-AES	Pb	69.1	ICP-AES
Cr	98.2	ICP-AES	Si	179	ICP-AES
Fe	[2.0]	ICP-AES	Sr	86.9	ICP-AES
K	1,080	ICP-AES	Zn	[4.0]	ICP-AES
Mo	23.1	ICP-AES	U	8.22E-01	KPA <sup>(b)</sup>
Inorganic Anion Results in $\mu\text{g/mL}$					
Cl	1,800	IC Anions	NO <sub>3</sub>	107,000	IC Anions
F	6,300	IC Anions	PO <sub>4</sub>	2,200	IC Anions
NO <sub>2</sub>	41,400	IC Anions	SO <sub>4</sub>	6,700	IC Anions
Carbon Results in $\mu\text{g C/mL}$					
TIC	9,100	Hot Persulfate	TIC	-- <sup>(c)</sup>	Furnace
TOC	12,300	Hot Persulfate	TOC	21,200	Furnace
Physical Properties Units as Reported, <u>M</u> is moles/L					
Hydroxide	0.18 <u>M</u>	Titration	Density	1.240 g/mL	Gravimetric
[ ] Analyte was detected within 10 times the IDL, and the uncertainty is estimated to exceed $\pm 15\%$ .					
(a) Uncertainty, including standards bias, ranged from -10% to +30%.					
(b) kinetic phosphorescence analysis					
(c) The TIC value could not be determined. TIC=TC-TOC; the TC and TOC values were the same.					

The compositions of selected anions and cations, as well as information important to technetium ion exchange, are collected in Table 3.2. The total anion normality, 5.23 N, is larger than the total cation normality, 4.82 N. This difference is approximately 9 percent and is within analytical and experimental uncertainties.

**Table 3.2.** Selected Composition of AN-102/C-104 Blended Feed (Envelope C Feed)

Analyte	Moles/Liter ( <u>M</u> ) (Normality)
Na <sup>+</sup>	4.78 (4.78)
K <sup>+</sup>	0.028 (0.028)
Ca <sup>+2</sup>	0.004 (0.007)
Total Cation (Normality)	4.82
AlO <sub>2</sub> <sup>- (a)</sup>	0.29 (0.29)
Cl <sup>-</sup>	0.051 (0.051)
CO <sub>3</sub> <sup>-2 (b)</sup>	0.76 (1.52)
CrO <sub>4</sub> <sup>-2 (a)</sup>	0.002 (0.004)
F <sup>-</sup>	0.33 (0.33)
NO <sub>2</sub> <sup>-</sup>	0.90 (0.90)
NO <sub>3</sub> <sup>-</sup>	1.73 (1.73)
OH <sup>-</sup>	0.18 (0.18)
PO <sub>4</sub> <sup>-3</sup>	0.023 (0.069)
SO <sub>4</sub> <sup>-2</sup>	0.07 (0.14)
Oxalate	0.016 (0.016)
Total Anion (Normality)	5.23
<sup>99</sup> Tc and Competing Ion Ratios	
<sup>99</sup> Tc, μCi/L (mg/L) <sup>(c)</sup>	79.5 (4.68)
<sup>99</sup> Tc as pertechnetate, μCi/L (mg/L)	39.9 (2.35)
NO <sub>3</sub> <sup>-</sup> / <sup>99</sup> Tc mole ratio	3.65E+04
CrO <sub>4</sub> <sup>-2</sup> / <sup>99</sup> Tc mole ratio	40
Solution Density, g/mL	1.240
(a) Elemental Al and Cr were determined by ICP-AES. The anionic forms, AlO <sub>2</sub> <sup>-</sup> and CrO <sub>4</sub> <sup>-2</sup> , are assumed on the basis of waste chemistry.	
(b) The carbonate value is based upon the TIC value from Urie, et al. 2002.	
(c) Uncertainty, including standards bias, ranged from -10% to +30%	

### 3.2 Batch Distribution

The equilibrium behavior of the sorbent material was assessed by measuring batch-distribution coefficients on the original feed and the feed spiked at two higher concentrations of technetium. The results of duplicate distribution-coefficient measurements are shown in Table 3.3. The sodium, nitrate, and technetium concentrations and the density reported in the column “Feed Conditions” reflect initial conditions. The nitrate-to-technetium mole ratio reflects the equilibrium concentration of nitrate and the equilibrium concentration of technetium. This ratio is important since nitrate is a known competitor with the SL-639 adsorption sites (Hamm et al. 2000; Rapko et al. 2001). Note that the reported batch-contact

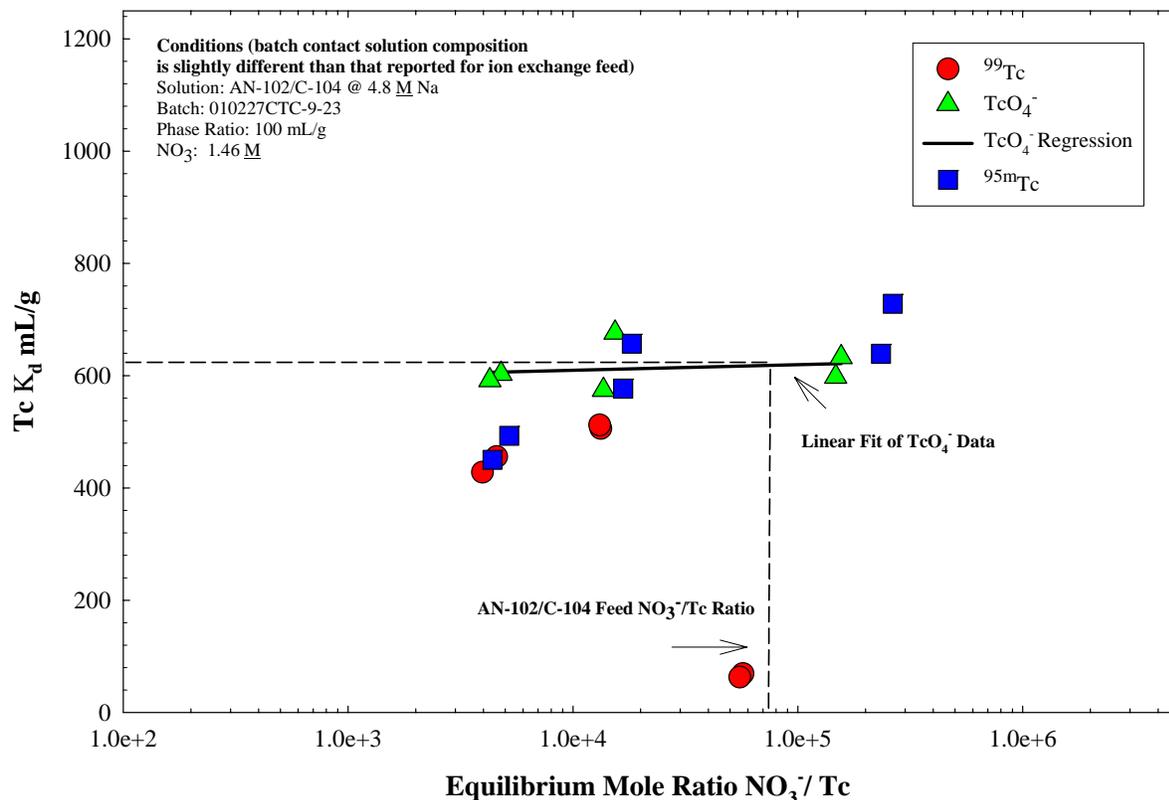
initial  $^{99}\text{Tc}$  value (70  $\mu\text{Ci/L}$ ) is not the same as the initial ion exchange feed  $^{99}\text{Tc}$  value (79  $\mu\text{Ci/L}$ ) as reported elsewhere within this report. The difference is caused by using a separate sample aliquot for the batch contacts. The batch-contact solution underwent evaporation and slight solids precipitation during storage. Based upon the analysis of the solution, water was added to adjust the sodium concentration from 6.3 M to approximately 5.0 M. Note also that the nitrate value for the batch-contact solution is lower than that of the ion exchange feed, possibly due to precipitation of nitrate solids during storage.

The distribution coefficients were determined by assessing the change in both the  $^{99}\text{Tc}$  and  $\text{TcO}_4^-$  in solution and are reported as either  $^{99}\text{Tc}$  or  $\text{TcO}_4^-$   $K_d$  values. The  $^{99}\text{Tc}$  concentration reflects the total technetium-solution concentration, which includes both pertechnetate and non-pertechnetate forms of technetium. The  $\text{TcO}_4^-$   $K_d$  values reflect only the pertechnetate in solution, which is approximately 50% of the total technetium concentration. Note the discussion in the first paragraph about the difference in composition of the batch-contact solution and the ion exchange composition reported in Tables 3.1 and 3.2.

**Table 3.3.** AN-102/C-104 Technetium  $K_d$  Values—Average of Duplicate Measurements

Sample	Feed Conditions	Total Tc $K_d$ , mL/g <sup>(a)</sup> (% Removal)	$\text{TcO}_4^-$ $K_d$ , mL/g <sup>(b)</sup> (% Removal)
AN-102/C-104 Original	Na = 4.8 <u>M</u> $^{99}\text{Tc}$ = 70 $\mu\text{Ci/L}$ <sup>(c)</sup> (2.77 mg/L) $\text{NO}_3^-/\text{Tc}$ , eq = 5.61E+04 $\text{NO}_3^-/\text{TcO}_4^-$ , eq = 1.52E+05 SL-639 batch #010227CTC-9-23	70 (38%)  RPD <sup>(d)</sup> = 9%	660 (85%)  RPD <sup>(d)</sup> = 6%
AN-102/C-104 Spike 1	Na = 4.8 <u>M</u> $^{99}\text{Tc}$ = 1500 $\mu\text{Ci/L}$ <sup>(c)</sup> (88.9 mg/L) $\text{NO}_3^-/\text{Tc}$ , eq = 1.32E+04 $\text{NO}_3^-/\text{TcO}_4^-$ , eq = 1.45E+04 SL-639 batch #010227CTC-9-23	550 (83%)  RPD <sup>(d)</sup> = 1%	670 (86%)  RPD <sup>(d)</sup> = 18%
AN-102/C-104 Spike 2	Na = 4.8 <u>M</u> $^{99}\text{Tc}$ = 4100 $\mu\text{Ci/L}$ <sup>(c)</sup> (241 mg/L) $\text{NO}_3^-/\text{Tc}$ , eq = 4.27E+03 $\text{NO}_3^-/\text{TcO}_4^-$ , eq = 4.53E+03 SL-639 batch #010227CTC-9-23	470 (81%)  RPD <sup>(d)</sup> = 6%	640 (86%)  RPD <sup>(d)</sup> = 2%
(a) The total Tc $K_d$ is assumed to be equal to the $^{99}\text{Tc}$ $K_d$ . (b) The $\text{TcO}_4^-$ $K_d$ is determined from pertechnetate speciation data. (c) Uncertainty, including standards bias, ranged from -10% to +30% (d) The RPD is the relative percent difference and is determined by taking (Initial $K_d$ - Duplicate $K_d$ )/Initial $K_d$ times 100.			

The equilibrium mole ratio of nitrate to technetium was determined using the equilibrium concentration of nitrate and the equilibrium concentration of either total technetium or pertechnetate. Both values are shown in Table 3.3. The  $^{99}\text{Tc}$   $K_d$  values are assumed to be equivalent to the total Tc  $K_d$ s. The percent differences between the duplicate measurements are indicated along with the average  $K_d$  values in the table. A plot of the  $K_d$ s versus the equilibrium mole ratio of nitrate to technetium is shown in Figure 3.1. Both the  $^{99}\text{Tc}$  and  $\text{TcO}_4^-$  derived  $K_d$  values, as well as the  $^{95\text{m}}\text{TcO}_4^-$  values, are shown in the figure.



**Figure 3.1.** <sup>99</sup>Tc and TcO<sub>4</sub><sup>-</sup> Derived K<sub>d</sub> Values

As can be seen in Figure 3.1, the <sup>99</sup>Tc K<sub>d</sub> values for the initial solution batch contact (largest ratio of NO<sub>3</sub>/<sup>99</sup>Tc) are significantly lower than the other K<sub>d</sub> values. This is due to the high fraction of non-perpertechnetate present in this solution. The other samples were spiked with pertechnetate to increase the total technetium concentration, thus the fraction of non-perpertechnetate is quite small in those samples. The figure also shows that the <sup>95m</sup>TcO<sub>4</sub><sup>-</sup> derived K<sub>d</sub> values correlate well with the TcO<sub>4</sub><sup>-</sup> K<sub>d</sub> values. Since the SL-639 only removed pertechnetate from solution, a linear fit of the pertechnetate data was used to interpolate the TcO<sub>4</sub><sup>-</sup> K<sub>d</sub> value at the initial feed conditions (mole NO<sub>3</sub>/<sup>99</sup>TcO<sub>4</sub><sup>-</sup>). The resulting TcO<sub>4</sub><sup>-</sup> K<sub>d</sub> value is ~620 mL/g, which results in an estimated λ value (K<sub>d</sub> × resin density) of ~310 BVs.

### 3.3 AN-102/C-104 Column Test – Sample Loading

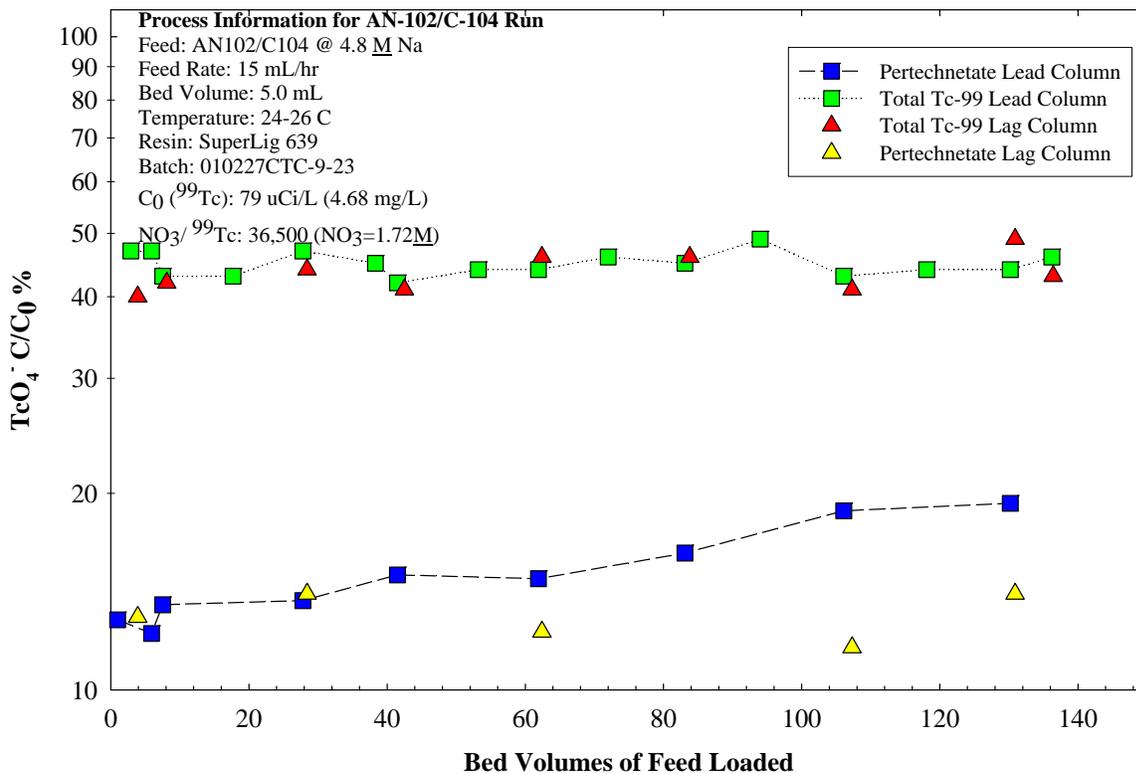
The ion exchange system was shaken down using an AW-101 simulant and then used to remove technetium from AP-101 (Burgeson et al. 2002a) as described in Section 2.6. Only the lead column was eluted during the AP-101 column run. After elution of the lead column, the lead column was regenerated using 0.25 M NaOH. The lead and lag columns were then switched; thus, the lead column in the AN-102/C-104 run was the non-eluted lag column from the AP-101 run.

Immediately before starting to load the AN-102/C-104 blended feed, the system was flushed again with 0.25 M NaOH (10 mL at 15 mL/h). The AN-102/C-104 feed was processed through the system beginning August 6, 2001. The loading stage lasted for 46 h, in which time 136 BVs (680 mL) of

AN-102/C-104 feed were processed. The flowrate was determined at each sampling event and was approximately 15 mL/h (3.0 BV/h) throughout the run. There were no observed anomalies, e.g., bed fluidization, gas bubbles, changes in resin bed height or color, etc. The pressure remained below 5 psi during the entire loading cycle.

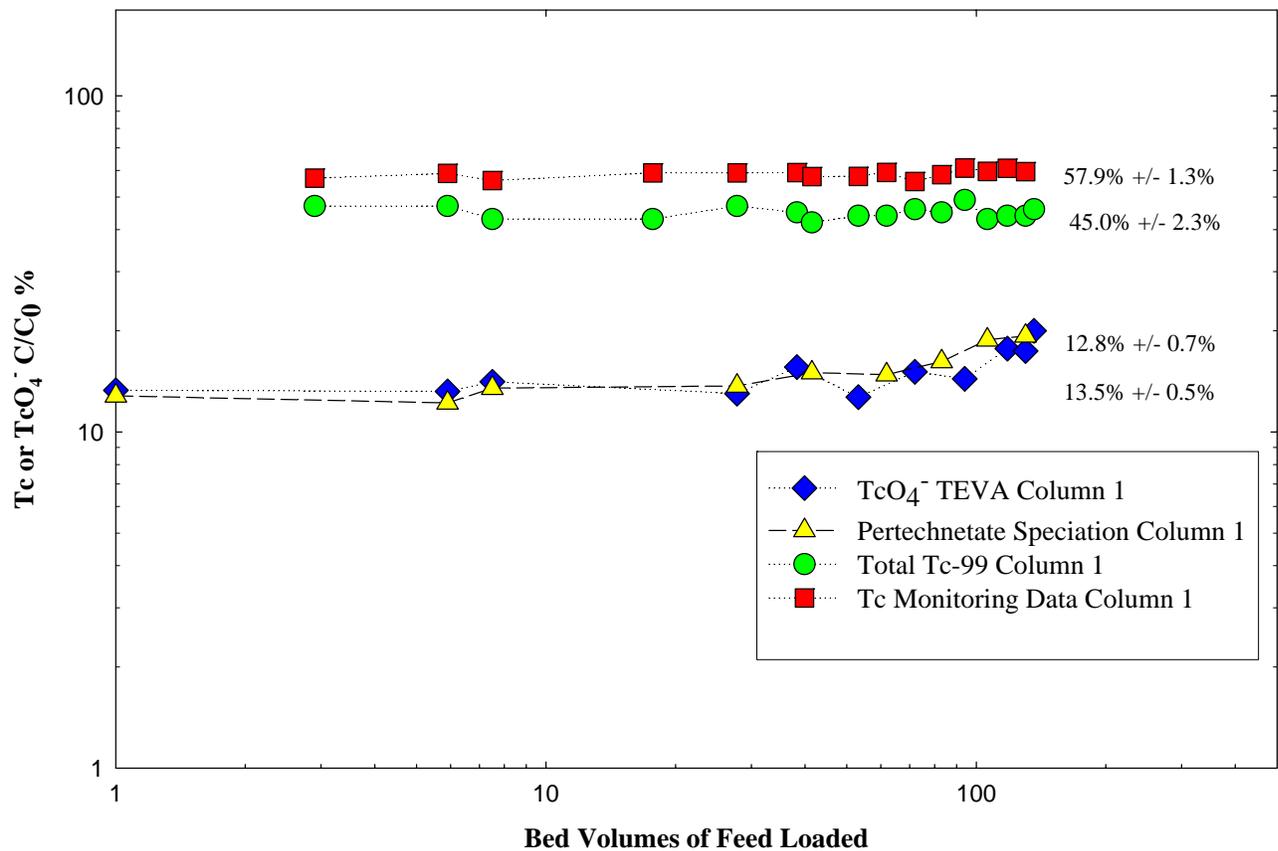
The feed was first processed through the column system until 1½ apparatus volumes had passed through the system. At this point, the first sample collected was considered to be 1 BV. Analytical samples were collected from the lead column at 1, 3, 5, and 7 BVs and then every 10<sup>th</sup> BV thereafter. The lag column was not sampled as frequently as the lead column; samples were collected after the 2<sup>nd</sup> and 5<sup>th</sup> BVs of processed feed, and then every 20 BVs thereafter.

The column loading was monitored in selected loading samples using a TEVA resin isolation of pertechnetate, which was then beta counted since the AN-102/C-104 blended feed was not spiked with a radiotracer. Using the counting data for these selected samples, the ratio of the measured  $\text{TcO}_4^-$  passing through the lead and lag column with respect to the initial amount of  $^{99}\text{Tc}$  in the ion exchange feed ( $C/C_0$ ) was calculated. These values are shown in Figure 3.2 for each sample. In addition, each loading sample was analyzed for total technetium ( $^{99}\text{Tc}$ ). The ratio of the initial  $^{99}\text{Tc}$  and the  $^{99}\text{Tc}$  measured in the loading samples is also shown in Figure 3.2. The  $C_0$  value for  $^{99}\text{Tc}$  was 79.5  $\mu\text{Ci/L}$  (4.68 mg/mL). The initial  $\text{TcO}_4^- C_0$  value was 39.9  $\mu\text{Ci/L}$  (2.35 mg/mL). The  $C/C_0$  % values are plotted on a probability scale.



**Figure 3.2.** AN-102/C-104 Technetium Loading Breakthrough Curves, Lead and Lag Columns

The technetium breakthrough, after processing approximately 680 mL of AN-102/C-104 blended feed, was 46% on the lead column and 43% on the lag column, based upon the  $^{99}\text{Tc}$  concentration values. The  $\text{TcO}_4^-$  breakthrough, measured by speciation, was 19% on the lead column and 14% on the lag column. There was significant scatter in the  $\text{TcO}_4^-$  speciation results; this reflects the uncertainty in the analytical method. As can be seen Figure 3.2, the analytical data results indicate that pertechnetate immediately broke through on both columns at a level of ~13%. The overall system change in pertechnetate breakthrough is approximately 6% during the course of the run. A separate radiochemical separation of  $^{99}\text{TcO}_4^-$ , column separation followed by solvent extraction and beta scintillation counting, was performed by the analytical laboratory. The analytical laboratory results are in excellent agreement with the values obtained while monitoring the column run, using TEVA resin separation and beta scintillation counting. The two pertechnetate separation procedures were quite different; however, both methods used beta cocktail scintillation counting to quantitate pertechnetate. The data from these two methods were also compared with the values obtained from the technetium monitoring system with the ICP-MS analyses since the technetium monitoring system oxidized all of the technetium to the  $\text{Tc}^{+7}$  state before quantitation. The lead column loading data from all four analytical methods are presented in Figure 3.3.



**Figure 3.3.** Pertechnetate and Total Technetium Loading Curves for Lead and Lag Columns Compared with the Technetium Monitoring and ICP-MS Results

The four curves in Figure 3.3 are from four different analytical methods: 1) TEVA resin separation of pertechnetate and quantitation by beta counting, 2) radiochemical separation and extraction of pertechnetate and quantitation by beta counting, 3) Tc monitoring system, which oxidizes pertechnetate to technetium and quantitates total technetium with beta counting, and 4) ICP-MS measurement of total technetium. The average percent breakthrough of the initial three data points for each analytical method is shown beside each plot. Two points about the data become readily apparent. One, there is very little change in technetium breakthrough from the beginning of the column run to the end of the column run. There is a definite upward trend in the pertechnetate breakthrough value for the lead column, approximately a 6% increase during the course of the run (from ~13% to ~19%). Returning to Figure 3.2, there is not a corresponding increase in the total technetium values of the lag column, indicating that the breakthrough of the lead column is contained by the lag column. Second, the difference between the ICP-MS total determination of technetium and the Tc monitoring system determination of total technetium is ~15%, approximately the value reported for pertechnetate using either speciation method. This strongly indicates that there is an interferent in the beta counting of pertechnetate and total technetium. However, the presence of an interferent is circumstantial; thorough evaluation of the counting data did not indicate the presence of either a beta or gamma interferent. Note that it was verified that there was no  $^{95m}\text{Tc}$  present in the loading samples. Thus, it is not apparent what is causing the elevated pertechnetate signal. The difference between the Tc monitor and ICP-MS results was attributed to holdup of  $^{121m}\text{Sn}$  on the AGMP-1 resin used for Tc separation in the Tc monitor<sup>a</sup>. The separation procedure was modified to eliminate this problem, and agreement between the monitor and ICP-MS in subsequent analysis of AN-102/ C-104 samples was excellent. The initial AN-102/ C-104 samples were consumed in analysis, so could not be re-analyzed.

The initial  $^{99}\text{Tc}$  DFs for the first and second columns (derived from the  $^{99}\text{Tc}$  concentration in the first sample from each column) are 1.9 and 2.2, respectively. The corresponding initial DFs for pertechnetate (similarly derived from the  $\text{TcO}_4^-$   $C/C_0$  in the first sample from each column) were 11 for both the lead and lag columns. The concentration of  $\text{TcO}_4^-$  in the effluent composite was 5.45  $\mu\text{Ci/L}$ , and the initial concentration of  $\text{TcO}_4^-$  was 39.9  $\mu\text{Ci/L}$ . The effluent composite concentration demonstrates a pertechnetate removal of 86%, or an overall  $\text{TcO}_4^-$  DF of 7.3. The concentration of  $^{99}\text{Tc}$  in the effluent composite was 44.1  $\mu\text{Ci/L}$ , and the concentration of  $^{99}\text{Tc}$  in the initial feed was 79.5  $\mu\text{Ci/L}$ . The effluent composite concentration demonstrates a total technetium removal of 44%, or an overall DF of 1.8.

The  $\lambda$  value is the number of BVs of processed feed at which the  $C/C_0$  value reaches 50% and is a direct indicator of the effective capacity of the resin. Since there was only ~19% breakthrough of technetium on the lead column, the  $\lambda$  value could not be experimentally determined. An estimate of the  $\lambda$  value can be obtained from extrapolating the loading-curve data plotted on a linear scale, assuming kinetics are not a significant contributor. This extrapolation provides a rough estimate of 500 BVs of feed that could be processed before reaching 50%  $C/C_0$  breakthrough.

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<sup>a</sup> Egorov OB, and MJ O'Hara. 2002. *Development and Testing of the Automated  $^{99}\text{Tc}$  Monitor: October 2001 – March 2002 Progress Report*. WTP-RPT-042, Battelle—Pacific Northwest Division, Richland, WA.

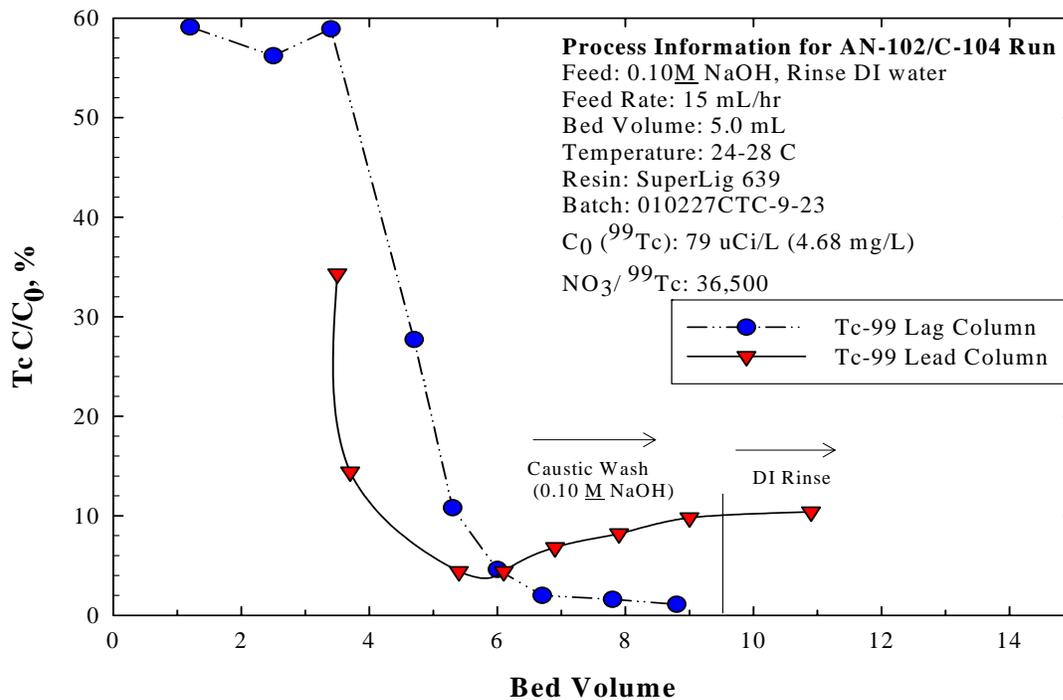
### 3.4 AN-102/C-104 Column Test – Feed Displacement and Column Rinse

Feed displacement was started immediately after the last load sample was collected. The beds were flushed in series with 0.10 M NaOH at 3.0 BV/h to displace the feed before elution.

The feed displacement was monitored using beta scintillation counting of the collected samples. The ratio of the measured  $^{99}\text{Tc}$  passing through the lead and lag column with respect to the initial amount of  $^{99}\text{Tc}$  in the ion exchange feed ( $C/C_0$ ) was calculated during displacement. Samples were collected from the lag column during feed displacement with occasional samples collected from the lead column to determine when the feed-displacement criteria,  $C/C_0 < 1\%$ , had been met. Based upon the beta monitoring of  $^{99}\text{Tc}$ , the feed displacement progressed for 9 BVs. After the feed displacement was stopped, enough of DI water (10 mL) was passed through the system to rinse the lead column.

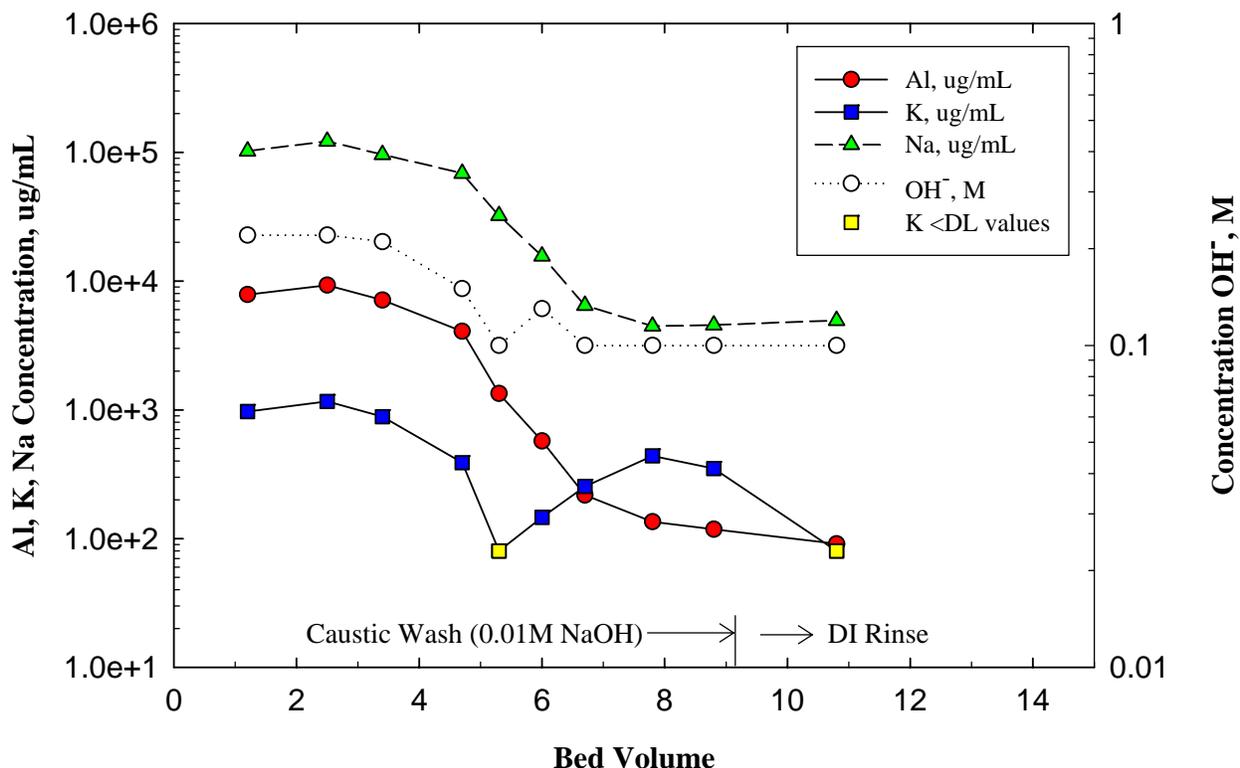
Inductively coupled plasma-mass spectrometry analysis of the solutions after completion of the run indicated that the lead column had begun to elute technetium from the column after 6 BVs. The  $^{99}\text{Tc}$  concentration from the lag column decreased steadily to  $\sim 1\%$   $C/C_0$  during the 9 BV of feed displacement. The lead column decreased to a low of  $\sim 4\%$  at 5 and 6 BV and then began to increase. The increase (elution) is caused by a decrease in the ionic strength of the solution in contact with the SL-639 resin. The elution of  $\text{TcO}_4^-$  with 0.10 M NaOH has been observed in previous column waste testing (Blanchard et al. 2000a, 2000b; Burgeson et al. 2002a).

The ratio of the ICP-MS measured  $^{99}\text{Tc}$  passing through the lead and lag column with respect to the initial amount of  $^{99}\text{Tc}$  in the ion exchange feed ( $C/C_0$ ) was calculated and is reported in Figure 3.4 for each sample for the feed displacement and water rinse.



**Figure 3.4.**  $^{99}\text{Tc}$   $C/C_0$  for Feed Displacement of Lead and Lag Columns

The feed-displacement solutions were analyzed to determine the concentration of Al, K, Na, and OH<sup>-</sup> present in each solution. The concentrations of sodium, potassium, aluminum, and hydroxide in the feed-displacement samples are shown in Figure 3.5. The concentrations of Na, K, and Al are indicated on the left hand axis while the OH<sup>-</sup> concentration is shown on the right-hand axis. Both Y-axes are logarithmic scales in order to clearly show the large decreases in the analyte concentrations. The samples were collected from the lag column after the solution had passed sequentially through both columns.



**Figure 3.5.** Component Concentrations in Lag Column Feed-Displacement Samples

The concentrations of the four feed components changed slightly during the first four BVs of processing. For all of the analytes, the second data solution reports a higher concentration than that observed for the first solution. This is likely due to sample preparation before analysis. The concentration of each analyte dropped significantly (at least 50%) after 3 BVs and continued through the feed-displacement process. The displacement of the feed is reasonably sharp, except for potassium. The potassium data are close to the method detection limit. In fact, two of the values shown in the figure for potassium were below detection and are shown as a different symbol to highlight this fact. The Na and OH<sup>-</sup> concentrations do not drop as low as the other analytes because they are present in the displacement solution. Table 3.4 reports the percent reduction of the analyte concentrations during feed displacement.

The 0.10 M NaOH feed displacement was followed by a room-temperature DI water rinse of only the lead column. The column was rinsed immediately after the feed displacement was completed. The DI water-rinse used approximately 2 BVs of water flowing at a rate of 3 BV/h. The rinse solution was collected as one sample. The concentrations of sodium, potassium, aluminum, and hydroxide were measured in the rinse sample and are also shown in Figure 3.5 and Table 3.4. The elution of technetium, started during feed displacement, continued during the water wash. The  $\text{TcO}_4^-$  was measured in the rinse sample at a  $C/C_0$  value of 10%.

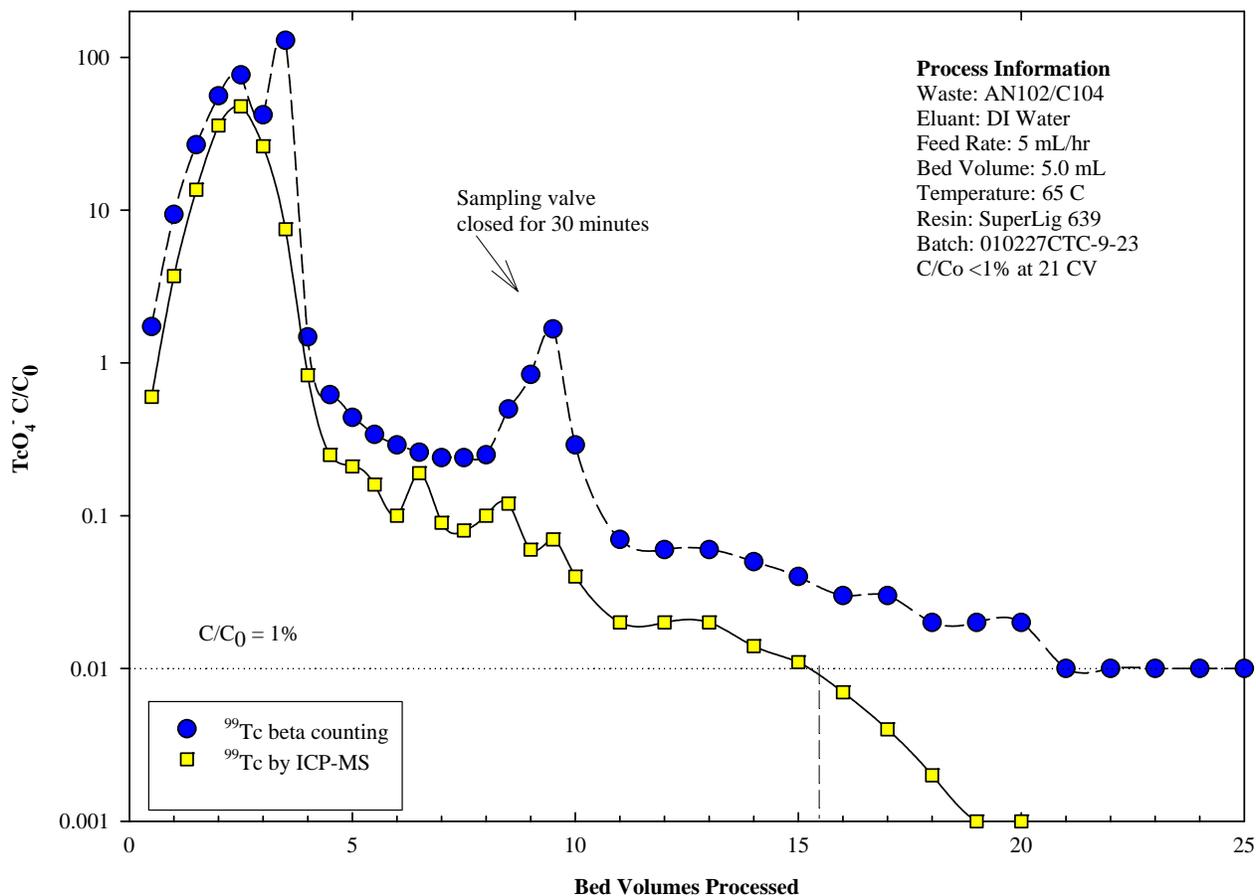
**Table 3.4.** Percent Displacement<sup>(a)</sup> of Feed Analytes from Resin Beds

Sample	Bed Volumes	Al, % displaced	K, % displaced	Na, % displaced	OH, % displaced
FD-1 Lag Column	1.2	-0.2	10.4	7.0	-4.9
FD-2 Lag Column	2.5	-18.3	-7.4	-10.5	-5.9
FD-3 Lag Column	3.4	9.4	18.2	12.7	-0.5
FD-6 Lag Column	4.7	48.2	64.0	37.5	30.5
FD-7 Lag Column	5.3	82.9	< 96	70.7	53.9
FD-9 Lag Column	6.0	92.7	86.5	85.8	39.7
FD-11 Lag Column	6.7	97.2	76.5	94.1	50.5
FD-13 Lag Column	7.8	98.3	59.4	95.9	50.8
FD-15 Lag Column	8.8	98.5	67.6	95.9	51.7
Rinse Lag Column	10.9	98.8	< 92	95.5	51.0
FD-4 Lead Column	3.5	61.5	68.4	50.4	100.0
FD-5 Lead Column	3.7	87.3	84.3	79.8	59.2
FD-8 Lead Column	5.4	97.6	76.6	95.1	41.7
FD-10 Lead Column	6.1	98.3	78.2	95.0	51.7
FD-12 Lead Column	6.9	98.2	84.3	94.2	49.9
FD-14 Lead Column	7.9	98.9	92.9	95.9	49.6
FD-16 Lead Column	9.0	98.8	93.7	95.4	52.9

(a) The percent displaced is determined from the difference of the original AN-102/C-104 feed composition and the analyzed feed-displacement samples.

### 3.5 AN-102/C-104 Column Test—Elution and Resin Regeneration

At the completion of the DI water rinse, the lead column was immediately eluted with 65°C DI water at a rate of 1 BV/h. The column was equilibrated for approximately 15 min before elution was started by running pre-equilibrated 65°C water through the column jacket. The eluate was initially collected in ½-BV increments, which was increased to 1-BV increments after 10 BV. The elution was monitored using beta-scintillation counting of the elution samples. After completion of the run, ICP-MS was used to determine the  $^{99}\text{Tc}$  for each of the elution samples. A plot of the  $^{99}\text{Tc}$   $C/C_0$  for both beta counting data and ICP-MS data is presented in Figure 3.6. The Y-axis is a logarithmic scale to clearly show the large range of  $C/C_0$  values.



**Figure 3.6.** Elution of Technetium from Lead Column

The concentrations of  $^{99}\text{Tc}$  by ICP-MS are significantly lower than those measured during beta scintillation counting of the elution samples. A beta interferent is clearly elevating the technetium signal. The culprit is the gamma emission from  $^{95\text{m}}\text{Tc}$ , which is observed in the elution samples. Analytical characterization of the elution composite indicates that  $1.83\text{E-}03 \mu\text{Ci/mL}$  of  $^{95\text{m}}\text{Tc}$  is present in the eluate composite. This concentration correlates well to the amount of technetium calculated to have been retained on the lag column during the AP-101 ion exchange run. Thus, the source of the  $^{95\text{m}}\text{Tc}$  is the non-eluted lag column created during processing of the AP-101 tank waste.

The elution of technetium peaked at approximately 2 BVs with a value approximately 48 times higher than the initial feed concentration. There was a slight fluctuation in the elution data during the peak elution stage of 9 to 11 BVs, which was due to an inadvertent closing of the sampling valve. The target elution value,  $C/C_0 \leq 1\%$ , was reached after processing 16 BVs of eluent. The dotted line in Figure 3.6 identifies the target elution value of 1% of the original feed value ( $C/C_0 = 1\%$ ). The vertical dashed line indicates the BV where this target elution was reached.

To assure that the  $C/C_0$  value had reached the target  $C/C_0 \leq 1\%$  value, a total of 25 BVs of eluent was passed through the lead column. The system was stored in the water eluent for 7 days before regenerating the lead column with 0.25 M NaOH. The delay was to allow time to resolve questions with the initial elution data. Ten milliliters of NaOH were passed through the lead column at a flowrate of 1 BV/h. The regenerate was analyzed for various analytes; the results of these analyses are shown in Table 3.5.

**Table 3.5.** Composition of Column Regenerate after AN-102/C-104 Column Run

Analyte	Concentration, $\mu\text{g/mL}$	Concentration, Molarity
Na	4180	1.82E-01
K	< 48	< 1.23E-03
Al	7.5	2.78E-04
OH <sup>-</sup>	1870	2.10E-01
<sup>99</sup> Tc	< 0.0001 (< 4.27E-05 $\mu\text{Ci/mL}$ )	< 2.54E-08

There was no measurable <sup>99</sup>Tc present in the regeneration sample. The measured concentration of sodium and hydroxide are right in line with the expected concentration of sodium and hydroxide, based upon a system holdup volume of approximately 20 mL. Although the target volume for regeneration was 20 mL, only half of that volume was processed during regeneration.

### 3.6 AN-102/C-104 Column Test—Elution Composite and Effluent Composite Analysis

The eluate samples were combined to produce a 132-mL composite sample, and an aliquot was submitted for characterization. The results are shown in Table 3.6. The required minimum reportable quantity (MRQ) levels for all of the requested analytes were met. Table 3.6 contains all of the analytes observed by each analytical method: titration, ICP-AES, ICP-MS, IC, AEA, <sup>90</sup>Sr, and GEA. Any observed, non-requested analytes are reported opportunistically.

The main chemical components of the eluate composite were sodium nitrate and nitrite. Although silicon and boron were both observed in the eluate composite, they are most likely due to leaching of the borosilicate glass vials used for sample storage. Aluminum, chloride, fluoride, and sulfate were observed in very low quantities. Nitrate and nitrite were observed at approximately 0.1% of the initial feed, indicating that there may have been some retention and subsequent elution of nitrate and nitrite by the resin. The TIC and TOC values were 110  $\mu\text{g C/mL}$  and 140  $\mu\text{g C/mL}$ , respectively. These values are approximately 1.1% of the initial feed composition. The amount of retained TIC/TOC is higher than that of the anions; however, the ratio of TIC to TOC is consistent with that of the original feed. Thus, there is no evidence of selective increase in either TIC or TOC, e.g., from decomposition of the resin. The major radionuclides observed in solution were <sup>99</sup>Tc and <sup>95m</sup>Tc. A <sup>95m</sup>TcO<sub>4</sub><sup>-</sup> tracer was not used to monitor the AN-102/C-104 testing; the <sup>95m</sup>Tc comes from the non-eluted lag column from the AP-101 column run. A slight amount of <sup>90</sup>Sr, <sup>137</sup>Cs, and total alpha was also present in the eluate composite, confirming that a slight amount of feed was potentially present in a sampling valve or held up in the system after feed displacement and rinsing.

**Table 3.6.** Analysis Results of AN-102/C-104 Eluant Composite

Analyte	Analytical Result	Analytical Method	Analyte	Analytical Result	Analytical Method
Radiochemical Results in $\mu\text{Ci/mL}$					
$^{90}\text{Sr}$	1.08E-03	Sep/ Counting	$^{155}\text{Eu}$	< 3.00E-05	GEA
$^{99}\text{Tc}$ <sup>(a)</sup>	1.80E-01	ICP-MS	$^{137}\text{Cs}$	1.55E-05	GEA
$^{241}\text{Am}$	<5.0E-05	AEA	$^{95}\text{Tc}$	9.24E-05	GEA
$^{154}\text{Eu}$	5.00E-05	GEA	$^{95\text{m}}\text{Tc}$	1.83E-03	GEA
Inorganic Analysis Results in $\mu\text{g/mL}$					
Al <sup>(b)</sup>	9	ICP-AES	La	< 1.2	ICP-AES
K <sup>(b)</sup>	< 50	ICP-AES	Mg	< 2.5	ICP-AES
Na <sup>(b)</sup>	280	ICP-AES	Mn	< 1.2	ICP-AES
B	20	ICP-AES	Mo	< 1.2	ICP-AES
Ba	< 2.5	ICP-AES	Ni	< 0.7	ICP-AES
Ca	< 3.2	ICP-AES	Pb	< 2.5	ICP-AES
Cd	< 6.7	ICP-AES	Si	26	ICP-AES
Co	< 1.2	ICP-AES	Sn	< 37	ICP-AES
Cr	< 0.5	ICP-AES	Ti	< 0.6	ICP-AES
Cu	< 0.6	ICP-AES	Zn	< 1.2	ICP-AES
Fe	< 0.6	ICP-AES	U	1.79E-03	U by KPA
Inorganic Anion Results in $\mu\text{g/mL}$					
Cl	3.0	IC Anions	$\text{NO}_3$	130	IC Anions
F	11	IC Anions	$\text{SO}_4$	9	IC Anions
$\text{NO}_2$	50	IC Anions	$\text{PO}_4$	< 0.25	IC Anions
Carbon Results in $\mu\text{g C/mL}$					
TOC	140	Hot Persulfate	TOC	< 100	Furnace
TIC	110	Hot Persulfate	TIC	-- <sup>(c)</sup>	Furnace
<p>(a) Uncertainty, including standards bias, ranged from -10% to +30%</p> <p>(b) These are the ICP-AES analytes of interest. All other analytes are reported opportunistically.</p> <p>(c) The TIC value could not be determined. TIC=TC-TOC; the TC and TOC values were the same within error.</p> <p>NOTE: 132 mL of composited eluate material was collected.</p>					

The effluent analytical samples were combined with the collected effluent, the feed displacement, and rinse solutions to produce a 682-mL composite sample, and an aliquot was submitted for characterization. The results are shown in Tables 3.7 and 3.8. The required MRQ levels for all of the requested analytes were met. Tables 3.7 and 3.8 contain all of the analytes observed by each analytical method: titration, ICP-AES, ICP-MS, IC, AEA, and GEA. Any observed, non-requested analytes are reported opportunistically. Boron (0.01 M) and silicon (0.01 M) are two such analytes; however, they were most likely leached from the glass sample vial and are not actually present in the effluent composite. The concentration of  $^{90}\text{Sr}$  and technetium present as pertechnetate were determined by radiochemical separation. The total anion normality, 4.78 N, is equal to the total cation normality, 4.78 N.

**Table 3.7.** Inorganic and Carbon Composition of Tc and Cs IX Effluent Composite

Analyte	Analytical Result	Analytical Method	Analyte	Analytical Result	Analytical Method
Inorganic Analysis Results in µg/mL					
Al	7,890	ICP-AES	Mo	22.5	ICP-AES
B	37.7	ICP-AES	Na	109,000	ICP-AES
Ba	< 0.25	ICP-AES	Ni	141	ICP-AES
Be	< 0.25	ICP-AES	P	618	ICP-AES
Ca	146	ICP-AES	Pb	67.5	ICP-AES
Cd	23.4	ICP-AES	Rb	-- <sup>(a)</sup>	ICP-AES
Co	[1.9]	ICP-AES	S	-- <sup>(a)</sup>	ICP-AES
Cr	96.4	ICP-AES	Si	[61]	ICP-AES
Cs	< 2.4E-03 <sup>(a)</sup>	ICP-MS	Sn	< 37	ICP-AES
Fe	[2]	ICP-AES	Sr	85.3	ICP-AES
K	1,060	ICP-AES	Zn	[4.2]	ICP-AES
La	< 1.3	ICP-AES	Zr	< 1.2	ICP-AES
Mg	< 2.5	ICP-AES	U	0.69	U by KPA <sup>(c)</sup>
Inorganic Anion Results in µg/mL					
Cl	1,650	IC Anions	NO <sub>3</sub>	98,100	IC Anions
F	5,800	IC Anions	PO <sub>4</sub>	1,750	IC Anions
NO <sub>2</sub>	38,100	IC Anions	SO <sub>4</sub>	5,900	IC Anions
Carbon Results in µg C/mL					
TIC	7,850 µg C/mL	Persulfate	TIC	-- <sup>(b)</sup>	Furnace
TOC	11,300 µg C/mL	Persulfate	TOC	18,800	Furnace
Physical Properties Units as Reported, <u>M</u> is moles/L					
Hydroxide	0.21 <u>M</u>	Titration	Density	1.23 g/mL	Gravimetric
[ ] Analyte was detected within 10 times the instrument detection limit and the uncertainty is estimated to exceed ± 15%.					
(a) Cs, Rb, and S could not be measured on the ICP-AES system. The reported Cs value is <sup>133</sup> Cs by ICP-MS.					
(b) The TIC value could not be determined; TIC=TC-TOC; the TC and TOC values were the same within error.					
(c) Kinetic phosphorescence analysis					

The two analytical methods for TIC and TOC produce a significant difference in the TIC/TOC values. The hot-persulfate wet-oxidation method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92 to 95°C for TOC, all on the same sample. The furnace method that determines the TOC is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 750°C for 30 min. The total carbon (TC) is determined on another aliquot of the sample by combusting at 1000°C for 30 min, and the TIC is obtained by the difference between the TC and TOC. The characterization of AN-102/C-104 (Urie et al. 2002b) also observes a different result between the two methods; however, the TC determined from either method was essentially the same. That is also the case for these analyses. In his report, Urie evaluates the two methods and identifies the hot-persulfate-based values as the best representation of the TIC and TOC content. Thus, the hot-persulfate results should be considered the more correct value.

**Table 3.8.** Radiochemical Composition of Cs and Tc Ion Exchange Effluent Composite

Analyte	Analytical Result	Analytical Method	Analyte	Analytical Result	Analytical Method
Radiochemical Results in $\mu\text{Ci/mL}$					
$^{99}\text{Tc}$ <sup>(a)</sup>	4.41E-02	ICP-MS	$\text{TcO}_4^-$	5.45E-03	Sep/ Counting
$^{237}\text{Np}$	9.33E-02	ICP-MS	$^{239}\text{Pu}$	1.64E-02	ICP-MS
$^{240}\text{Pu}$	9.30E-04	ICP-MS	$^{241}\text{Pu/Am}$	1.22E-03	ICP-MS
$^{90}\text{Sr}$	1.59E+00	Sep/Counting	$^{239/240}\text{Pu}$	1.74E-05	Sep/AEA
$^{60}\text{Co}$	3.16E-02	GEA	$^{154}\text{Eu}$	2.10E-02	GEA
$^{137}\text{Cs}$	1.38E-02	GEA	$^{155}\text{Eu}$	1.21E-02	GEA
$^{241}\text{Am}$	1.05E-02	AEA	Total $\alpha$	1.17E-02	alpha counting
<sup>(a)</sup> Uncertainty, including standards bias, ranged from -10% to +30%					

Several analytes are reported for the effluent composite (Table 3.8) that were not measured for the initial technetium ion exchange feed (Table 3.1). These analytes,  $^{237}\text{Np}$ ,  $^{239}\text{Pu}$ ,  $^{240}\text{Pu}$ ,  $^{239/240}\text{Pu}$  and  $^{241}\text{Pu/Am}$ , contribute to the total alpha activity and are reported to provide details on the alpha content.

### 3.7 Comparison of AN-102/C-104 Initial Feed and Effluent Composite Composition

The analysis results of the effluent composite can be compared with the initial feed-analysis results to determine the effect, if any, on the analytes in solution upon passing through the resin bed. Table 3.9 compares the initial feed characterization with the final effluent composition. Note that the effluent composite contains the column effluent, feed-displacement solutions (28 g), and column wash solution (9.2 g). The addition of the feed displacement and rinse solutions resulted in a dilution to the effluent of approximately 5%. The percent change in concentration from the initial feed composition is also shown in the table. A negative percent change indicates that the effluent composition was lower than the initial feed composition. Changes of less than 15% are likely insignificant since a combination of the typical analytical uncertainty ( $\pm 10\%$ ) and the dilution (5%) could result in an observed difference of 5 to 15%.

Comparing the initial feed and effluent composite analyses indicates that the only significant concentration changes occurred in the  $^{99}\text{Tc}$  and pertechnetate values. The silicon results are most likely artifacts of leaching from the glass vials where the samples were stored. The phosphate value is suspect, since the phosphorus values determined by ICP-MS show an opposite trend—the values increase by 10% (560 in the initial feed and 620 in the effluent). The hydroxide difference is within the observed analytical uncertainty. The hydroxide value for the initial feed to the Cs ion exchange system was 0.20 M, which is the same as measured in the technetium ion exchange effluent composite.

**Table 3.9.** Comparison of Initial Feed and Effluent Composite Composition

Analyte	Initial Feed $\mu\text{Ci/mL}$	Effluent Composite $\mu\text{Ci/mL}$	Percent Change
$^{99}\text{Tc}$ <sup>(a)</sup>	7.95E-02	4.41E-02	-56
$\text{TcO}_4^-$	3.99E-02	5.45E-03	-86
$^{137}\text{Cs}$	1.40E-02	1.38E-02	-2
$^{90}\text{Sr}$	1.52E+00	1.59E+00	5
$^{60}\text{Co}$	3.41E-02	3.16E-02	-4
$^{241}\text{Am}$	1.04E-02	1.05E-02	1
	<b><math>\mu\text{g/mL}</math>, except where noted</b>	<b><math>\mu\text{g/mL}</math> except where noted</b>	
Al <sup>(b)</sup>	7,840	7,890	1
K <sup>(b)</sup>	1,080	1,060	-2
Na <sup>(b)</sup>	110,000	109,000	-1
Ba	< DL	< DL	--
Ca	149	146	-2
Cd	24.2	23.4	-3
Co	[1.9]	[1.9]	0
Cr	98.2	96.4	-2
Cu	< DL	< DL	--
Fe	[2.0]	[2.0]	0
La	< DL	< DL	--
Mg	< DL	< DL	--
Mn	< DL	< DL	--
Mo	23.1	22.5	-3
Ni	145	141	-3
P	562	618	10
Pb	69.1	67.5	-2
Si	179	[61]	-66 <sup>(c)</sup>
Sn	< DL	< 37	--
Sr	86.9	85.3	-2
Zn	[4.0]	[4.2]	5
U	0.822	0.69	-16
Hydroxide	0.18 <u>M</u> (moles/L)	0.21 <u>M</u> (moles/L)	17
TIC	9,100	7,850	-14
TOC	12,300	11,300	-8
Cl	1,800	1,650	-8
F	6,300	5,800	-8
$\text{NO}_3$	107,000	98,100	-8
$\text{NO}_2$	41,400	38,100	-8
$\text{SO}_4$	6,700	5,900	-12
$\text{PO}_4$	2,200	1,750	-20
Density	1.24 g/mL	1.23 g/mL	8
<p>[ ] Analyte was detected within 10 times the instrument detection limit and the uncertainty is estimated to exceed <math>\pm 15\%</math>.            &lt;DL-- Indicates analyte was not observed above the detection limit.            (a) Uncertainty, including standards bias, ranged from -10% to +30%            (b) These are the ICP-AES analytes of interest. All other analytes are reported opportunistically.            (c) Si appears to be a contaminant originating from the glass vials used during sample preparation.</p>			

### 3.8 Technetium Recovery

Some stages of the column run were monitored by beta scintillation counting. Multiple samples were collected during each process step: loading, feed displacement, water rinse, elution, and regeneration. All samples were analyzed for <sup>99</sup>Tc by ICP-MS. After the individual samples had been analyzed and the data evaluated, an effluent composite and eluate composite were formed. Both of the composites were analyzed for <sup>99</sup>Tc by ICP-MS. Since there is overlapping analytical data available for each processing stage, multiple mass balances are shown. Section 3.8.1 details the recovery of the column system based upon the analysis of the composite solutions for <sup>99</sup>Tc by ICP-MS. Section 3.8.2 details the recovery for the lead column loading and elution using <sup>99</sup>Tc by ICP-MS. Section 3.8.3 calculates the estimated technetium retained on the resin.

#### 3.8.1 System Recovery Using Composite Solution <sup>99</sup>Tc Data

The system recovery for <sup>99</sup>Tc in the AN-102/C-104 ion exchange run is shown in Table 3.10. The recovery is based upon the analysis of the final effluent and eluate composite samples and the regeneration solution from the lead column. These three solutions account for all of the samples collected during the column run. The effluent composite solution contains the ion exchange effluent, the loading samples taken during processing, the water rinse sample, and the feed-displacement samples. The eluate composite was simply a collection of each of the elution fractions. All values are based upon ICP-MS analysis of <sup>99</sup>Tc.

**Table 3.10.** Recovery for Technetium Recovery During Processing Using Composite Solution Data<sup>(a)</sup>

Column Processing Step	Technetium Activity, $\mu\text{Ci}$ (mg)	Percent of <sup>99</sup> Tc in Total Feed
Feed	49.7 (2.93)	100
Effluent composite <sup>(b)</sup>	28.9 (1.70)	58.1
Lead column eluate	19.1 (1.13)	38.4
Lead column regeneration	< 0.0011 (<0.00006)	<0.002
Total technetium recovered	48.0 (2.83)	97
(a) Based upon <sup>99</sup> Tc analysis of final solutions.		
(b) Effluent composite contains effluent, loading samples, DI water rinse, and feed-displacement solutions.		

A total of 49.7  $\mu\text{Ci}$  of <sup>99</sup>Tc was processed through the column system. The total recovery, based upon composite solutions, is 97% of the processed technetium. The data reported in Table 3.10 do not account for the technetium loaded on the lag column. The lag column loading and retention of technetium is included in Section 3.8.3.

#### 3.8.2 Recovery for Lead Column Loading and Elution Using Individual <sup>99</sup>Tc Sample Data

The loading and elution curves for the lead and lag columns combined with the feed displacement, water rinse, and regeneration solutions provide an accounting of all of the technetium passing through the system during processing of the AN-102/C-104 blended feed. A recovery for the lead-column loading

and elution of AN-102/C-104 tank-waste feed is shown in Table 3.9. The amount of technetium loaded onto the lead column was calculated by the following equation:

$$\sum_{i=0}^{i=N} [(1 - C / C_0)_i (C_0) (\Delta V)_i] \quad (3.1)$$

where  $C/C_0$  = ratio of Tc in each sample versus the Tc present in the initial feed  
 $C_0$  = initial feed technetium concentration  
 $\Delta V$  = volume of feed processed between each sample (including sample volume)  
 $N$  = number of samples collected during loading.

The amount of technetium eluted from the lead column during elution was determined using Equation 3.2.

$$\sum_{i=0}^{i=N} [(C / C_0)_i (C_0) (\Delta V)_i] \quad (3.2)$$

where  $C/C_0$  = ratio of Tc in each elution sample versus the Tc present in the initial feed  
 $C_0$  = initial feed technetium concentration  
 $\Delta V$  = volume of each elution sample  
 $N$  = number of samples collected during elution.

The lead column loading and elution recovery calculated for total technetium are shown in Table 3.11.

**Table 3.11.** Recovery for Loading and Elution of Technetium During Processing of AN-102/C-104 Blended Feed

Column Processing Step	Technetium Activity, μCi (mg)	Percent of <sup>99</sup> Tc in Total Feed, %
Total Technetium Processed	49.7 (2.93)	100
Loaded on Lead Column	25.0 (1.47)	50.3
Eluted from Lead Column <sup>(a)</sup>	25.2 (1.49)	50.7
Fraction Eluted from Lead Column	101%	
<sup>(a)</sup> Based upon the sum of μCi eluted during elution (25.13), feed displacement (0.030), water rinse (0.067), and regeneration stages (<0.0011).		

Since elution also occurred during feed displacement, rinsing, and regeneration of the lead column, the contribution of those samples was also included in the table. The data indicate that there was less than 1% retention of the technetium loaded on the column. The estimate of technetium retained on the column is based solely on solution analyses. When all actual waste testing is completed, the resin will be dissolved and analyzed to determine residual Tc and other metals.

### 3.8.3 Estimate of Technetium Retained on Lead and Lag Columns During Processing of AN-102/C-104 Blended Feed

Table 3.12 estimates the amount of technetium retained on the lead and lag columns during processing of the AN-102/C-104 blended feed. The calculation of the technetium loaded onto the lead column was determined using Equation 3.1. The amount of technetium eluted from the lead column was based upon the analysis of the individual eluate samples combined with the analysis of the regeneration samples, feed displacement samples, and water-rinse sample.

Direct analysis of the eluate sample composite should provide a more accurate value than summing the individual eluate sample results since it does not combine the analytical uncertainty of 37 measurements. However, the eluate composite result (19.2  $\mu\text{Ci}$ ) was 24% lower than the sum of the individual eluate samples (25.2  $\mu\text{Ci}$ ). This discrepancy is unexpectedly high, but is likely due to sample-preparation methods. An evaluation to determine the effect that sample preparation has on the measurement of technetium demonstrated that the technetium-solution concentration decreases as the digestion method becomes more vigorous (acid dilution, versus two types of acid digest). Thus, technetium was likely lost from the sample during acid digestion.

Equation 3.3 was used to calculate the amount of technetium passing through the lag column during processing of AN-102/C-104 blended feed:

$$\sum_{i=0}^{i=N} [(C/C_0)_i (C_0) (\Delta V_L)_i] \quad (3.3)$$

- where
- $C/C_0$  = ratio of Tc exiting the lead column with respect to the initial technetium feed concentration
  - $C_0$  = initial feed technetium concentration
  - $\Delta V_L$  = volume of feed processed by the lag column
  - $N$  = number of samples collected during loading of the lag column.

The concentration of technetium exiting the lead column, which represents the technetium entering the lag column, is calculated in the term  $(C/C_0) \times C_0$ . Equation 3.2 was used to calculate the technetium passing through the lag column during loading.

Essentially all of the technetium loaded onto the lead column was eluted; the calculations indicate that 1% of the processed technetium was retained on the two columns. The calculated retention of the lead column resulted in a negative value. It is possible that the amount of  $^{99}\text{Tc}$  eluted from the lead column includes  $^{99}\text{Tc}$  that was retained by the column during processing of AP-101. However, it may also be due to analytical uncertainty. The calculated system retention value, then, is due entirely to the lag column, which was not eluted. Note that the estimate of technetium retained on both columns is based solely on solution analyses. When all actual waste testing is completed, the resin will be dissolved and analyzed to determine residual Tc and other metals.

**Table 3.12.** Estimated Technetium Retained on Lead and Lag Columns During Processing of AN-102/C-104 Blended Feed

Column Processing Step	Technetium Activity, $\mu\text{Ci}$ (mg)	Percent of $^{99}\text{Tc}$ in Total Feed, %
Loaded on Lead Column	25.0 (1.47)	50.3
Eluted from Lead Column <sup>(a)</sup>	25.2 (1.49)	50.7
Estimated Retained on Lead Column	-0.23 (-0.02)	-0.5
Technetium Entering Lag Column	27.2 (1.61)	54.8
Eluted from Lag Column	26.6 (1.57)	53.4
Estimate Retained on Lag Column	0.6 (0.04)	1.3
Total Retained on Both Columns	0.6 (0.04)	1.3
(a) Based upon the sum of $\mu\text{Ci}$ eluted during elution (25.13), feed displacement (0.030), water rinse (0.067), and regeneration stages (<0.0011).		

### 3.9 Summary of Objectives and System Performance

The objectives of this testing were to

- demonstrate the effectiveness of all SL-639 process steps, including loading, feed displacement, DI water washing, elution, and resin regeneration
- investigate the potential for resin and/or column fouling
- demonstrate the  $^{99}\text{Tc}$  decontamination of an Envelope C material (Tank AN-102 blended with permeate from the filtration, washing and leaching of C-104 solids)
- provide batch-distribution measurements on actual tank waste feed.

Each of the objectives is discussed in detail below.

#### Demonstrate the effectiveness of all SL-639 process steps, including loading, feed displacement, DI water washing, elution, and resin regeneration

The processing of the column system using AN-102/C-104 blended feed and SL-639 resin progressed without difficulties. A total of 136 BVs of feed (680 mL) were processed through the dual-column system. The lead- and lag-column breakthroughs were approximately 46% and 43%, respectively, based upon  $^{99}\text{Tc}$  analytical data. The quantitation of the pertechnetate is suspected of being affected by the presence of an unidentified beta or gamma emitter; however, an increase in the pertechnetate breakthrough of approximately 6% can be observed. A comparison of the  $^{99}\text{Tc}$  and  $\text{TcO}_4^-$  values demonstrates that 50% of the technetium was in the pertechnetate form.

Feed displacement progressed smoothly as did the column rinse. The feed was reduced to less than 1% within the target of 2 apparatus volumes of 0.10 M NaOH. The elution of the technetium-loaded columns with DI water at 65°C progressed quickly. The concentration of  $^{99}\text{Tc}$  peaked in the 2<sup>nd</sup> BV, giving a  $C/C_0 = 48$ . The target  $C/C_0$  value of <0.01 was reached at the 16<sup>th</sup> BV.

The resin was regenerated with 2 BVs of 0.25 M NaOH. This volume was adequate to rinse the lead resin bed with caustic. The regeneration effluent was collected in one batch in which <sup>99</sup>Tc was not detected by the analytical method.

Investigate the potential for resin and/or column fouling

There were no anomalies observed during the column run. For example, during loading, feed displacement, and elution, there was no observed back pressure, no decrease in flowrate, no resin color change, no change in resin bed height or glass wool height, and no evidence of channeling.

Demonstrate the <sup>99</sup>Tc decontamination of an Envelope C material (Tank AN-102 blended with permeate from the filtration, washing, and leaching of C-104 solids)

Tank waste feed from Tank AN-102 blended with permeate from the filtration, washing, and leaching of Envelope D solids (C-104) was successfully processed through a dual 5.0-mL resin-bed column system that had previously been loaded and eluted with AP-101 tank waste feed. The AN-102/C-104 blended feed contained 79.5 μCi/L <sup>99</sup>Tc, and 680 mL of feed (136 BV) were processed through the column system. The total amount of technetium processed through the two columns was 49.7 μCi. The final effluent composite <sup>99</sup>Tc concentration was 44.1 μCi/L, which correlates to 44% removal of technetium. An overall <sup>99</sup>Tc DF value of 1.8 was obtained using the two 5.0-mL beds in series.

The <sup>99</sup>Tc concentration in the effluent from this test exceeds the plant target of removing <sup>99</sup>Tc to a level that would permit production of a LAW glass with a concentration of <sup>99</sup>Tc < 0.1 Ci/m<sup>3</sup>. The effluent <sup>99</sup>Tc concentration does not necessarily exceed the contract requirement however, since the contract limit of <sup>99</sup>Tc < 0.1 Ci per m<sup>3</sup> of LAW glass is based on a running average. These data illustrate the need for a study (calculations) of a best estimated running average <sup>99</sup>Tc concentration for the first ten or so LAW batches.

Provide batch-distribution measurements on actual tank waste feed

Batch distributions were measured with the AN-102/C-104 blended feed using SL-639 resin batch #010227CTC-9-23. The SL-639 resin demonstrated excellent removal of technetium: the <sup>99</sup>TcO<sub>4</sub><sup>-</sup> K<sub>d</sub> value was 660 mL/g, providing 85% removal of technetium. The <sup>99</sup>Tc K<sub>d</sub> value was low, ~70, due to the high fraction of non-per technetate present in the sample. Interpolation of the isotherm data provides an estimated TcO<sub>4</sub><sup>-</sup> K<sub>d</sub> value of ~620 at the initial feed conditions. The batch-λ value derived from the TcO<sub>4</sub><sup>-</sup> K<sub>d</sub> data interpolated to the initial feed conditions is ~310 BV.

## 4.0 Conclusions

All objectives of the testing were met.

- All process steps for  $^{99}\text{Tc}$  decontamination of AN-102/C-104 blended waste feed were successfully demonstrated.
- No resin or column fouling was observed.
- The  $^{99}\text{Tc}$  decontamination of an Envelope C waste (AN-102) blended with permeate from the filtration, washing, and leaching of Envelope D solids (C-104) was successfully demonstrated. A total of 136 BV (680 mL @ 79.5  $\mu\text{Ci } ^{99}\text{Tc/L}$ ) of feed was processed, resulting in 44% removal of  $^{99}\text{Tc}$ .
- The total technetium removal at the 250 BVs is predicted to meet or exceed the target criteria of 25%.
- The Tc effluent composite (~650 mL, 44  $\mu\text{Ci } ^{99}\text{Tc/L}$ ) and the Tc eluate composite (132 mL, 180  $\mu\text{Ci } ^{99}\text{Tc/L}$ ) solutions are available but have not been provided for downstream testing because of a change in the treatment-plant flowsheet.
- Batch-distribution measurements were made on the AN-102/C-104 blended feed. The batch- $\lambda$  value derived from the  $\text{TcO}_4^- K_d$  data interpolated to the initial feed conditions is ~310 BV.
- A comparison of the  $^{99}\text{Tc}$  and  $\text{TcO}_4^-$  results demonstrates that 50% of the technetium was in the pertechnetate form.

## 5.0 References

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## **APPENDIX A**

### **Sample Identification**

## Appendix A: Sample Identification

Sample ID	Sample #	Description
AN2-Tc-0	02-777	Initial feed sample
AN2-Tc-PL1 through 6	Not submitted for laboratory analyses	Lead and lag column reservoir collected during initial loading
AN2-Tc-LL1 through LL17	01-2040 to 2056	Loading samples from lead column
AN2-Tc-LP1 through PL9	01-2057 to 2065	Loading samples from lag column
AN2-Tc-FD-P1 through P16	01-2188 to 01-2203	Feed displacement samples
AN2-Tc-RL1	01-2204/01-2082	DI water rinse sample
AN2-Tc-E1-1 through E1-37	01-2083 to 2119	Lead column eluate samples
AN2-Tc-Regen 1	01-2205/01-2120	Lead column regeneration sample
Tc-AN2-Effluent	02-778	Effluent composite sample (contains feed displacement and rinse solutions)
Tc-AN2-Eluate	02-776	Eluate composite sample

## **APPENDIX B**

### **Column Run Spreadsheets**

## Appendix B: Column Run Spreadsheets

**Loading**

Feed: AN102/C104 supernate with Cs-137 removed by ion exchange (SL644)  
 Resin: SuperLig 639, batch #010227CTC-9-23  
 Tracer: none used  
 Bed volume, Col 1 = 5.0 mL (3.3 cm)  
 Bed volume, Col 2 = 5.0 mL (3.4 cm)  
 Empty Eff Bottle w/cap (wo/h 120.55 g)  
 Bed volumes diverted to waste none  
 Supernate Density = 1.2398  
 Feed analyses: Tc-99,  $\mu\text{Ci/mL}$   
 Flow Rate: approx 16 mL/hr or ~3.2 CV/hr  
 Start Date and Time: 8/6/2001 8:00 AM  
 End Date and Time: 8/6/2001 9:40 AM

Table 1. Column Loading Initial Sample Data Sheet

Sample	Source	# column volumes	Elapsed Time at Start, hh:mm	Projected Date/Time	Vol., mL	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Eff. Bottle Mass (g)	Sample + Vial + Cap Mass, g	Sample Mass, g	Count date	Count start time	file name
Effluent Bottle	--	--	--	--	--	120.55	--	--	/	--	--	No Counting or ICP-MS done		
AN2-Tc-0-C	Feed Bot	--	0:00	--	5	17.092	--	--		--	--			
AN2-Tc-0b-C	Feed Bot	--	0:00	--	10	16.96	--	--		--	--			
AN2-Tc-PL1	Col 2	0	0:00	8/6/2001 8:00 AM	5	17.068	8:00 AM	8:20 AM		21.6115	4.5436			
AN2-Tc-PL2	Col 2	1	0:20	8/6/2001 8:20 AM	5	17.104	8:20 AM	8:54 AM		25.4609	8.3571			
AN2-Tc-PL3	Col 2	2	0:40	8/6/2001 8:40 AM	5	17.113	8:54 AM	9:10 AM		18.2624	1.1499			
AN2-Tc-PL4	Col 2	3	1:00	8/6/2001 9:00 AM	5	16.867	9:10 AM	9:20 AM		19.6562	2.7895			
AN2-Tc-PL5	Col 2	4	1:20	8/6/2001 9:20 AM	5	16.947	9:20 AM	9:40 AM		22.7900	5.8428			
AN2-Tc-PL6	Col 2	5	1:40	8/6/2001 9:40 AM	5	17.044	--	--	--	--				

/ indicates no information relevant to these cells  
 -- indicates no data

Bed Height of Column 1 @ beginning	3.2 cm	Height of glass wool in Column 1 @ beginning	0.4 cm
Bed Height of Column 2 @ beginning	3.2 cm	Height of glass wool in Column 2 @ beginning	0.3 cm
Bed Height of Column 1 @ end	3.2 cm	Height of glass wool in Column 1 @ end	0.4 cm
Bed Height of Column 2 @ end	3.2 cm	Height of glass wool in Column 2 @ end	0.3 cm

B.1

**Loading**

Feed: AN102/C104 supernate with Cs-137 removed by ion exchange (SL644)  
 Resin: SuperLig 639, batch #010227CTC-9-23  
 Tracer: none used  
 Bed volume, Col 1 = 5.0 mL (3.3 cm)  
 Bed volume, Col 2 = 5.0 mL (3.4 cm)  
 MT Eff Bot. w/cap (w/hole) 120.55 g  
 Bed volumes diverted to waste = none  
 Supernate Density = 1.2398  
 Feed analyses: Tc-99,  $\mu\text{Ci/mL}$  (by TEVA Resin IX and scintillation counting)  
 Flow Rate: approx 16 mL/hr or ~3.2 CV/hr  
 Start Date and Time: 8/6/2001 8:00 AM  
 End Date and Time: 8/8/2001 7:17 AM

Table 2. Data Collection for Tc Loading of Lead Column in AN102/C104 Run

Sample	Source	# CV	Elapsed Time at Start, hh:mm	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Eff. Bottle Mass (g)	Sample+ Vial+ Cap Mass (g)	Sample Mass (g)	Effluent Mass, g	Est. Effluent Vol	Est. Flow Rate	#CV
AN2-Tc-0	Feed bot.				10		--		129.41		--		1.2398	g/mL	--
AN2-Tc-LL1	Col 1	1	0:00	8/6/2001 9:40 AM	3	17.0582	9:40 AM	9:43 AM	129.41 <sup>a</sup>	17.8278	0.7696	0.8	0.6	13.45	0.1
AN2-Tc-LL2	Col 1	3	1:40	8/6/2001 10:40 AM	5	17.0891	10:40 AM	10:45 AM	137.70	23.1990	6.1099	15.2	12.2	14.45	2.4
AN2-Tc-LL3	Col 1	6	5:00	8/6/2001 11:40 AM	3	17.0531	11:40 AM	11:43 AM	144.30	20.5329*	3.4798	31.4	25.4	14.85	5.1
AN2-Tc-LL4	Col 1	9	8:20	8/6/2001 12:40 PM	3	17.0064	12:40 PM	12:43 PM	150.70	20.5466*	3.5402	41.4	33.4	12.55	6.7
AN2-Tc-LL5	Col 1	19	11:40	8/6/2001 4:00 PM	5	17.0987	4:00 PM	4:05 PM	215.10	23.2591	6.1604	115.6	93.2	13.94	18.6
AN2-Tc-LL6	Col 1	29	15:00	8/6/2001 7:20 PM	3	17.1352	7:18 PM	7:30 PM	272.00	20.7847*	3.6495	176.1	142.1	14.44	28.4
AN2-Tc-LL7	Col 1	39	18:20	8/6/2001 10:40 PM	3	17.0801	10:40 PM <sup>b</sup>	10:52 PM	326.60	20.7302*	3.6501	240.8	194.2	14.71	38.8
AN2-Tc-LL8	Col 1	49	21:40	8/7/2001 2:00 AM	5	17.0631	2:00 AM	2:20 AM	343.20	23.1765	6.1134	263.5	212.5	12.71	42.5
AN2-Tc-LL9	Col 1	59	25:00	8/7/2001 5:20 AM	3	16.9905	5:20AM <sup>c</sup>	5:32 AM	405.70	20.7341*	3.7436	333.5	269.0	13.53	53.8
AN2-Tc-LL10	Col 1	69	28:20	8/7/2001 8:40 AM	3	17.0350	8:40 AM	8:43 AM	455.60	20.6040	3.5690	387.0	312.2	13.45	62.4
AN2-Tc-LL11	Col 1	79	31:40	8/7/2001 12:00 PM	5	17.1138	12:00 PM	12:05 PM <sup>d</sup>	508.70	23.1839*	6.0701	452.4	364.9	13.67	73.0
AN2-Tc-LL12	Col 1	89	35:00	8/7/2001 3:20 PM	3	16.9370	3:22 PM	3:25 PM	571.50	20.8957	3.9587	519.2	418.8	14.01	83.8
AN2-Tc-LL13	Col 1	99	38:20	8/7/2001 6:40 PM	3	17.0953	6:40 PM	6:43 PM <sup>e</sup>	631.00	21.1069	4.0116	586.8	473.3	14.24	94.7
AN2-Tc-LL14	Col 1	109	41:40	8/7/2001 10:00 PM	5	17.0800	10:00 PM	10:05 PM	701.80	24.3858	7.3058	664.9	536.3	14.60	107.3
AN2-Tc-LL15	Col 1	119	45:00	8/8/2001 1:20 AM	3	17.0128	1:20 AM	1:32 AM	761.70	21.1041	4.0913	736.3	593.9	14.89	118.8
AN2-Tc-LL16	Col 1	129	48:20	8/8/2001 4:40 AM	3	16.9624	4:40 AM	4:43 AM	832.50	21.2930	4.3306	811.4	654.5	15.14	130.9
AN2-Tc-LL17	Col 1	139	51:40	8/8/2001 7:16 AM	1	17.0350	7:16 AM <sup>f</sup>	7:17 AM	861.10	18.3941	1.3591	845.8	682.2	14.94	136.4

Notes:

- \*samples weighed after removing 100 $\mu\text{L}$  for counting
- <sup>a</sup>mass of effluent bottle with lid (8.8605g). Previous mass was bottle w/o lid
- <sup>b</sup>valve 3 left closed from ~11:40 until ~1AM. Pressure built up to 30 psi. Confirmed - sampling began at 10:40 AM
- <sup>c</sup>thought the wrong column was sampled, but it was taken from valve 2
- <sup>d</sup>changed flow rate to 55% @ 12:25 PM 8/7/01
- <sup>e</sup>increased pump setting to 60% at 6:59 PM
- <sup>f</sup>column loading complete, check sample pulled at end for TcO<sub>4</sub> values

TcO4- initial feed 0.0273 uCi/mL  
Tc-99 initial feed 0.074485881 uCi/mL  
Tc-99 initial feed (mass balance) 0.07947118 uCi/mL

Table 2. (continued) Data Collection for Tc Loading of Lead Column in AN102/C104 Run - Counting

Sample	cpm Tc-99, LSC counting	Tc-99 C/Co%	Tc-99 Data mCi/mL	Dilution Factor	Tc-99 mCi/mL	Tc-99 % C/Co	$\mu$ Ci/mL TcO <sub>4</sub> <sup>-</sup>	TcO <sub>4</sub> <sup>-</sup> % C/Co	uCi/mL	Volume	uCi	uCi/mL	Volume	uCi
AN2-Tc-0	7758	--	1.72E-05	4334	7.45E-02	4.28E+00	2.73E-02		TcO <sub>4</sub> Mass Balance			Tc-99 Mass Balance		
AN2-Tc-LL1	1029	13%	9.84E-05	675	6.64E-02	89%	3.49E-03	13%	0.02381	0.6	0.015	0.0086	0.6	0.005
AN2-Tc-LL2	--	--	6.12E-05	683	4.18E-02	56%	3.33E-03	12%	0.02397	11.6	0.278	0.0349	11.6	0.405
AN2-Tc-LL3	1027	13%	6.41E-05	661	4.23E-02	57%	--	--	0.02379	13.1	0.312	0.0343	13.1	0.450
AN2-Tc-LL4	1091	14%	5.95E-05	652	3.88E-02	52%	3.69E-03	14%	0.02361	8.0	0.189	0.0381	8.0	0.305
AN2-Tc-LL5	--	--	6.40E-05	601	3.85E-02	52%	--	--	0.02358	59.9	1.412	0.0384	59.9	2.300
AN2-Tc-LL6	1009	13%	6.14E-05	687	4.21E-02	57%	3.75E-03	14%	0.02355	48.8	1.150	0.0345	48.8	1.685
AN2-Tc-LL7	1213	16%	6.18E-05	645	3.99E-02	54%	--	--	0.02338	52.1	1.219	0.0369	52.1	1.926
AN2-Tc-LL8	--	--	6.01E-05	619	3.72E-02	50%	4.09E-03	15%	0.02321	18.3	0.425	0.0398	18.3	0.728
AN2-Tc-LL9	1001	13%	6.15E-05	644	3.97E-02	53%	--	--	0.02324	56.5	1.313	0.0372	56.5	2.100
AN2-Tc-LL10	--	--	6.14E-05	634	3.89E-02	52%	4.03E-03	15%	0.02327	43.1	1.004	0.038	43.1	1.637
AN2-Tc-LL11	1172	15%	6.08E-05	675	4.10E-02	55%	--	--	0.02308	52.8	1.218	0.0357	52.8	1.884
AN2-Tc-LL12	--	--	6.14E-05	661	4.06E-02	54%	4.42E-03	16%	0.02288	53.8	1.232	0.0362	53.8	1.947
AN2-Tc-LL13	1121	14%	6.45E-05	671	4.33E-02	58%	--	--	0.02253	54.5	1.229	0.0333	54.5	1.814
AN2-Tc-LL14	--	--	6.18E-05	617	3.81E-02	51%	5.12E-03	19%	0.02218	63.0	1.397	0.0388	63.0	2.446
AN2-Tc-LL15	1376	18%	6.42E-05	609	3.91E-02	53%	--	--	0.0221	57.6	1.272	0.0377	57.6	2.173
AN2-Tc-LL16	1352	17%	6.39E-05	621	3.97E-02	53%	5.28E-03	19%	0.02202	60.6	1.334	0.0371	60.6	2.251
AN2-Tc-LL17	1554	20%	6.17E-05	667	4.12E-02	55%	--	--	0.02202	27.8	0.611	0.0356	27.8	0.987
									total vol	682	15.6	uCi Tc total		23.5
									total CV	136	0.92	mg Tc total		1.38

-- indicates no data

Table 3. Data Collection for Tc Loading of Lag Column in AN102/C104 Run

Sample	Source	# CV	Elapsed Time at Start	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Eff. Bottle Mass (g)	Sample+ Vial+ Cap Mass (g)	Sample Mass (g)	Effluent Mass, g	Est. Effluent Vol	Est. Flow Rate	#CV
AN2-Tc-LP1	Col 2	3	1:40	8/6/2001 10:40 AM	5	16.9099	11:00 AM	11:20 AM	137.70	23.0913	6.1814	14.5	11.7	14.11	4
AN2-Tc-LP2	Col 2	9	8:20	8/6/2001 12:40 PM	3	16.9424	11:20 AM	11:23 AM	150.70	20.6002*	3.6578	31.1	25.1	10.08	8
AN2-Tc-LP3	Col 2	29	15:00	8/6/2001 7:20 PM	5	17.1533	7:30 PM	7:50 PM	272.00	23.5432	6.3899	158.8	128.1	13.75	25
AN2-Tc-LP4	Col 2	49	21:40	8/7/2001 2:00 AM	3	17.0438	2:20 AM	2:32 AM	343.20	20.8458*	3.8020	233.8	188.6	11.98	43
AN2-Tc-LP5	Col 2	69	28:20	8/7/2001 8:40 AM	5	17.0089	8:52 AM	9:12 AM	455.60	23.2612	6.2523	352.5	284.3	12.67	62
AN2-Tc-LP6	Col 2	89	35:00	8/7/2001 3:20 PM	3	16.9527	3:34 PM	3:46 PM	571.50	21.0589	4.1062	472.5	381.1	13.09	84
AN2-Tc-LP7	Col 2	109	41:40	8/7/2001 10:00 PM	5	16.9735	10:20 PM	10:40 PM	701.80	24.3608	7.3873	610.2	492.1	13.74	107
AN2-Tc-LP8	Col 2	129	48:20	8/8/2001 4:40 AM	3	16.9008	4:52 AM	5:04 AM	832.50	21.3611	4.4603	745.3	601.2	14.15	131
AN2-Tc-LP9	Col 2	149	55:00	8/8/2001 7:20 AM	1	17.1193	7:20 AM	7:24 AM	861.10	18.7308	1.6115	775.5	625.5	29.20	136

Notes:

\*samples weighed after removing 100µL for counting

<sup>f</sup>column loading complete, sample pulled for TcO4 value measurement

- indicates no data

Table 3. (continued) Data Collection for Tc Loading of Lag Column in AN102/C104 Run

cpm Tc-99, LSC counting	beta Tc-99 C/Co%	Tc-99 Data mCi/mL	Dilution Factor	Tc-99 mCi/mL	Tc-99 % C/Co	μCi/mL TcO <sub>4</sub> <sup>-</sup>	TcO <sub>4</sub> <sup>-</sup> % C/Co	Out of Lag	Into Lag	[Tc] into lag	V into lag column	uCi into lag	uCi out of lag
No Data		5.71E-05	627	3.58E-02	48%	3.53E-03	13%	48%	89%	0.071	11.7	0.83	0.45
		6.05E-05	612	3.71E-02	50%	--	--	50%	57%	0.045	13.4	0.61	0.53
		6.41E-05	616	3.94E-02	53%	3.83E-03	14%	53%	52%	0.041	103.0	4.23	4.33
		5.87E-05	624	3.66E-02	49%	--	--	49%	54%	0.043	60.5	2.57	2.36
		6.07E-05	671	4.07E-02	55%	3.35E-03	12%	55%	53%	0.042	95.7	4.05	4.16
		6.64E-05	618	4.10E-02	55%	--	--	55%	55%	0.044	96.8	4.24	4.23
		6.01E-05	616	3.70E-02	50%	3.17E-03	12%	50%	58%	0.046	111.1	5.13	4.39
		5.85E-05	754	4.41E-02	59%	3.83E-03	14%	59%	53%	0.042	109.0	4.55	5.13
		6.04E-05	631	3.82E-02	51%	--	--	51%	53%	0.042	24.4	1.03	0.99

-- indicates no data

uCi in	27.24	26.57	uCi out
		0.6634	uCi retained

**Wash**

Feed: 0.1 M NaOH  
 Resin: SuperLig 639, batch #010227CTC-9-23  
 Tracer: none  
 Bed volume, Col 1 = 5.0 mL (3.3 cm)  
 Bed volume, Col 2 = 5.0 mL (3.4 cm)  
 Bed volumes diverted to waste = none  
 NaOH Density = 1.008 g/mL  
 Feed analyses: Tc-99, µCi/mL  
 Flow Rate: approx 16 mL/hr or 3.2 CV/hr  
 Start Date and Time: 8/8/2001 7:25 AM  
 End Date and Time: 8/8/2001 12:04 PM

Table 4. Data Collection during 0.10 M NaOH Feed Displacement of Columns 1 and 2

Sample	Source	# column volumes	Elapsed Time	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial+ Cap Mass (g)	Bed Height Col 1 / Glass Wool Ht	Bed Height Col 2 / Glass Wool Ht	Sample Mass (g)
0.10 M NaOH Feed Displacement Solution					5							
AN2-Tc-FDP1	Col 2	0	0:00	8/8/2001 7:25 AM	5	17.0608	7:25 AM	7:45 AM	24.2879	3.25/0.35	3.2/0.25	7.2271
AN2-Tc-FDP2	Col 2	1.00	0:20	8/8/2001 7:45 AM	5	17.0192	7:45 AM	8:10 AM	25.5470	3.25/0.35	3.2/0.25	8.5278
AN2-Tc-FDP3	Col 2	0.66	0:40	8/8/2001 8:05 AM	5	17.1018	8:10 AM	8:25 AM	22.4323	3.25/0.35	3.2/0.25	5.3305
AN2-Tc-FDP4	Col 1	1.00	1:00	8/8/2001 8:25 AM	1	17.0329	8:25 AM	8:29 AM	17.5939	3.25/0.35	3.2/0.25	0.5610
AN2-Tc-FDP5	Col 2	1.33	1:20	8/8/2001 8:45 AM	5	17.0854	8:29 AM	8:31 AM <sup>c</sup>	18.1777	3.25/0.35	3.2/0.25	1.0923
AN2-Tc-FDP6	Col 2	1.66	1:40	8/8/2001 9:05 AM	5	17.0402	8:31 AM	8:51 AM	23.3959	3.25/0.35	3.2/0.25	6.3557
AN2-Tc-FDP7	Col 2	1.99	2:00	8/8/2001 9:25 AM	4	17.1528	8:51 AM	9:03 AM	20.7076	3.25/0.35	3.2/0.25	3.5548
AN2-Tc-FDP8	Col 1	2.32	2:20	8/8/2001 9:45 AM	1	16.8872	9:03 AM	9:07 AM	17.9031	3.25/0.35	3.2/0.25	1.0159
AN2-Tc-FDP9	Col 2	2.65	2:40	8/8/2001 10:05 AM	4	17.0429	9:07 AM	9:19 AM	20.4710	3.25/0.35	3.2/0.25	3.4281
AN2-Tc-FDP10	Col 1	3.00	3:00	8/8/2001 10:25 AM	1	17.2420	9:19 AM	9:23 AM	18.2579 <sup>b</sup>	3.25/0.35	3.2/0.25	1.0159
AN2-Tc-FDP11	Col 2	3.33	3:20	8/8/2001 10:45 AM	5	17.2247	9:23 AM	9:35 AM <sup>a</sup>	20.6360	3.25/0.35	3.2/0.25	3.4113
AN2-Tc-FDP12	Col 1	3.66	3:40	8/8/2001 11:05 AM	1	17.1543	10:20 AM	10:24 AM <sup>a</sup>	18.1755 <sup>b</sup>	3.25/0.35	3.2/0.25	1.0212
AN2-Tc-FDP13	Col 2	3.99	4:00	8/8/2001 11:25 AM	5	17.1262	11:00 AM	11:20 AM	22.7119	3.25/0.35	3.2/0.25	5.5857
AN2-Tc-FDP14	Col 1	4.32	4:20	8/8/2001 11:45 AM	1	17.1436	11:20 AM	11:24 AM <sup>a</sup>	18.1606 <sup>b</sup>	3.25/0.35	3.2/0.25	1.0170
AN2-Tc-FDP15	Col 2	4.65	4:40	8/8/2001 12:05 PM	5	17.0468	11:40 AM	12:00 PM	22.5697	3.25/0.35	3.2/0.25	5.5229
AN2-Tc-FDP16	Col 1	4.98	5:00	8/8/2001 12:25 PM	1	17.2788	12:00 PM	12:04 PM <sup>a</sup>	18.2971 <sup>b</sup>	3.25/0.35	3.2/0.25	1.0183

Notes

<sup>a</sup> Stopped pump

<sup>b</sup> Weighed after removing 100µL for counting

<sup>c</sup> AN2-Tc-FD4 and AN2-Tc-FD5 were combined prior to performing measurements

 indicates no information relevant to these cells

 indicates no data

1.2298	density AN102/C104 (used in spreadsheet calculations)
1.008	density 0.10 M NaOH (used in spreadsheet calculations)

Table 4. (continued) Data Collection during 0.10 M NaOH Feed Displacement of Columns 1 and 2 - Counting

Sample	Approximate Volume mL	Calculated # CV's	cpm, 100µL (Teva IX)	Tc Beta Data µCi/mL	Tc-99 Data µCi/mL	Dilution Factor	Tc-99 µCi/mL	Tc-99 % C/Co	uCi/mL Tc (eluted)	Volume (during elution)	uCi Tc (eluted)
0.10 M NaOH	/		7758	0.0273 uCi/mL		/		0.0795 uCi/mL			
AN2-Tc-FDP1	5.88	1.2	--	--	6.05E-05	626	3.79E-02	48%	This stage in feed displacement proceeded as planned, the feed was displaced. However starting at FDP10, Tc began to elute. Thus, it is necessary to calculate the amount of Tc eluted starting with FDP10		
AN2-Tc-FDP2	6.93	2.6	--	--	5.82E-05	619	3.60E-02	45%			
AN2-Tc-FDP3	4.33	3.4	--	--	5.87E-05	643	3.78E-02	47%			
AN2-Tc-FDP4	0.46	3.5	--	--	3.19E-05	690	2.20E-02	28%			
AN2-Tc-FDP5	0.89	3.7	73920	953%	1.48E-05	625	9.22E-03	12%			
AN2-Tc-FDP6	5.17	4.7	--	--	3.23E-05	551	1.78E-02	22%			
AN2-Tc-FDP7	3.53	5.4	--	--	1.14E-05	609	6.92E-03	8.7%			
AN2-Tc-FDP8	1.01	5.6	13189	170%	4.37E-06	642	2.81E-03	3.5%			
AN2-Tc-FDP9	3.40	6.3	--	--	4.81E-06	618	2.97E-03	3.7%			
AN2-Tc-FDP10	1.01	6.5	6188	80%	4.42E-06	645	2.85E-03	3.6%	0.0028	32.6	--
AN2-Tc-FDP11	3.38	7.2	--	--	2.03E-06	619	1.26E-03	1.6%	--	--	--
AN2-Tc-FDP12	1.01	7.4	5289	68%	6.69E-06	653	4.37E-03	5.5%	0.0044	37.0	--
AN2-Tc-FDP13	5.54	8.5	--	--	1.60E-06	652	1.04E-03	1.3%	--	--	--
AN2-Tc-FDP14	1.01	8.7	4587	59%	8.02E-06	652	5.23E-03	6.6%	0.0052	43.5	--
AN2-Tc-FDP15	5.48	9.8	--	--	1.09E-06	623	6.79E-04	0.9%	--	--	--
AN2-Tc-FDP16	1.01	10.0	4449	57%	9.78E-06	642	6.28E-03	7.9%	0.0063	50.0	--
area calculation <sup>d</sup>									1/2*b*h =	0.030	uCi eluted

<sup>d</sup> elution during feed displacement starts at FDP-10 and continues linearly through FDP-16. The area is described by a triangle ; therefore 1/2\*b\*h using sample results from FDP10, 12, 14 and 16 will give area under curve

/	indicates no information relevant to these cells
--	indicates no data

**Rinse**

Feed: Deionized water  
 Resin: SuperLig 639, batch #010227CTC-9-23  
 Tracer: none  
 Bed volume, Col 1 = 5 mL (3.3 cm)  
 Bed volumes diverted to waste = none  
 Supernate Density = 0.997 g/mL  
 Feed analyses: Tc-99, mCi/mL  
 Flow Rate: approx 16 mL/hr or 3.2 CV/hr  
 Start Date and Time: 8/8/2001 12:41 PM  
 End Date and Time: 8/8/2001 1:22 PM

Table 5. Data Collection for DI water Rinse of Resin Beds

Sample	Source	# column volumes	Elapsed Time	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial + Cap Mass (g)	Bed Height Col 1 / Glass Wool Ht	Bed Height Col 2 / Glass Wool Ht	Sample Mass (g)
AN2-Tc-RL1	Col 1	1	0:40	8/8/2001 12:41 PM	10	17.2683	12:41 PM	1:22 PM	26.6851	3.25/0.35	3.2/0.25	9.4168

B.8

Sample	Tc-99 Data $\mu\text{Ci/mL}$	Dilution Factor	Tc-99 $\mu\text{Ci/mL}$	Tc-99 % C/Co	$\text{uCi/mL}$	Volume, mL	$\text{uCi}$	$\text{uCi/mL}$	Volume, mL	$\text{uCi}$
					$^{95\text{m}}\text{TcO}_4$ Mass Balance		Tc-99 Mass Balance			
AN2-Tc-RL1	8.00E-05	84	6.69E-03	8.98%	no data		7.13E-03	9.42	0.0672	

Tc-99 control = 

0.0745	$\text{uCi/mL}$
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 Rinse Solution = 

0.079471	$\text{uCi/mL}$
----------	-----------------

 indicates no information relevant to these cells

**Elution**

Feed: H<sub>2</sub>O at 65°C  
 Resin: SuperLig 639, batch #010227CTC-9-23  
 Tracer: none used  
 Bed volume, Col 1 = 5.0 mL (3.3 cm)  
 Bed volume, Col 2 = 5.0 mL (3.4 cm)  
 Bed volumes diverted to waste = none  
 Feed analyses: Tc-99, mCi/mL  
 Flow Rate: 5 mL/hr or 1 CV/hr  
 Start Date and Time: 8/8/2001 1:30 PM  
 End Date and Time: 8/9/2001 5:00 PM

Table 6. Data Collection for Tc Elution of Column 1 (lead column) in AN102/C104 Run @ 65C

Sample	Source	# column volumes	Elapsed Time	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial+ Cap Mass (g)	Bed Height Col 1 / Glass Wool Ht	Sample Mass (g)	Count Date, Tc-99	count time (min)
AN2-Tc-E-0C	DI water eluent	--	--	--	--	16.9874	--	--	--	--	--	8/14/2001	20
AN2-Tc-E1-1	Col 1	0.5	0:00	8/8/2001 1:30 PM	2.5	17.1048	1:30 PM	2:00 PM	19.7525	3.25/.25	2.6477	8/14/2001	20
AN2-Tc-E1-2	Col 1	1	0:30	8/8/2001 2:00 PM	2.5	16.9233	2:00 PM	2:03 PM	19.2694	3.25/.25	2.3461	8/14/2001	20
AN2-Tc-E1-3	Col 1	1.5	1:00	8/8/2001 2:30 PM	2.5	16.9907	2:03 PM	3:00 PM	19.3118	3.25/.25	2.3211	8/14/2001	20
AN2-Tc-E1-4	Col 1	2	1:30	8/8/2001 3:00 PM	2.5	16.9924	3:00 PM	3:30 PM	19.3047	3.25/.25	2.3123	8/14/2001	20
AN2-Tc-E1-5	Col 1	2.5	2:00	8/8/2001 3:30 PM	2.5	17.0219	3:30 PM	4:00 PM	19.3065	3.25/.25	2.2846	8/14/2001	20
AN2-Tc-E1-6	Col 1	3	2:30	8/8/2001 4:00 PM	2.5	17.1525	4:00 PM	4:30 PM	19.4261	3.25/.25	2.2736	8/14/2001	20
AN2-Tc-E1-7	Col 1	3.5	3:00	8/8/2001 4:30 PM	2.5	16.8280	4:30 PM	5:00 PM	19.1145	3.25/.25	2.2865	8/14/2001	20
AN2-Tc-E1-8	Col 1	4	3:30	8/8/2001 5:00 PM	2.5	17.0494	5:00 PM	5:30 PM	19.3303	3.25/.25	2.2809	8/14/2001	20
AN2-Tc-E1-9	Col 1	4.5	4:00	8/8/2001 5:30 PM	2.5	17.0536	5:30 PM	6:00 PM	19.3124	3.25/.25	2.2588	8/14/2001	20
AN2-Tc-E1-10	Col 1	5	4:30	8/8/2001 6:00 PM	2.5	17.1193	6:00 PM	6:30 PM	19.7454	3.25/.25	2.6261	8/14/2001	20
AN2-Tc-E1-11	Col 1	5.5	5:00	8/8/2001 6:30 PM	2.5	17.0797	6:30 PM	7:00 PM	19.7006	3.25/.25	2.6209	8/14/2001	20
AN2-Tc-E1-12	Col 1	6	5:30	8/8/2001 7:00 PM	2.5	17.1300	7:00 PM	7:30 PM	19.7737	3.25/.25	2.6437	8/14/2001	20
AN2-Tc-E1-13	Col 1	6.5	6:00	8/8/2001 7:30 PM	2.5	17.2099	7:30 PM	8:00 PM	19.8234	3.25/.25	2.6135	8/14/2001	20
AN2-Tc-E1-14	Col 1	7	6:30	8/8/2001 8:00 PM	2.5	17.1649	8:00 PM	8:30 PM	19.5961	3.25/.25	2.4312	8/14/2001	20
AN2-Tc-E1-15	Col 1	7.5	7:00	8/8/2001 8:30 PM	2.5	17.0878	8:30 PM	9:00 PM	19.4241	3.25/.25	2.3363	8/14/2001	20
AN2-Tc-E1-16	Col 1	8	7:30	8/8/2001 9:00 PM	2.5	17.1139	9:00 PM	9:30 PM	18.3332*	3.25/.25	1.2193	8/14/2001	20
AN2-Tc-E1-17	Col 1	8.5	8:00	8/8/2001 9:30 PM	2.5	17.0883	9:30 PM	10:00 PM	19.5859	3.25/.25	2.4976	8/14/2001	20

B.9

 indicates no information relevant to these cells  
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TcO4- initial feed 0.0273 uCi/mL (used in spreadsheet calculations)  
 Tc-99 initial feed 0.0745 uCi/mL (used in spreadsheet calculations)  
 Tc-99 initial feed (mass balance) 0.079471 uCi/mL (used in spreadsheet calculations)

Table 6. (continued) Data Collection for Tc Elution of Column 1 (lead column) in AN102/C104 Run @ 65C - Counting

Sample	Vol counted, mL	Net counts Tc-99	%C/Co Tc-99	Tc-99 Data mCi/mL	Dilution Factor	Tc-99 mCi/mL	Tc-99 % C/Co	uCi/mL	Volume, mL	uCi	uCi/mL	Volume, mL	uCi
AN2-Tc-E-0C	0.1	7739	/					<sup>95m</sup> TcO <sub>4</sub> Mass Balance			Tc-99 Mass Balance		
AN2-Tc-E1-1	0.1	13354	1.73	6.28E-05	691	4.34E-02	60%	4.71E-02	2.65	0.125	4.75E-02	2.65	0.126
AN2-Tc-E1-2	0.1	72521	9.37	2.04E-04	1322	2.70E-01	372%	2.56E-01	2.35	0.600	2.96E-01	2.35	0.694
AN2-Tc-E1-3	0.1	206918	26.74	6.32E-04	1559	9.85E-01	1357%	7.30E-01	2.32	1.694	1.08E+00	2.32	2.504
AN2-Tc-E1-4	0.1	433664	56.04	1.93E-03	1345	2.59E+00	3577%	1.53E+00	2.31	3.537	2.84E+00	2.31	6.573
AN2-Tc-E1-5	0.1	593252	76.66	2.69E-03	1287	3.46E+00	4775%	2.09E+00	2.28	4.781	3.79E+00	2.28	8.670
AN2-Tc-E1-6	0.1	325390	42.05	1.50E-03	1263	1.90E+00	2619%	1.15E+00	2.27	2.610	2.08E+00	2.27	4.731
AN2-Tc-E1-7	0.1	1000533	129.28	3.34E-04	1617	5.41E-01	745%	3.53E+00	2.29	8.070	5.92E-01	2.29	1.354
AN2-Tc-E1-8	0.1	11430	1.48	3.67E-05	1648	6.04E-02	83%	4.03E-02	2.28	0.092	6.62E-02	2.28	0.151
AN2-Tc-E1-9	0.1	4828	0.62	1.36E-05	1345	1.82E-02	25.1%	1.70E-02	2.26	0.038	2.00E-02	2.26	0.045
AN2-Tc-E1-10	0.1	3413	0.44	9.13E-06	1633	1.49E-02	20.6%	1.20E-02	2.63	0.032	1.63E-02	2.63	0.043
AN2-Tc-E1-11	0.1	2610	0.34	6.73E-06	1684	1.13E-02	15.6%	9.21E-03	2.62	0.024	1.24E-02	2.62	0.033
AN2-Tc-E1-12	0.1	2277	0.29	5.63E-06	1342	7.55E-03	10.4%	8.03E-03	2.64	0.021	8.27E-03	2.64	0.022
AN2-Tc-E1-13	0.1	2042	0.26	8.08E-06	1742	1.41E-02	19.4%	7.20E-03	2.61	0.019	1.54E-02	2.61	0.040
AN2-Tc-E1-14	0.1	1856	0.24	7.28E-05	88	6.43E-03	8.9%	6.55E-03	2.43	0.016	7.04E-03	2.43	0.017
AN2-Tc-E1-15	0.1	1825	0.24	7.00E-05	82	5.75E-03	7.9%	6.44E-03	2.34	0.015	6.30E-03	2.34	0.015
AN2-Tc-E1-16	0.1	1915	0.25	7.52E-05	101	7.61E-03	10.5%	6.76E-03	1.22	0.008	8.34E-03	1.22	0.010
AN2-Tc-E1-17	0.1	3846	0.50	1.01E-04	87	8.83E-03	12.2%	1.36E-02	2.50	0.034	9.67E-03	2.50	0.024

B.10



indicates no information relevant to these cells

indicates no data

Table 6 (continued). Data Collection for Tc Elution of Column 1 (lead column) in AN102/C104 Run @ 65C

Sample	Source	# column volumes	Elapsed Time	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial+ Cap Mass (g)	Bed Height Col 1 / Glass Wool Ht	Sample Mass (g)	Count Date, Tc-99	count time (min)
AN2-Tc-E1-18	Col 1	9	8:30	8/8/2001 10:00 PM	2.5	17.1791	10:00 PM	10:30 PM	19.6689	3.25/.25	2.4898	8/14/2001	20
AN2-Tc-E1-19	Col 1	9.5	9:00	8/8/2001 10:30 PM	2.5	17.1647	10:30 PM	11:00 PM	19.6215	3.25/.25	2.4568	8/14/2001	20
AN2-Tc-E1-20	Col 1	10	9:30	8/8/2001 11:00 PM	2.5	17.1991	11:00 PM	12:00 AM	22.0273	3.25/.25	4.8282	8/14/2001	20
AN2-Tc-E1-21	Col 1	11	10:30	8/9/2001 12:00 AM	5	17.1254	12:00 AM	1:00 AM	21.9612	3.25/.25	4.8358	8/14/2001	20
AN2-Tc-E1-22	Col 1	12	11:30	8/9/2001 1:00 AM	5	17.1144	1:00 AM	2:00 AM	21.9836	3.25/.25	4.8692	8/14/2001	20
AN2-Tc-E1-23	Col 1	13	12:30	8/9/2001 2:00 AM	5	17.1062	2:00 AM	3:00 AM	21.9569	3.25/.25	4.8507	8/14/2001	20
AN2-Tc-E1-24	Col 1	14	13:30	8/9/2001 3:00 AM	5	17.1589	3:00 AM	4:00 AM	21.9997	3.25/.25	4.8408	8/14/2001	20
AN2-Tc-E1-25	Col 1	15	14:30	8/9/2001 4:00 AM	5	17.1890	4:00 AM	5:00 AM	22.0240	3.25/.25	4.8350	8/14/2001	20
AN2-Tc-E1-26	Col 1	16	15:30	8/9/2001 5:00 AM	5	17.1751	5:00 AM	6:00 AM	22.0422	3.25/.25	4.8671	8/14/2001	20
AN2-Tc-E1-27	Col 1	17	16:30	8/9/2001 6:00 AM	5	17.2799	6:00 AM	7:00 AM	22.1560	3.25/.25	4.8761	8/15/2001	20
AN2-Tc-E1-28	Col 1	18	17:30	8/9/2001 7:00 AM	5	17.2745	7:00 AM	8:00 AM	22.1749	3.25/.25	4.9004	8/15/2001	20
AN2-Tc-E1-29	Col 1	19	18:30	8/9/2001 8:00 AM	5	17.2651	8:00 AM	9:00 AM	22.1971	3.25/.25	4.9320	8/10/2001	20
AN2-Tc-E1-30	Col 1	20	19:30	8/9/2001 9:00 AM	5	17.3086	9:00 AM	10:00 AM	22.1833	3.25/.25	4.8747	8/10/2001	20
AN2-Tc-E1-31	Col 1	21	20:30	8/9/2001 10:00 AM	5	17.3086	10:00 AM	11:00 AM	22.2413	3.25/.25	4.9327	8/10/2001	20
AN2-Tc-E1-32	Col 1	22	21:30	8/9/2001 11:00 AM	5	17.2724	11:00 AM	12:00 AM	22.1974	3.25/.25	4.9250	8/10/2001	20
AN2-Tc-E1-33	Col 1	23	22:30	8/9/2001 12:00 PM	5	17.2872	12:00 AM	1:00 PM	22.2289	3.25/.25	4.9417	8/10/2001	20
AN2-Tc-E1-34	Col 1	24	23:30	8/9/2001 1:00 PM	5	17.2115	1:00 PM	2:00 PM	22.0988	3.25/.25	4.8873	8/10/2001	20
AN2-Tc-E1-35	Col 1	25	24:30	8/9/2001 2:00 PM	5	17.2175	2:00 PM	3:00 PM	22.0906	3.25/.25	4.8731	8/10/2001	20
AN2-Tc-E1-36	Col 1	26	25:30	8/9/2001 3:00 PM	5	17.2366	3:00 PM	4:00 PM	22.0492	3.25/.25	4.8126	8/10/2001	20
AN2-Tc-E1-37	Col 1	27	26:30	8/9/2001 4:00 PM	5	17.3324	4:00 PM	5:00 PM	22.1822	3.25/.25	4.8498	8/10/2001	20

B.11

 indicates no information relevant to these cells  
 - indicates no data

Table 6. (continued) Data Collection for Tc Elution of Column 1 (lead column) in AN102/C104 Run @ 65C - Counting

Sample	Vol counted, mL	Net counts Tc-99	%C/Co Tc-99	Tc-99 Data mCi/mL	Dilution Factor	Tc-99 mCi/mL	Tc-99 % C/Co	uCi/mL	Volume, mL	uCi	uCi/mL	Volume, mL	uCi	
AN2-Tc-E1-18	0.1	6531	0.84	5.82E-05	81	4.70E-03	6.5%	2.30E-02	2.49	0.057	5.15E-03	2.49	0.013	
AN2-Tc-E1-19	0.1	12918	1.67	6.15E-05	78	4.81E-03	6.6%	4.56E-02	2.46	0.112	5.27E-03	2.46	0.013	
AN2-Tc-E1-20	0.1	2209	0.29	3.54E-05	78	2.77E-03	3.82%	7.79E-03	4.83	0.038	3.03E-03	4.83	0.015	
AN2-Tc-E1-21	0.1	519	0.07	1.66E-05	81	1.35E-03	1.86%	1.83E-03	4.84	0.009	1.48E-03	4.84	0.007	
AN2-Tc-E1-22	0.1	464	0.06	1.57E-05	78	1.22E-03	1.69%	1.64E-03	4.87	0.008	1.34E-03	4.87	0.007	
AN2-Tc-E1-23	0.1	435	0.06	1.49E-05	80	1.20E-03	1.65%	1.53E-03	4.85	0.007	1.31E-03	4.85	0.006	
AN2-Tc-E1-24	0.1	368	0.05	1.29E-05	79	1.01E-03	1.40%	1.30E-03	4.84	0.006	1.11E-03	4.84	0.005	
AN2-Tc-E1-25	0.1	290	0.04	9.39E-06	88	8.26E-04	1.14%	1.02E-03	4.84	0.005	9.05E-04	4.84	0.004	
AN2-Tc-E1-26	0.1	218	0.03	5.99E-06	80	4.80E-04	0.66%	7.69E-04	4.87	0.004	5.26E-04	4.87	0.003	
AN2-Tc-E1-27	0.1	204	0.03	3.48E-06	80	2.78E-04	0.38%	7.20E-04	4.88	0.004	3.04E-04	4.88	0.001	
AN2-Tc-E1-28	0.1	146	0.02	1.89E-06	79	1.49E-04	0.21%	5.15E-04	4.90	0.003	1.63E-04	4.90	0.001	
AN2-Tc-E1-29	0.1	129.9	0.02	1.11E-06	83	9.22E-05	0.13%	4.58E-04	4.93	0.002	1.01E-04	4.93	0.000	
AN2-Tc-E1-30	0.1	117	0.02	5.91E-07	78	4.59E-05	0.06%	4.13E-04	4.87	0.002	5.03E-05	4.87	0.000	
AN2-Tc-E1-31	0.1	104	0.01	3.91E-07	91	3.57E-05	0.05%	3.67E-04	4.93	0.002	3.91E-05	4.93	0.000	
AN2-Tc-E1-32	0.1	99	0.01	3.16E-07	80	2.52E-05	0.03%	3.49E-04	4.93	0.002	2.76E-05	4.93	0.000	
AN2-Tc-E1-33	0.1	95	0.01	1.82E-07	80	1.45E-05	0.02%	3.35E-04	4.94	0.002	1.59E-05	4.94	0.000	
AN2-Tc-E1-34	0.1	90	0.01	2.28E-07	82	1.87E-05	0.03%	3.17E-04	4.89	0.002	2.05E-05	4.89	0.000	
AN2-Tc-E1-35	0.1	86	0.01	1.99E-07	82	1.62E-05	0.02%	3.03E-04	4.87	0.001	1.78E-05	4.87	0.000	
AN2-Tc-E1-36	0.1	86.39	0.01	1.66E-07	78	1.30E-05	0.02%	3.05E-04	4.81	0.001	1.43E-05	4.81	0.000	
AN2-Tc-E1-37	0.1	84.27	0.01	1.65E-07	79	1.30E-05	0.02%	2.97E-04	4.85	0.001	1.43E-05	4.85	0.000	
Sum									132.68	21.98	uCi	132.7	25.13	uCi
									27 BV	1.30	mg		1.48	mg



indicates no information relevant to these cells

indicates no data

B.12

**Regeneration**

Feed: 0.25M NaOH  
 Resin: SuperLig 639, batch #010227CTC-9-23  
 Tracer: none used  
 Bed volume, Col 1 = 5.0 mL (3.3 cm)  
 Bed volume, Col 2 = 5.0 mL (3.4 cm)  
 Bed volumes diverted to waste = none  
 Feed analyses: Tc-99, mCi/mL  
 Flow Rate: 5 mL/hr or 1 CV/hr  
 Start Date and Time: 8/17/2001 11:59 AM  
 End Date and Time: 8/17/2001 1:51 PM

Table 7. Data Collection for Regeneration of Column 1 (lead column) in AN102/C104 Run

Sample	Source	# column volumes	Elapsed Time	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial + Cap Mass (g)	Bed Height Col 1 / Glass Wool Ht	Sample Mass (g)
AN2-Tc-Regen-1	Feed bot.	4	4 hrs	8/17/2001 11:59	20	15.0418	11:59 AM	1:51 PM	38.7876	3.2/0.3	23.7458

B.13

density 0.25 M NaOH	1.014	g/mL	(used in spreadsheet calculations)
Tc-99 initial feed	0.0745	uCi/mL	(used in spreadsheet calculations)
Tc-99 initial feed (mass balance)	0.0794712	uCi/mL	(used in spreadsheet calculations)

Sample	Tc-99 Data mCi/mL	Dilution Factor	Tc-99 mCi/mL	Tc-99 % C/Co	uCi/mL	Volume, mL	uCi	uCi/mL	Volume, mL	uCi
AN2-Tc-Regen-1	5.59E-07	76	<4.27E-05	< 0.059%	<sup>95m</sup> TcO <sub>4</sub> Mass Balance		Tc-99 Mass Balance			
					no data		4.68E-05	23.42	< 0.0011	
		indicates no information relevant to these cells								
		-- indicates no data								

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