

Small Column Testing of SuperLig[®] 639 for Removing ⁹⁹Tc from Hanford Tank Waste Envelope B (Tank 241-AZ-101)

I. E. Burgeson
D. L. Blanchard Jr.
J. R. Deschane

March 2004

Prepared for Bechtel National, Inc.
under Contract 24590-101-TSA-W000-0004

LEGAL NOTICE

This report was prepared by Battelle Memorial Institute (Battelle) as an account of sponsored research activities. Neither Client nor Battelle nor any person acting on behalf of either:

MAKES ANY WARRANTY OR REPRESENTATION, EXPRESS OR IMPLIED, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, process, or composition disclosed in this report may not infringe privately owned rights; or

Assumes any liabilities with respect to the use of, or for damages resulting from the use of, any information, apparatus, process, or composition disclosed in this report. References herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by Battelle. The views and opinions of authors expressed herein do not necessarily state or reflect those of Battelle.

**Small Column Testing of SuperLig[®] 639 for
Removing ⁹⁹Tc from Hanford Tank Waste
Envelope B (Tank 241-AZ-101)**

I. E. Burgeson
D. L. Blanchard Jr.
J. R. Deschane

Li HA for W.L. Tamosaitis
3/8/04

March 2004

**ACCEPTED FOR
WTP PROJECT USE**

Test specification: 24590-PTF-TSP-RT-01-002, Rev 1
Test plan: TP-RPP-WTP-111, Rev. 0
Test exceptions: None
R&T focus area: Pretreatment
Test Scoping Statement(s): B-45

Battelle—Pacific Northwest Division
Richland, Washington 99352

COMPLETENESS OF TESTING

This report describes the results of work and testing specified by Test Specification 24590-PTF-TSP-RT-01-002, Rev 1 and Test Plan TP-RPP-WTP-111, 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

3/1/04
Date

History Sheet

History Sheet			
Rev	Date	Reason for revision	Revised by
0	January 2003	New Document	IE Burgeson
1	February 2004	Noted higher uncertainty in the total ⁹⁹ Tc values in Tables 3.1, 3.2, 3.3, 3.6, and 3.7 as footnotes.	IE Burgeson

Summary

The River Protection Project-Waste Treatment Plant (RPP-WTP) baseline process for ^{99}Tc removal from Hanford tank waste is ion exchange. The current flowsheet includes the use of TcO_4^- -selective sorbent SuperLig[®] 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.

This report documents the results of dual-column testing of the SL-639 sorbent with waste from Tank 241-AZ-101 (AZ-101) for Bechtel National, Inc. in accordance with Test Specification 24590-PTF-TSP-RT-01-002, Rev. 1 (Toth 2002). 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-45, 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, including loading, feed displacement, water rinse, elution, and resin regeneration
- investigate the potential for resin and/or column fouling
- demonstrate the ^{99}Tc decontamination of an Envelope B material (Tank AZ-101); remove at least 98% of technetium within 250 bed volumes (BVs) of processed feed
- elute technetium from the resin to a target technetium $C/C_0 < 1\%$ within 22 BV of eluant
- provide a technetium-decontaminated sample for downstream process testing (i.e., LAW melter-feed testing and vitrification)
- provide batch-distribution measurements on actual tank-waste feed

Conduct of Testing

This report documents the results of dual-column testing of the SL-639 sorbent with AZ-101 feed 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 three previous technetium removal tests: AW-101 simulant, AP-101 tank waste feed, and AN-102/C-104 blended feed. The AZ-101 supernate was processed through the cross-flow filtration unit and processed for Cs removal by ion exchange using SuperLig[®] 644 before technetium removal. Approximately 1.6 L of the prepared waste feed was processed for technetium removal; this volume correlates to ~318 BVs of feed. All ion exchange process

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

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

Results and Performance Against Objectives

After processing 318 BVs of feed (1590 mL), the ⁹⁹Tc lead and lag column final breakthroughs were 31% and 0.1%, respectively. The initial ⁹⁹Tc decontamination factors (DFs) for the feed passing through the first column and the whole column system (derived from the ⁹⁹Tc concentration in the first sample from each column) were 980 and 1530, respectively, indicating that ≥ 99.9% of the technetium was removed from both the initial lead and lag column samples. The final DF of the effluent was 205, as measured by the concentration of ⁹⁹Tc present in the initial and processed feed. The ⁹⁹Tc concentration in the effluent composite was 1.83 μCi/L, providing an overall ⁹⁹Tc removal of 99.5%, which meets the success criteria of ≥ 98% removal of technetium. This performance exceeds the design basis, which is to remove 98% of the technetium within 250 BV of processed feed. It should be noted that the NO₃/Tc ratio was nearly an order of magnitude lower in this tank waste feed, 3.80E+03, than in the previous two tank waste feeds tested, AP-101 and AN-102/C-104 (Burgeson et al. 2002 and Burgeson et al. 2003).

Both columns were monitored during loading; however, only the lead column was eluted (distilled and deionized [DI] water at 65°C). The peak ⁹⁹Tc concentration of the eluate was 81 times the ⁹⁹Tc concentration in the feed and was found in the 2nd BV. The elution dropped slightly after this and then leveled out until the 8th BV. At this point, the ⁹⁹Tc concentration dropped quickly: 17 BVs of eluant were required for the ⁹⁹Tc concentration to drop below C/C₀ = 0.01. The ⁹⁹Tc concentration of the composited eluate was 4578 μCi/L, which correlates to a C/C₀ of 12.2.

The column distribution value, λ, approximately represents the effective capacity of the SL-639 resin when kinetics are not a significant factor. It represents the number of BVs processed when the concentration of pertechnetate reaches 50% of the feed concentration (C/C₀=0.5). Based upon the batch-distribution measurements and resin-bed density (K_d x resin density), it was expected that ~460 BVs would be processed before reaching 50% breakthrough. This value compares adequately with the column λ value of ~360 BVs, which was extrapolated from ~31% breakthrough (see Table S1).

Table S1. Summary of Observed and Predicted Column Performance for AZ-101 Feed

Flowrate, BV/h	Extrapolated ⁹⁹ Tc Column λ Value ^(a)	Composite ⁹⁹ Tc Decontamination Factor ^(b)	⁹⁹ Tc K _d , mL/g	λ Value ^(c)
3.0	360	205	920	460
(a) Extrapolated from final breakthrough at 318 BV(31%) to 50% C/C ₀ . (b) The decontamination factor is determined by dividing the final effluent technetium concentration by the initial feed concentration. (c) Calculated using the batch contact ⁹⁹ Tc K _d value, 920 mL/g and the resin bulk density (0.50 g/mL).				

Quality Requirements

Battelle—Pacific Northwest Division (PNWD) 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 (homogenization and subsampling) 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 (solids removal, Cs and Tc ion exchange, and solution analyses) 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 earlier experiments was not impacted by the change in requirements.

Battelle—Pacific Northwest Division addressed verification activities by conducting an independent technical review of the final data report in accordance with Procedure QA-RPP-WTP-604. The 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
AV	apparatus volume
BNI	Bechtel National, Inc.
BV	bed volume
CUF	crossflow filtration system (cell unit filter)
DF	decontamination factor
DI	distilled and deionized
GEA	gamma energy analysis
HLRF	High-Level Radiochemistry Facility
IC	ion chromatography
ICP-AES	inductively coupled plasma/atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
ID	inner diameter
λ	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
RPL	Radiochemical Processing Laboratory
RPP	River Protection Project
SAL	Shielded Analytical Laboratory
SOW	statement of work
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

Contents

Summary	iii
Terms, Symbols, and Abbreviations	vii
1.0 Introduction	1.1
2.0 Experimental	2.1
2.1 AZ-101 Sample History	2.1
2.2 Feed Preparation.....	2.1
2.3 SL-639 Resin and Bed Preparation	2.2
2.4 History of Technetium Processing with Resin Bed.....	2.3
2.5 Batch-Distribution Measurement	2.4
2.6 Tc Removal Column System.....	2.5
2.7 Column Ion Exchange Test Procedure and Conditions.....	2.5
3.0 Results and Discussion.....	3.1
3.1 Feed Composition	3.1
3.2 Batch Distribution	3.2
3.3 AZ-101 Column Test – Sample Loading	3.5
3.4 AZ-101 Column Test – Feed Displacement and Column Rinse	3.7
3.5 AZ-101 Column Test—Elution and Resin Regeneration.....	3.9
3.6 AZ-101 Column Test—Elution Composite and Effluent Composite Analysis	3.10
3.7 Comparison of AZ-101 Initial Feed and Effluent Composite Composition	3.14
3.8 Technetium Recovery	3.15
3.8.1 System Recovery Using Composite Solution ⁹⁹ Tc Data.....	3.15
3.8.2 Recovery for Lead Column Loading and Elution Using Individual ⁹⁹ Tc Sample Data.....	3.16
3.8.3 Estimate of Technetium Retained on Lead and Lag Columns.....	3.17

3.9 Summary of Objectives and System Performance	3.18
4.0 Conclusions	4.1
5.0 References	5.1
Appendix A: Sample Identification	A.1
Appendix B: Column Run Spreadsheets.....	B.1

Figures

2.1. Schematic Representation of Column System for Removing Technetium.....	2.5
3.1. ^{99}Tc - and $^{95\text{m}}\text{TcO}_4^-$ -Derived K_d Values.....	3.4
3.2. ^{99}Tc Batch Contact Isotherm.....	3.5
3.3. AZ-101 Technetium Loading Breakthrough Curves, Lead and Lag Columns	3.6
3.4. Tc and $^{95\text{m}}\text{TcO}_4^-$ C/C_0 for Lead Column Feed Displacement and Rinse	3.8
3.5. Elution of Technetium from Lead Column.....	3.9

Tables

S1. Summary of Observed and Predicted Column Performance for AZ-101 Feed	iv
Table 2.1. Sieving Data for SL-639 Batch Number 010227CTC-9-23	2.2
Table 2.2. Summary of SL-639 Resin and Column Parameters	2.3
Table 2.3. Experimental Conditions of AZ-101 Supernate Small-Scale ^(a) Ion Exchange	2.6
Table 2.4. Sampling Interval and Analyses of Small-Scale Ion Exchange Column Process Stages	2.7
Table 3.1. Composition of AZ-101 Supernate (Envelope B Feed)	3.1
Table 3.2. Selected Composition of AZ-101 Supernate (Envelope B Feed)	3.2
Table 3.3. AZ-101 Technetium K _d Values—Average of Duplicate Measurements	3.3
Table 3.4. Composition of Column Regenerate after AZ-101 Column Run	3.10
Table 3.5. Analysis Results for AZ-101 Technetium Eluate Composite	3.11
Table 3.6. Composition of AZ-101 Tc IX Effluent Composite	3.13
Table 3.7. Comparison of Initial Feed and Effluent Composite Composition.....	3.14
Table 3.8. Recovery for Technetium During Processing Using Composite Solution Data	3.15
Table 3.9. Recovery for Lead Column Loading and Elution of Technetium Using Sum of Individual Sample ⁹⁹ Tc Analyses.....	3.17
Table 3.10. Estimate of Technetium Retained on Lead and Lag Columns During Processing of AZ-101	3.18

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^(a) precipitation,^(b) 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. The current flowsheet for technetium ion exchange removal includes the use of Superlig[®] 639^(c) (SL-639) for removing the pertechnetate form of technetium-99 (⁹⁹Tc) from the aqueous fraction of the waste.

This report describes the results of small-column testing of the SL-639 sorbent with 241-AZ-101 tank waste supernate.^(d) (The 241 prefix, common to all Hanford tanks, will not be used hereafter). Approximately 1.6 L of tank-waste feed (at 4.8 M Na) was processed through dual small-scale ion exchange columns. This tank-waste feed underwent solids removal by cross-flow filtration (Geeting et al. 2002), and then was vacuum filtered and processed for Cs removal by ion exchange using SuperLig[®] 644 (Fiskum et al. 2002) before technetium ion exchange. The removal goal for the AZ-101 feed is to reduce the total Tc concentration by at least 98% after processing ≥ 250 bed volumes (BVs) of solution.

The objectives of this work were to

- demonstrate the effectiveness of all SL-639 process steps, including loading, feed displacement, water rinse, elution, and resin regeneration
- investigate the potential for resin and/or column fouling
- demonstrate the ⁹⁹Tc decontamination of an Envelope B material (Tank AZ-101); remove at least 98% of technetium within 250 bed volumes (BVs) of processed feed
- elute technetium from the resin to a target technetium $C/C_0 < 1\%$, within 22 BV of eluant
- provide a technetium-decontaminated sample for downstream process testing (i.e., LAW melter-feed testing and vitrification)
- provide batch-distribution measurements on actual tank-waste feed.

(a) TRU = transuranic.

(b) Strontium/TRU precipitation is only performed with Envelope C tank-waste feed. Since AZ-101 is an Envelope B feed, Sr/TRU processing was not performed.

(c) The SL-639 material is an organic resin that has been developed and supplied by IBC Technologies, Inc., American Fork, Utah.

(d) Work performed according to test plan TP-RPP-WTP-111 Rev 0 *Tank 241-AZ-101 and 241-AZ-102 Waste Sample Ion Exchange Testing* and test instruction TI-RPP-WTP-172 Rev 0 *AZ-101 Tc Column Run Test Instructions*.

This report discusses the experimental conditions for the column ion exchange test procedure. Work was performed in accordance with the test specification 24590-PTF-TSP-RT-01-002,^(e) Rev. 1, and test plan TP-RPP-WTP-111, Rev. 0. The results of the feed composition, the batch distribution, the AZ-101 column test, the comparison of the AZ-101 initial feed and effluent composite composition, and the technetium mass balance are presented.

(e) 24590-PTF-TSP-RT-01-002, "Tank 241-AZ-101 and 241-AZ-102 Ion Exchange Test Specification," JR Toth, Rev. 1, Pretreatment, R&T, BNI/WGI, October 2001.

2.0 Experimental

This section describes the sampling history for the AZ-101 core samples used in this testing, the feed preparation, and the SL-639 resin and bed preparation. It also covers the history of technetium processing with a resin bed, the batch-distribution measurements, and the column system for removing Tc. The column ion exchange test procedure and conditions are described.

2.1 AZ-101 Sample History

Hanford site personnel core sampled Tank AZ-101 from Riser 59 in August 2000. The core sampling event collected a full core with eighteen 19-in. segments to obtain a full vertical profile of the tank waste. To collect enough solids to fulfill the ICD-23^(a) sample requirements, an additional two segments of the sludge were collected from the bottom 39 in. of the tank. The AZ-101 core sample was obtained after tank mixing pumps were run in the tank in April and May of 2000.^(b) Data collected at the time of the mixer pump testing demonstrated that most of the sludge in the tank was suspended, and the sludge became well homogenized both vertically and horizontally. The core was extruded, inspected, and stored at 222-S until being sent to Battelle—Pacific Northwest Division (PNWD) in May 2001 for complete characterization and process testing.

2.2 Feed Preparation

Fifteen samples of the AZ-101 supernate and solids, a total of 6759 g, were received in the Shielded Analytical Laboratory (SAL) at the Radiochemical Processing Laboratory (RPL) in May 2001. These 15 bottles contained the full core segment plus two retake segments of solids collected in the August 2000 sampling event. All of the tank-waste material was composited into a single container, thoroughly mixed, and sub-sampled into 18 containers (Urie et al. 2002). Ten of these homogenized aliquots were set aside for analytical and physical characterization; the results are reported in Urie et al. (2002). The other eight aliquots and material remaining from the three physical testing samples were delivered to the High-Level Radiochemistry Facility (HLRF) for cross-flow filtration tests. A total of 4313 g of slurry at 7.5 wt% solids was processed by crossflow filtration (Geeting et al. 2002). After processing by the cross flow filtration system, or CUF, approximately 2.5 L of the supernatant was delivered for cesium ion exchange processing. Cesium ion exchange was performed at two different loading flowrates, 1.5 BV/h and 2.7 BV/h (Fiskum et al. 2002). The effluent from the second cesium ion exchange processing contained significant amounts of ¹³⁷Cs. Thus, this effluent (~850 mL) was processed for additional cesium ion exchange. The effluent was processed through a 10-mL column of IE-911 resin for cesium removal. This resulting solution was combined with the effluent from the first cesium column test (~820 mL), providing 1672 mL of supernatant for technetium ion exchange processing.

After combining the two supernatants from cesium ion exchange processing, 138 μCi of ^{95m}Tc ($t_{1/2} = 61$ days, decays to stable ⁹⁵Mo) was added to the AZ-101 supernate to act as a tracer for monitoring the removal of pertechnetate (TcO_4^-). The ^{95m}Tc provides a strong gamma emission peak at 205 KeV.

(a) 24590-WTP-ICD-MG-01-023 , Rev 1, ICD-23 Interface Control Document for Waste Treatability Samples, August 2002.

(b) Preliminary Test 241-AZ-101 Mixer Pump Test, RPP-6548, Rev. 0., July 2000.

The radiotracer was added using a small volume (220 μL) of 0.63-mCi/mL ammonium pertechnetate ($\text{NH}_4^{95\text{m}}\text{TcO}_4$) prepared in 0.05 M ammonium hydroxide. The mole ratio of ^{99}Tc to $^{95\text{m}}\text{Tc}$ was 5.8×10^9 , demonstrating that the contribution of the tracer to the total technetium is negligible. A 2-mL aliquot of the spiked feed provided 55,500 counts (peak area) in 5 min.

2.3 SL-639 Resin and Bed Preparation

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

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

Sieve Number	Particle Size, μm	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
Totals		9.9879	100.0

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 ± 0.05 mL, slurried, and quantitatively transferred to the ion exchange column.

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) can range from 20 to 30:1 based upon the sieve data, 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 1 cm of quartz glass wool to prevent floating of the resin during contact with the AZ-101 supernate.

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 ± 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 as-received exchanger in a 5-mL graduated cylinder (0.50 g/mL).

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 was estimated to be 2.5 g. Table 2.2 summarizes these values. The columns were prepared for AZ-101 loading by flushing them 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.

Table 2.2. Summary of SL-639 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 ^(a)	Column Diameter to Resin Diameter Ratio	20–30
Resin Diameter	460-670 μm ^(b)	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.			

2.4 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 ⁹⁹Tc-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 μCi/L technetium). Elution was performed for each column until the C/C₀ was <0.01.

An actual tank-waste run was then performed using tank-waste supernate from AP-101 (Burgeson et al. 2002). 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 μCi/L technetium), resulting in a breakthrough of ~5% on the lead column and ~0.2% on the lag column. The lead column was eluted until the C/C₀ was <0.01.

A second actual tank waste run was then performed using AN-102 supernate blended with C-104 solid leach and permeate solutions (Burgeson et al. 2003). 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. During processing of the blended AN-102/C-104 tank-waste feed, the lead column only was rinsed, eluted, and regenerated. The system processed 136 BV of feed (680 mL at 80 μCi/L technetium, 40 μCi/L pertechnetate), resulting in a breakthrough of ~50% technetium due to the presence of high levels of non-pertechnetate, on both the lead and lag column. The lead column was eluted until the C/C₀ was <0.01.

Before processing the AZ-101 feed, the lead column from the AN-102/C-104 column run was switched to the lag-column position. This lag column had not been eluted and exhibited a significant pertechnetate breakthrough (14%) while processing the AN-102/C-104 blended feed. Both the lead and lag columns were prepared for the AZ-101 loading by flushing with 4 BV (20 mL) of 0.25 M NaOH. This was done primarily to assure that the bed was sufficiently settled before starting the column run.

2.5 Batch-Distribution Measurement

Some of the AZ-101 supernatant delivered for Tc ion exchange was used to determine technetium batch-distribution coefficients (K_d values) before running the column. The AZ-101 feed was processed to remove cesium before using the material for technetium batch-distribution measurements. The resin was contacted with AZ-101 feed spiked with approximately 17 μCi of $^{95\text{m}}\text{TcO}_4^-$. The $^{95\text{m}}\text{Tc}$ was used as a tracer for following the removal of pertechnetate (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}Tc to $^{95\text{m}}\text{Tc} = 3.8 \times 10^9$); therefore, the added tracer was not expected to affect the batch-distribution values. The $^{95\text{m}}\text{Tc}$ spiked AZ-101 supernatant was subdivided into three aliquots. The concentration of technetium in the AZ-101 supernatant was approximately 375 $\mu\text{Ci/L}$. The total technetium concentration was increased in three aliquots to approximately 600, 2000, and 3200 $\mu\text{Ci/L}$ using a stock solution of ^{99}Tc (11, 67, and 122 μL 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. Ten milliliters of AZ-101 supernatant were contacted with 0.10 g of exchanger. The exchanger mass was determined to an accuracy of ± 0.0001 g. The waste volume was transferred by pipette, and the actual volume was determined from the mass difference (± 0.0002 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 22°C and 23°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- μm nylon filter. The total ^{99}Tc concentration in the AZ-101 feed was determined by inductively coupled plasma-mass spectroscopy (ICP-MS), which in turn was used to calculate the ^{99}Tc K_d s.

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

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

where

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

V = the volume of the liquid sample (mL)

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

F = the mass of a sample of dried resin divided by its mass before drying. The resin was sampled for F-factor determination at the same time resin samples for the batch contacts were measured to minimize mass changes due to changes in atmospheric humidity.

2.6 Tc Removal Column System

Figure 2.1 is 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 $\frac{1}{8}$ -in. OD and $\frac{1}{16}$ -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, WI) was used to deliver feed to the columns. The flowrate was controlled with a metering pump head (Fluid Metering, Inc., Oyster Bay, NY) 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 (AV) was measured to be 20 mL (4 BV) with approximately 10 mL (2 BV) volume holdup from the feed line to the lead column.

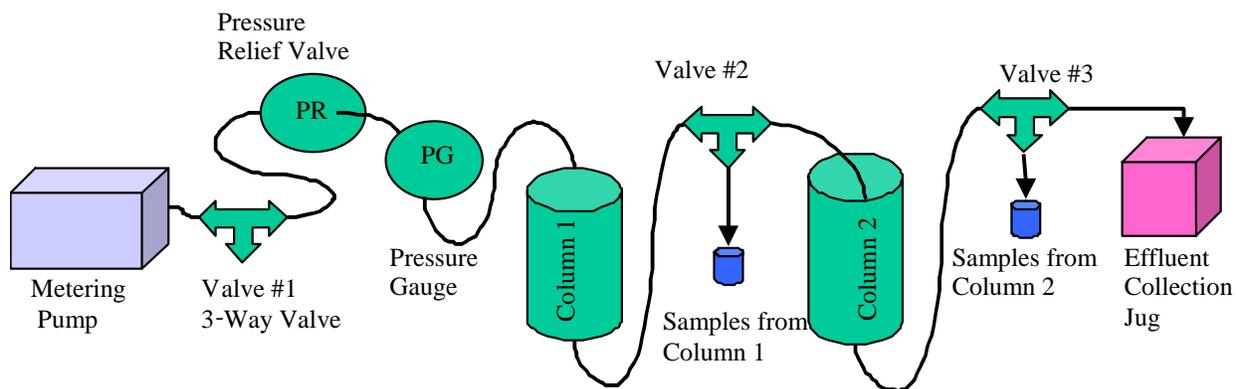


Figure 2.1. Schematic Representation of Column System for Removing Technetium

2.7 Column Ion Exchange Test Procedure and Conditions

Table 2.3 shows the experimental conditions for each process step of the column ion exchange test. 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. Target Experimental Conditions of AZ-101 Supernate Small-Scale^(a) 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 M NaOH	20 (4)	15 (3)	1.3 (80)
Lead and Lag	Loading	AZ-101	1590(318)	15 (3)	105
Lead and Lag	Feed Displacement	0.10 M NaOH ^(b)	40 (2 AV)	15 (3)	2.7 (160)
Lead and Lag	Rinse	DI water	10 (2) (½ AV)	15 (3)	0.7 (40)
Lead	Elution	DI @ 65°C	200 (40)	5 (1)	40 (2400)
Lead	Regeneration	0.25 M NaOH	20 (4)	5 (1)	4 (240)

(a) The BV for each column was 5.0 ± 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.

The adjustable plungers at the top of each column were used to minimize the volume of solution above each of the resin beds. The resin beds were gently packed with approximately 1 cm of quartz glass wool to prevent the resin from floating during contact with the AZ-101 feed. 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 23 to 24°C. A recirculating water bath was used to circulate heated water through the column jacket during elution.

Table 2.4 shows the sampling-and-analysis protocol. The $^{95m}\text{TcO}_4^- C/C_0$ was determined in all samples by counting the ^{95m}Tc gamma emission at 205 KeV with a portable gamma spectrometer equipped with a Ge flat crystal detector. Each of the analytical samples collected during the loading stage was approximately 2 mL. The analytical samples were weighed to within ± 0.0001 g and counted for ^{95m}Tc . The counting data were normalized to 2.0 mL of solution using the density of the AZ-101 feed and were compared to the counts of 2.0 mL of the AZ-101 feed solution. All other analytical samples were collected in 5 mL or larger increments; thus, 2-mL sample aliquots were counted. The $\text{TcO}_4^- C/C_0$ ratios were determined by taking the ratio of the peak area of the original feed with the peak area of the loading and the effluent samples. The original feed sample was recounted periodically to minimize the effects of the ^{95m}Tc decay on the C/C_0 calculations. This method allowed near real-time monitoring of the loading and elution steps. After completing the loading and elution cycles, the samples were recounted over a 2-day period to provide more consistent counting data in samples with low levels of technetium (i.e., $^{95m}\text{TcO}_4^-$).

The total technetium (^{99}Tc) levels were determined by ICP-MS in selected samples encompassing the entire processing scheme after the run was concluded. 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	Analytical Sample Vol (mL)	Analyses ^(a)
Original Feed	--	--	--	ICP-MS, ICP-AES, OH ⁻ , IC ^(c) anions and pertechnetate
Column Prep.	--	--	--	--
Loading	Every 10 BV	Every 20 BV	2	ICP-MS
Feed Displacement	--	Every 1 BV	5	ICP-AES, ICP-MS, OH ⁻
DI water rinse	2 BV	--	10	ICP-AES, ICP-MS, OH ⁻
Elution	Every ½ BV for 10 BV then every BV ^(b)	--	Every 2.5 BV for 10 BV then every 5 BV ^(b)	ICP-MS
Regeneration	--	1 composite	10	ICP-AES, ICP-MS, OH ⁻
End of Run Composite Samples				
Effluent	--	1 composite	20	ICP-AES, IC ^(c) , TOC ^(d) TIC, ^(e) IC, OH ⁻ , ICP-MS, U, Radchem ^(f)
Eluant	1 composite	--	20	ICP-AES, TIC, TOC, IC, OH ⁻ ICP-MS, U, Radchem ^(g)
<p>(a) The composite samples were analyzed for both the ion exchange analytes and the high level waste and LAW vitrification analytes of interest. This table identifies only the Tc ion exchange analytes of interest.</p> <p>(b) Initial samples (first 10 BV) were taken in ½ BV increments, resulting in 2.5-mL samples.</p> <p>(c) IC = ion chromatography</p> <p>(d) TOC = total organic carbon</p> <p>(e) TIC = total inorganic carbon</p> <p>(f) Radiochemical analyses were separation/alpha energy analysis (AEA), extended counting gamma energy analysis (GEA), and pertechnetate speciation.</p> <p>(g) Radiochemical analyses were ⁹⁰Sr and GEA.</p> <p>-- Indicates data cell is not applicable</p>				

3.0 Results and Discussion

This section shows the composition of the feed and the batch distribution. The sample loading, the feed displacement and column rinse, and the elution and resin regeneration are given for the AZ-101 column test. The elution and effluent composite analysis is provided, and the composition of the AZ-101 initial feed and the effluent composite are compared. Data regarding technetium recovery are provided, and the objectives and system performance are summarized.

3.1 Feed Composition

Table 3.1 shows the composition of the AZ-101 supernate used for the batch contacts and column ion exchange. Table 3.1 contains all of the analytes observed by each analytical method: ICP-AES, titration, ICP-MS, IC anions, and GEA. The concentration of technetium present as pertechnetate was determined by radiochemical separation. The total anion normality, 4.55 N, is larger than the total cation normality, 4.35 N. This difference is approximately 5 percent and is within analytical and experimental uncertainties.

Table 3.1. Composition of AZ-101 Supernate (Envelope B Feed)

Analyte	Analytical Result	Method	Analyte	Analytical Result	Method
Radiochemical Results in $\mu\text{Ci/mL}$					
$^{99}\text{Tc}^{(a)}$	3.75E-01	ICP-MS	^{137}Cs	6.49E-02	GEA
TcO_4^-	3.78E-01	Sep/ Counting	^{154}Eu	< 6.0E-05	GEA
^{60}Co	< 4.0E-05	GEA	$^{239+240}\text{Pu}$	< 5.0E-05	Sep/ AEA
Inorganic Analysis Results in $\mu\text{g/mL}$					
Al	5,070	ICP-AES	Pb	[6]	ICP-AES
As	[12]	ICP-AES	Si	[76]	ICP-AES
B	17	ICP-AES	Sn	[40]	ICP-AES
Cr	541	ICP-AES	Ti	[2.5]	ICP-AES
Fe	< 0.5	ICP-AES	V	[1.2]	ICP-AES
K	3,640	ICP-AES	W	[52]	ICP-AES
Mo	81	ICP-AES	Zn	[1.6]	ICP-AES
Na	97,900	ICP-AES	Zr	[1.8]	ICP-AES
P	456	ICP-AES	U	0.0022	KPA
Inorganic Anion Results in $\mu\text{g/mL}$					
Cl	[270]	IC Anions	PO_4	1,600	IC Anions
F	2,000	IC Anions	SO_4	16,900	IC Anions
NO_2	62,800	IC Anions	oxalate	1,050	IC Anions
NO_3	52,600	IC Anions			
Physical Properties Units as Reported; <u>M</u> is moles/L					
Hydroxide	0.63 <u>M</u>	Titration	Density	1.229 g/mL	Gravimetric
[] Analyte was detected within 10 times the instrument detection limit and the uncertainty is estimated to exceed $\pm 15\%$.					
< Indicates the analyte was not observed above the instrument detection limit					
(a) Uncertainty, including standards bias, ranged from -10% to +30%					

The compositions of selected anions and cations as well as information important to technetium ion exchange are collected in Table 3.2.

Table 3.2. Selected Composition of AZ-101 Supernate (Envelope B Feed)

Analyte	Moles/Liter (M) (Normality)
Na ⁺	4.26 (4.26)
K ⁺	0.09 (0.09)
Total Cation (Normality)	4.35
AlO ₂ ⁻ (a)	0.19 (0.19)
Cl ⁻	0.004 (0.004)
CO ₃ ⁻² (b)	0.49 (0.97)
CrO ₄ ⁻² (a)	0.01 (0.02)
F ⁻	0.10 (0.10)
NO ₂ ⁻	1.37 (1.37)
NO ₃ ⁻	0.85 (0.85)
OH ⁻	0.63 (0.63)
PO ₄ ⁻³	0.017 (0.05)
SO ₄ ⁻²	0.18 (0.35)
Oxalate	0.01 (0.02)
Total Anion (Normality)	4.55
⁹⁹ Tc and Competing Ion Ratios	
⁹⁹ Tc, μCi/L (mg/L) (c)	375 (22.1)
⁹⁹ Tc as pertechnetate, μCi/L (mg/L)	378 (22.3)
NO ₃ ⁻ / ⁹⁹ Tc mole ratio	3.80E+03
CrO ₄ ⁻² / ⁹⁹ Tc mole ratio	47
Solution Density, g/mL	1.229
(a) Elemental Al and Cr were determined by ICP-AES. The anionic forms, AlO ₂ ⁻ and CrO ₄ ⁻² , 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%	

Table 3.2 shows that the NO₃/Tc ratio is quite low in this tank waste. The NO₃/Tc ratio was 3.65E+04 for the blended AN-102/C-104 tank waste feed (Burgeson et al. 2003) and 6.25E+04 for diluted AP-101 tank waste feed (Burgeson et al. 2002).

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 three higher concentrations of technetium. Table 3.3 shows the results of duplicate distribution-coefficient measurements. 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. 2003).

Table 3.3. AZ-101 Technetium K_d Values—Average of Duplicate Measurements

Sample	Feed Conditions	$^{95m}\text{TcO}_4^- K_d$, mL/g ^(a) (% Removal ^(b))	Total Tc K_d , mL/g ^(a) (% Removal ^(b))
AZ-101 Original	Na = 4.8 M ^{99}Tc = 362 $\mu\text{Ci/L}$ ^(c) (21.3 mg/L) NO_3/Tc , eq = 4.71E+04 SL-639 batch #010227CTC-9-23	820 (89%) RPD ^(d) = 8%	1180 (92%) RPD ^(d) = 8%
AZ-101 Spike 1	Na = 4.8 M ^{99}Tc = 606 $\mu\text{Ci/L}$ ^(c) (35.7 mg/L) NO_3/Tc , eq = 2.51E+04 SL-639 batch #010227CTC-9-23	740 (87%) RPD ^(d) = 7%	1080 (91%) RPD ^(d) = 11%
AZ-101 Spike 2	Na = 4.8 M ^{99}Tc = 1996 $\mu\text{Ci/L}$ ^(c) (118 mg/L) NO_3/Tc , eq = 8.33E+03 SL-639 batch #010227CTC-9-23	740 (87%) RPD ^(d) = 10%	1080 (91%) RPD ^(d) = 19%
AZ-101 Spike 3	Na = 4.8 M ^{99}Tc = 3180 $\mu\text{Ci/L}$ ^(c) (187 mg/L) NO_3/Tc , eq = 4.11E+03 SL-639 batch #010227CTC-9-23	600 (85%) RPD ^(d) = 1%	880 (89%) RPD ^(d) = 3%
<p>(a) The total Tc K_d is assumed to be equal to the ^{99}Tc K_d.</p> <p>(b) The % removal indicates the Tc removed from solution or retained on the resin and is determined by taking (Initial Tc in solution – Final Tc in solution)/Initial Tc in solution times 100.</p> <p>(c) Uncertainty, including standards bias, ranged from -10% to +30%</p> <p>(d) The RPD is the relative percent difference and is determined by taking (Initial K_d – Duplicate K_d)/Initial K_d times 100.</p>			

The distribution coefficients were determined by assessing the change in both the ^{99}Tc and $^{95m}\text{TcO}_4^-$ in solution and are reported as either ^{99}Tc or $^{95m}\text{TcO}_4^- K_d$ values. The ^{99}Tc concentration reflects the total technetium-solution concentration, which includes both pertechnetate and non-pertechnetate forms of technetium. The ^{99}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. Figure 3.1 shows a plot of the K_d s versus the equilibrium mole ratio of nitrate to technetium. It shows both the ^{99}Tc derived K_d values and the $^{95m}\text{TcO}_4^-$ values. The $^{95m}\text{TcO}_4^- K_d$ values track well with the ^{99}Tc K_d values, except that they are lower than the ^{99}Tc K_d values. The $^{95m}\text{TcO}_4^- K_d$ results are likely lower than the $^{99}\text{Tc}K_d$ values due to a counting problem with the portable gamma detector used to measure the $^{95m}\text{TcO}_4^-$ levels.

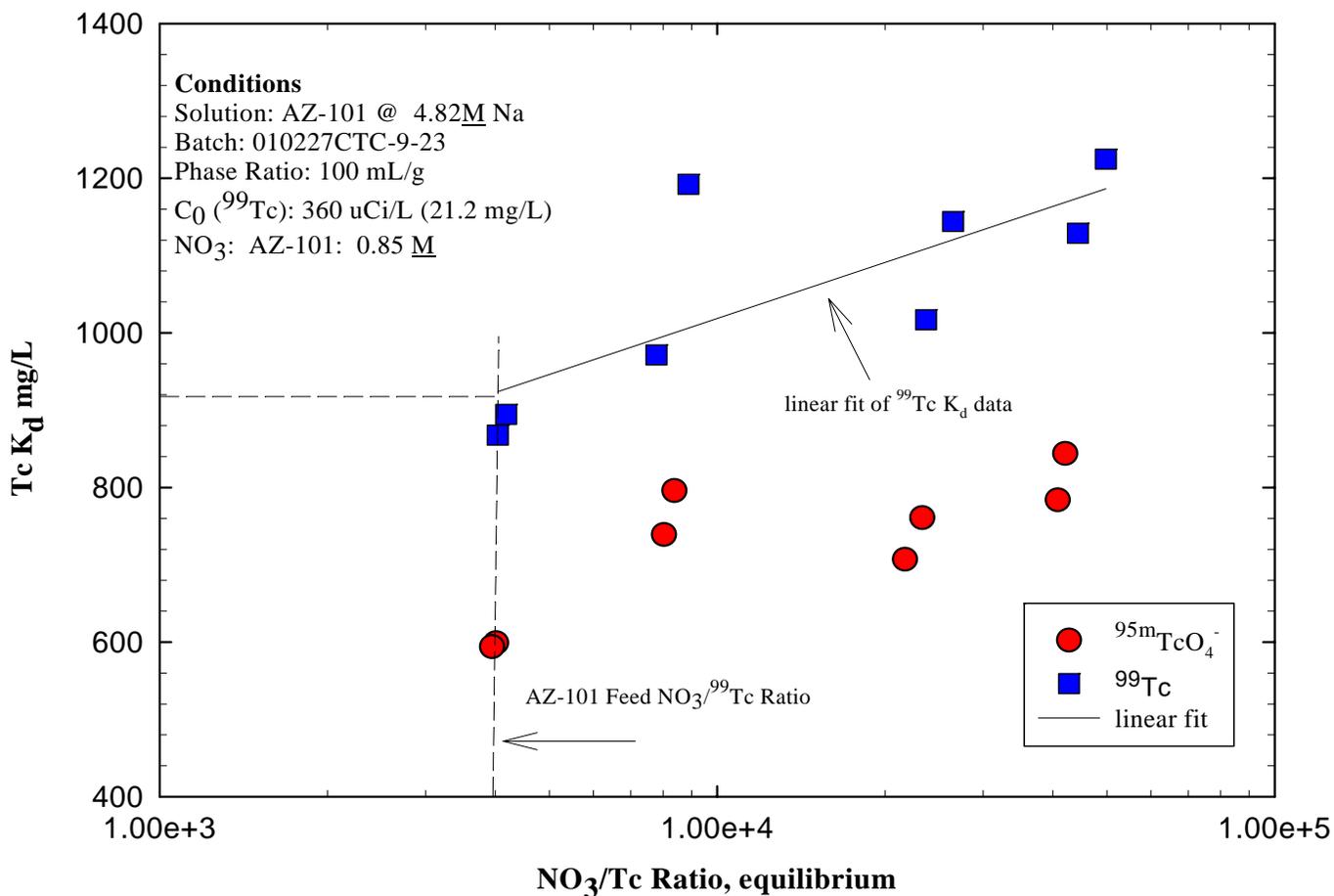


Figure 3.1. ⁹⁹Tc- and ^{95m}TcO₄⁻-Derived K_d Values

The original feed was spiked with ⁹⁹Tc in order to provide a range of NO₃⁻/Tc (initial) ratios that would bound the feed NO₃⁻/Tc ratio at equilibrium. However, the initial AZ-101 ⁹⁹Tc concentration was high enough that the specified spiking levels were too low. Thus, the K_d value at the original feed NO₃⁻/Tc ratio was extrapolated from a least squares modeling of the ⁹⁹Tc K_d data and the NO₃⁻/Tc ratios. The linear fit is shown in Figure 3.1. The NO₃⁻/⁹⁹Tc ratio for the AZ-101 initial feed conditions is shown in the figure with a vertical, dashed line. The corresponding ⁹⁹Tc K_d value is ~920 mL/g. This results in an estimated λ value (K_d x resin bed density) of ~460 BVs. As can be seen in Figure 3.1, the ⁹⁹Tc K_d values appear somewhat scattered and have a small slope. To determine if the resin was behaving in a predictable fashion, a K_d isotherm was prepared and is shown in Figure 3.2.

The data in Figure 3.2 are presented as the moles of analyte in solution at equilibrium versus moles of analyte present per gram of resin at equilibrium. As can be seen in Figure 3.2, this relationship is linear, indicating that the technetium is behaving in a predictable fashion. Additionally, the linear plot of this relationship has an R² value of 0.974. It appears that in the concentration range examined, a plateau in the isotherm had not yet been reached.

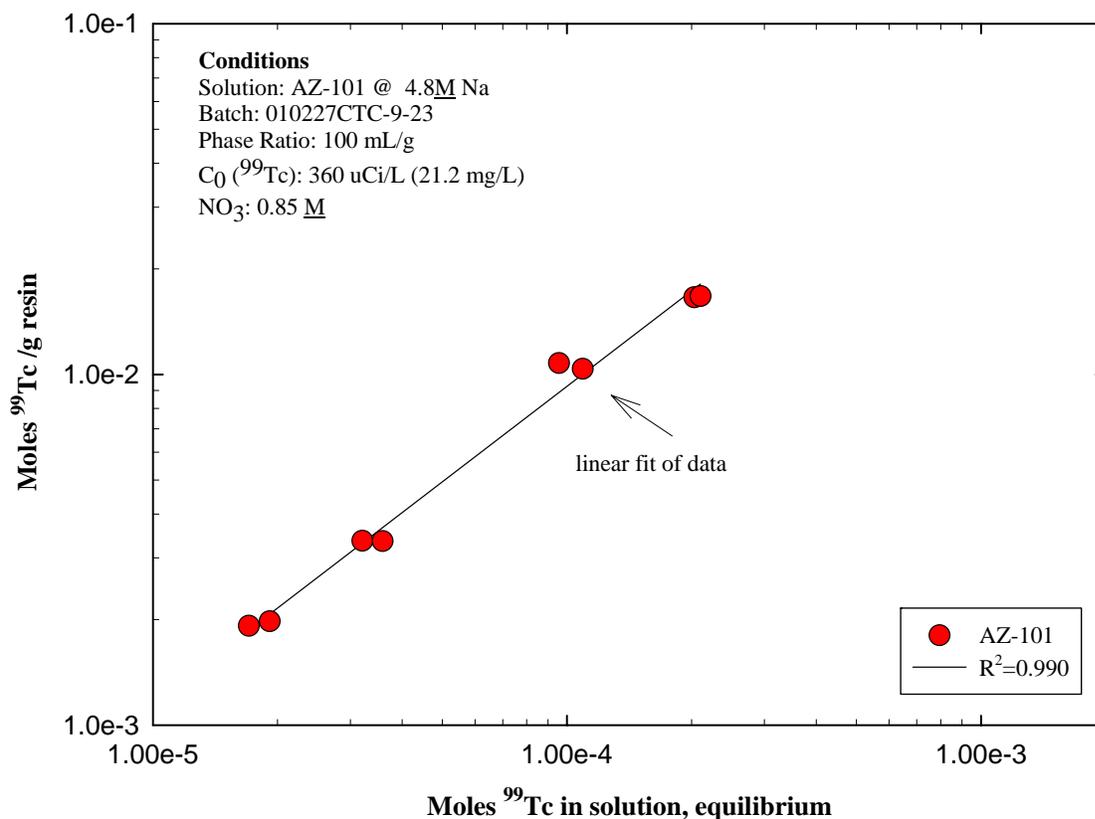


Figure 3.2. ^{99}Tc Batch Contact Isotherm

3.3 AZ-101 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. 2002) and AN-102/C-104 blended feed (Burgeson et al. 2003) as described in Section 2.6. Only the lead column was eluted during the AN-102/C-104 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 AZ-101 run was the non-eluted lag column from the AN-102/C-104 run.

Immediately before starting to load the AZ-101 feed, the system was flushed with fresh 0.25 M NaOH (20 mL at 15 mL/h). The AZ-101 feed was processed through the system beginning April 24th, 2002. The loading stage lasted for 105 h, in which time 318 BVs of AZ-101 feed were processed. The flowrate was determined at each sampling event and averaged approximately 15 mL/h (3.0 BV/h). There were no observed anomalies, i.e., fluidized bed, gas bubbles, changes in resin bed height or color, etc. The pressure remained below 2 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, 5, and 14 BVs of processed feed. After the fourteenth

BV of feed was processed, one analytical sample was collected every 10 BVs from the lead column. After the slope of the technetium breakthrough was clearly identified, the sampling frequency was decreased to every 20 BVs from the lead column (at 212 BV). An initial sample was collected from the lag column at 5 BVs of processed feed and then collected approximately every 20 BVs thereafter. After processing 245 BVs, the lag column sampling frequency was decreased to every 40 BVs.

The column loading was monitored using gamma emission spectrometry analysis of ^{95m}Tc , which reflects the concentration of total pertechnetate present in each sample. The ratio of the measured ^{95m}Tc passing through the lead and lag column with respect to the initial amount of ^{95m}Tc in the ion exchange feed (C/C_0) was calculated and is reported in Figure 3.3 for each sample. In addition, selected loading samples were analyzed for total technetium (^{99}Tc). Figure 3.3 also shows the ratio of the initial ^{99}Tc and the ^{99}Tc measured in these selected loading samples.

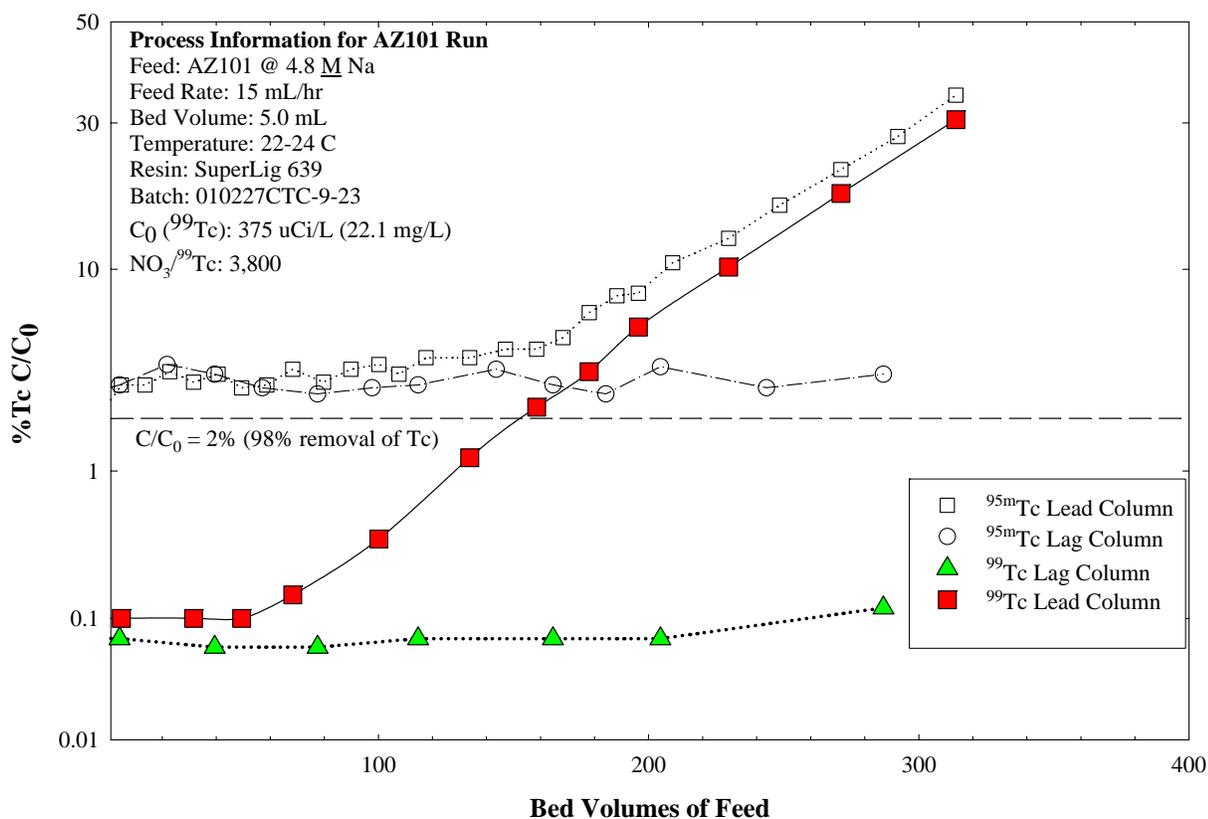


Figure 3.3. AZ-101 Technetium Loading Breakthrough Curves, Lead and Lag Columns

Figure 3.3 shows that the $^{95m}\text{TcO}_4^-$ tracer indicates there is approximately 2% initial $^{95m}\text{TcO}_4^-$ coming through the system. It is not clear why the tracer shows an initial breakthrough of pertechnetate. If there were carryover from the previous processing, one would expect the breakthrough to drop and eventually reestablish a new, lower baseline.

The C_0 value for ^{99}Tc was $375 \mu\text{Ci/L}$ (22.1 mg/mL).^(a) The initial $^{95\text{m}}\text{TcO}_4^- C_0$ was 1930 counts/min/mL of sample. This decayed to 1700 counts/min/mL of sample by the end of the run. The $^{95\text{m}}\text{TcO}_4^- C_0$ was recounted periodically to account for this decay. The C/C_0 % values are plotted on a log/probability scale. The C/C_0 value of 2% is marked on the plot. This corresponds to 98% removal of technetium, the target technetium removal for Envelope A and B waste feeds.

The technetium breakthrough, after processing approximately 1590 mL of AZ-101 feed, was 31% on the lead column and 0.1% on the lag column, based upon the ^{99}Tc concentration values. The $^{95\text{m}}\text{TcO}_4^-$ breakthrough, measured during monitoring of the loading, was approximately 35% on the lead column and 3.3% on the lag column, indicating that the $^{95\text{m}}\text{TcO}_4^-$ tracer estimated the system performance approximately 2 to 3% high. Based upon analytical measurements, all of the technetium was in the pertechnetate form.

The initial ^{99}Tc decontamination factors (DFs) for the first and second columns (derived from the ^{99}Tc concentration in the first sample from each column) are 980 and 1530, respectively. A better measurement of the technetium decontamination is the total DF observed for the system, i.e., the concentration of technetium remaining in the effluent compared to the initial concentration of the ion exchange feed. The overall ^{99}Tc DF was 205. The concentration of ^{99}Tc in the effluent composite was $1.83 \mu\text{Ci/L}$, and the concentration of ^{99}Tc in the initial feed was $375 \mu\text{Ci/L}$. The effluent composite concentration demonstrates a total technetium removal of 99.5%.

The λ value is the number of BVs of processed feed at which the C/C_0 value reaches 50%, if kinetics are not a significant factor, and is a direct indicator of the effective capacity of the resin. Since there was only ~30% breakthrough of technetium on the lead column, the λ value could not be experimentally determined. The λ value can be estimated by extrapolating the loading-curve data plotted on a linear scale. This extrapolation provides an estimate of 360 BVs of feed that could be processed before reaching 50% C/C_0 breakthrough. This value is lower than the λ value calculated from the batch-contact data, i.e., 460 BVs.

3.4 AZ-101 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 gamma emission spectrometry analysis of $^{95\text{m}}\text{Tc}$, which reflects the concentration of total pertechnetate present in each sample. The ratio of the measured $^{95\text{m}}\text{Tc}$ passing through the lead and lag column with respect to the initial amount of $^{95\text{m}}\text{Tc}$ in the ion exchange feed (C/C_0) was calculated during displacement. After rinsing for 4 BVs, a small sample was collected from the lead column every 1 BV and measured for $^{95\text{m}}\text{TcO}_4^-$. The C/C_0 decreased to a minimum value of 25% at 5 BVs and then increased to 40% by 6.5 BVs. At this point, the feed displacement was terminated. The target feed displacement of $C/C_0 = 1\%$ for the lead column was not realized. The elution of TcO_4^- with 0.10 M NaOH has been observed in previous column waste testing (Blanchard et al. 2000a,

(a) Note that the ^{99}Tc concentration ($375 \mu\text{Ci/L}$) of the ion exchange feed is not the same as that reported for the batch-distribution ^{99}Tc concentration ($360 \mu\text{Ci/L}$). This difference of 4% is within the stated analytical uncertainty.

2000b; Burgeson et al. 2002a, 2002b, 2002c). Once the feed displacement was stopped, 2 BVs of DI water were passed through the system to rinse the lead column.

The ratio of the measured ^{95m}Tc and ^{99}Tc passing through the lead and lag column with respect to the initial amount of ^{95m}Tc and ^{99}Tc , respectively, in the ion exchange feed (C/C_0) was calculated for all feed-displacement samples. Figure 3.4 presents the ^{95m}Tc and ^{99}Tc data for the lead-column feed displacement and rinse samples.

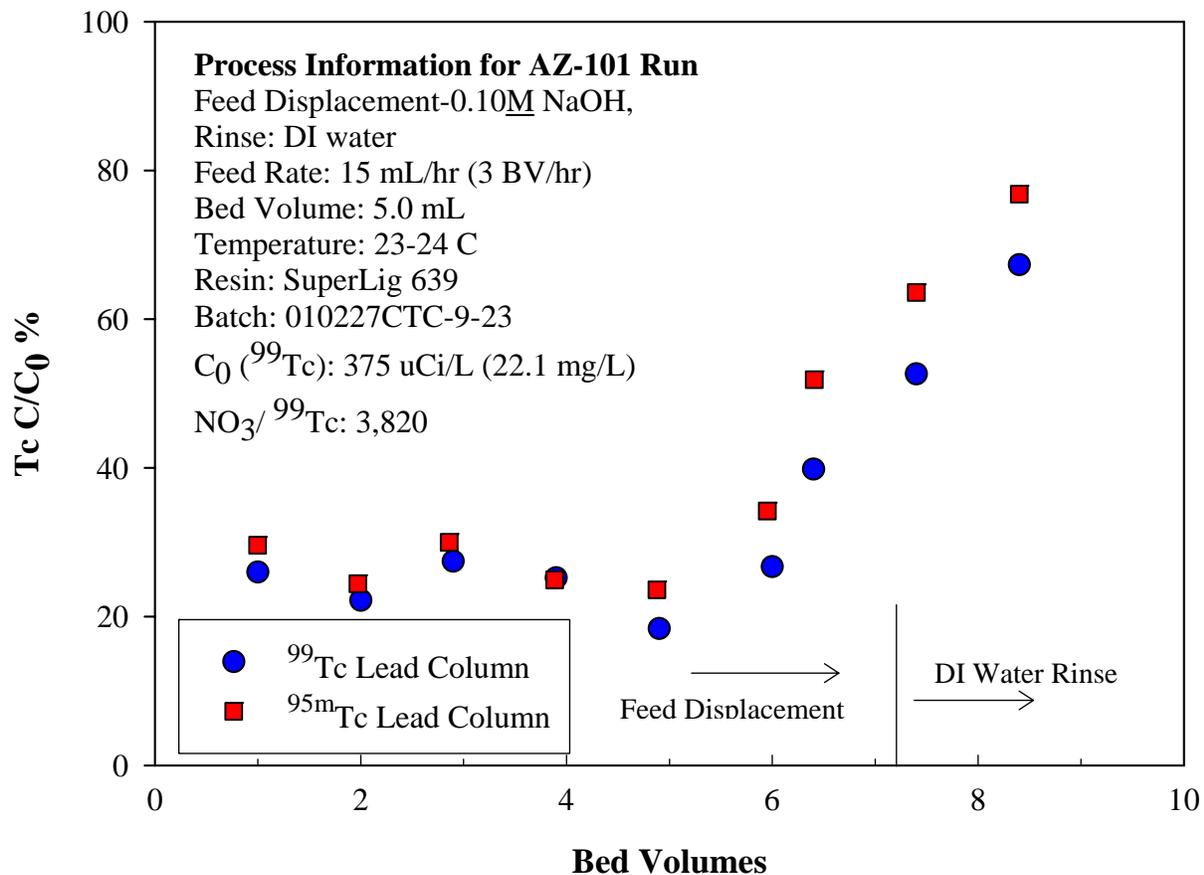


Figure 3.4. Tc and $^{95m}\text{TcO}_4^-$ C/C_0 for Lead Column Feed Displacement and Rinse

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 stage used approximately 2 BVs of water flowing at a rate of 3 BV/h. The rinse was collected as two samples. Figure 3.4 includes the data from both of the rinse samples as the last two data points. The vertical line located at the x-axis identifies which data are a result of feed displacement and which are a result of the water rinse. As can be seen in Figure 3.4, the elution of technetium, which started during feed displacement, continued during the water wash. The $^{99}\text{TcO}_4^-$ was measured in the rinse samples at C/C_0 values of 53% and 67%.

3.5 AZ-101 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 30 min before elution was started by running pre-equilibrated 65°C water through the column jacket. The elapsed time between the rinse cycle and the start of the elution was 50 min. The eluate was collected in 1-BV increments and monitored using gamma emission spectrometry analysis of ^{95m}Tc .

Figure 3.5 indicates the ratio of the measured ^{95m}Tc eluted from the lead column with respect to the initial amount of ^{95m}Tc in the ion exchange feed ($^{95m}\text{Tc} C/C_0$), which was calculated for each sample during the elution stage. In addition, the elution samples were analyzed for total technetium (^{99}Tc). Figure 3.5 also shows the ratio of the initial ^{99}Tc and the ^{99}Tc measured in the elution samples. The Y-axis is a logarithmic scale to clearly show the large range of C/C_0 values.

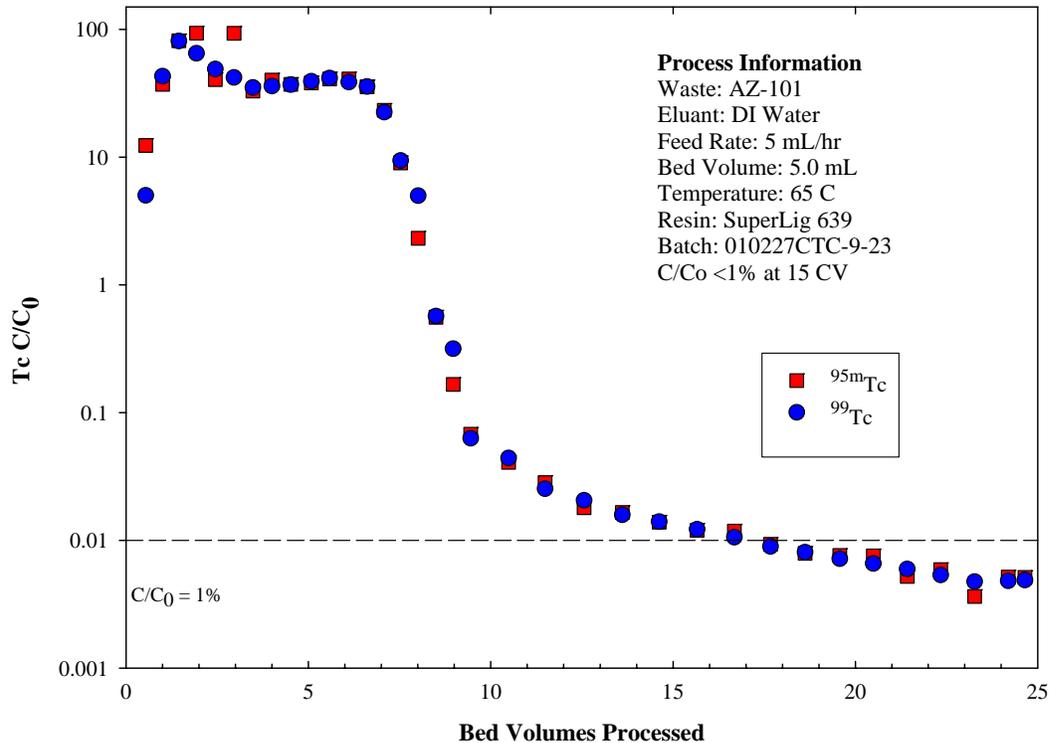


Figure 3.5. Elution of Technetium from Lead Column

The concentrations of ^{99}Tc and $^{95m}\text{TcO}_4^-$ paralleled one another quite nicely except at the higher concentrations of TcO_4^- where increased detector dead times would be expected to slightly impact the $^{95m}\text{TcO}_4^-$ counting data. The elution of technetium peaked at approximately 2 BVs with a value approximately 81 times higher than the initial feed concentration. There was a hump in the TcO_4^- elution, which may be due to elution of KTcO_4 . The elution was readily followed to $C/C_0 \leq 1\%$, the target column

elution value, using both ^{95m}Tc counting data and ^{99}Tc ICP-MS data. The target of $C/C_0 \leq 1\%$, was reached after processing 17 BVs of eluant.

The lead column was immediately regenerated with 0.25 M NaOH. Forty milliliters of NaOH were passed through the lead column at a flowrate of 1 BV/h. The regenerate was collected in two 20-mL aliquots and analyzed for ICP-AES analytes, technetium, and hydroxide. Table 3.4 shows the results of these analyses.

Table 3.4. Composition of Column Regenerate after AZ-101 Column Run

Analyte	Concentration, $\mu\text{g/mL}$	Concentration, <u>M</u>
Regeneration Solution #1		
^{99}Tc	0.0081(1.37E-03 $\mu\text{Ci/mL}$)	8.16E-07
Al	10	3.87E-04
B	47	4.32E-03
Cr	0.47	9.07E-06
K	< 40	< 1.03E-03
Na	3410	1.48E-01
OH	1500	8.8 E-02
Si	170	5.92E-03
Regeneration Solution #2		
^{99}Tc	0.001 (2.30E-05 $\mu\text{Ci/mL}$)	1.37E-08
Al	13	4.76E-04
B	78	7.24E-03
Cr	< 0.4	< 7.63E-06
K	< 40	< 1.01E-03
Na	6960	3.03E-01
OH	3230	1.9E-01
Si	360	1.29E-02
< indicates the analyte was not observed above the instrument detection limit		

Boron, chromium, and silicon were observed by ICP-AES and are reported opportunistically. Boron and silicon are possibly present due to leaching of the borosilicate glass vials used for sample storage; however, it is also possible that these two analytes are actually present in the regeneration solution. The concentration of ^{99}Tc present in the regeneration samples is approximately 0.4% of the amount of Tc observed in the initial feed.

3.6 AZ-101 Column Test—Elution Composite and Effluent Composite Analysis

Test plan TP-RPT-WTP-111, *Tank 241-AZ-101 and 241-AZ-102 Waste Sample Ion Exchange Testing*, merged the analytical requirements of pretreatment with those of vitrification. Thus, the analyte list for the analysis of the effluent and eluate composites was extensive. The data reported within the body of this report represent only those analytes that were present in the initial feed or analytes that were observed in both the effluent and eluate composites.

The eluate samples were combined to produce a 127-mL composite sample, and an aliquot was submitted for characterization. Table 3.5 reports all of the analytes observed by each analytical method: titration, ICP-AES, ICP-MS, IC, and Radiochemistry. The required minimum reportable quantity (MRQ) levels for all of the reported analytes were met. Any observed, non-requested analytes are reported opportunistically.

Table 3.5. Analysis Results for AZ-101 Technetium Eluate Composite

Analyte	Analytical Result	Analytical Method	Analyte	Analytical Result	Analytical Method
Radiochemical Results in $\mu\text{Ci/mL}$					
^{99}Tc (a)	4.58E+00	ICP-MS	^{95}Tc	1.34E-02	Sep/Count
^3H	2.07E-05	Sep/Count	^{137}Cs	< 3E-04	GEA
^{14}C	7.44E-05	Sep/Count	^{154}Eu	< 5E-05	GEA
^{60}Co	< 3E-05	GEA	^{238}Pu	9.88E-08	AEA
^{90}Sr	3.51E-03	Sep/Count	$^{239/240}\text{Pu}$	3.72E-07	AEA
$^{95\text{m}}\text{Tc}$	3.42E-01	Sep/Count	^{241}Am	1.82E-06	AEA
Inorganic Analysis Results in $\mu\text{g/mL}$					
Al	< 1.2	ICP-AES	Pb	< 2.0	ICP-AES
As	< 5.0	ICP-AES	Si	[19]	ICP-AES
B	92.7	ICP-AES	Sn	< 10	ICP-AES
Cr	[0.51]	ICP-AES	Ti	< 0.5	ICP-AES
Fe	< 0.5	ICP-AES	V	< 1.0	ICP-AES
K	[43]	ICP-AES	W	< 10	ICP-AES
Mo	< 1.0	ICP-AES	Zn	< 1.0	ICP-AES
Na	219	ICP-AES	Zr	< 1.0	ICP-AES
P	< 2.0	ICP-AES	U	< 40	ICP-AES
Inorganic Anion Results in $\mu\text{g/mL}$					
Cl	0.6	IC Anions	PO_4	1.4	IC Anions
F	2.0	IC Anions	SO_4	13	IC Anions
NO_2	44	IC Anions	oxalate	0.9	IC Anions
NO_3	43	IC Anions			
Carbon Results in $\mu\text{g C/mL}$					
TIC	24 $\mu\text{g C/mL}$	Persulfate	TIC	<70	Furnace
TOC	<34 $\mu\text{g C/mL}$	Persulfate	TOC	<420	Furnace
Physical Properties Units as Reported					
Hydroxide	no inflection	Titration	Density	1.00 g/mL	Gravimetric
[] signifies that the analyte was detected within 10 times the instrument detection limit and the uncertainty is estimated to exceed $\pm 15\%$.					
< indicates the analyte was not observed above the instrument detection limit					
(a) Uncertainty, including standards bias, ranged from -10% to +30%					

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. Chromium, chloride, fluoride, oxalate, phosphate, and sulfate were observed in very low quantities. The anions chloride, fluoride, nitrate, nitrite, oxalate, phosphate, and sulfate were observed at approximately 0.1% of the initial feed, strongly indicating that there was a slight amount of the original feed collected during elution. It is also possible that the resin may have retained and subsequently eluted some of each of the anions. The major radionuclides observed in solution were ^{99}Tc and $^{95\text{m}}\text{Tc}$. A slight amount of ^3H , ^{14}C , ^{90}Sr , ^{238}Pu , $^{239/40}\text{Pu}$, and ^{241}Am was also present in the eluate composite, confirming that a slight amount of feed was likely present in the system during elution. The concentration of radionuclides and inorganic analytes present in the elution composite is approximately 1000 fold less than that of the original feed. Since the solutions in the system were mixed during displacement, it is reasonable to assume that a slight amount of diluted feed could be present after rinsing with 43 mL of solution (33 mL 0.10 M NaOH and 10 mL of water). The most likely place for this residue is in the pump head, where there is a holdup volume of approximately 5 mL.

The effluent analytical samples were combined with the collected effluent to produce a 1595-mL composite sample, and an aliquot was submitted for characterization. Table 3.6 shows the results. The required MRQ levels for all of the requested analytes were met. Table 3.6 reports all of the analytes observed by each analytical method: titration, ICP-AES, ICP-MS, IC and Radiochemistry. Any observed, non-requested analytes are reported opportunistically. The total anion normality, 4.40 N, is larger than the total cation normality, 4.26 N. This difference is approximately 3 percent and is well within the analytical and experimental uncertainties.

Table 3.6. Composition of AZ-101 Tc IX Effluent Composite

Analyte	Analytical Result	Analytical Method	Analyte	Analytical Result	Analytical Method
Radiochemical Results in $\mu\text{Ci/mL}$					
^{99}Tc (a)	1.83E-03	ICP-MS	TcO_4^-	9.33E-04	Sep/Count
^3H	3.24E-02	Sep/Count	^{137}Cs	3.56E-02	GEA
^{14}C	1.92E-03	Sep/Count	^{154}Eu	< 2.00E-05	GEA
^{60}Co	1.68E-05	GEA	^{238}Pu	1.07E-06	AEA
^{90}Sr	1.66E-01	Sep/Count	$^{239/240}\text{Pu}$	9.25E-06	AEA
$^{95\text{m}}\text{Tc}$	9.87E-04	GEA	^{241}Am	6.75E-07	AEA
^{95}Tc	5.32E-05	GEA			
Inorganic Analysis Results in $\mu\text{g/mL}$					
Al	5,280	ICP-AES	Pb	[6.7]	ICP-AES
As	[14]	ICP-AES	Si	[53]	ICP-AES
B	[7.8]	ICP-AES	Sn	[44]	ICP-AES
Cr	570	ICP-AES	Ti	[2.6]	ICP-AES
Fe	[0.58]	ICP-AES	V	[1.4]	ICP-AES
K	3,800	ICP-AES	W	[51]	ICP-AES
Mo	85.3	ICP-AES	Zn	[1.7]	ICP-AES
Na	99,000	ICP-AES	Zr	[2.0]	ICP-AES
P	482	ICP-AES	U	< 40	ICP-AES
Inorganic Anion Results in $\mu\text{g/mL}$					
Cl	[130]	IC Anions	PO_4	1,600	IC Anions
F	1,900	IC Anions	SO_4	16,500	IC Anions
NO_2	61,900	IC Anions	oxalate	1,000	IC Anions
NO_3	52,950	IC Anions			
Carbon Results in $\mu\text{g C/mL}$					
TIC	6,830 $\mu\text{g C/mL}$	Persulfate	TIC	6,840	Furnace
TOC	345 $\mu\text{g C/mL}$	Persulfate	TOC	<240	Furnace
Physical Properties Units as Reported, <u>M</u> is moles/L					
Hydroxide	0.66 <u>M</u>	Titration	Density	1.229 g/mL	Gravimetric
<p>[] signifies that the analyte was detected within 10 times the instrument detection limit and the uncertainty is estimated to exceed $\pm 15\%$.</p> <p>< indicates the analyte was not observed above the instrument detection limit</p> <p>(a) Uncertainty, including standards bias, ranged from -10% to +30%</p>					

The TIC values reported by the furnace oxidation and the hot persulfate oxidation analytical methods agree quite well. The TOC values reported by these two analytical methods are not in good agreement. There appears to be a matrix –related problem with the TOC furnace value, as the matrix spike recovery was outside the $\pm 25\%$ recovery (52% recovery). An evaluation of the results indicates the problem is definitely matrix-related since multiple analyses of the effluent sample yielded similar results. The furnace TOC value for the effluent is expected to be low. The hot persulfate-based TOC value should be considered the best representation of the TOC content. This is consistent with past observations of

TIC/TOC data where it has been concluded that the hot persulfate-based values are generally considered the best representation of the TIC and TOC content (Urie et al. 2002).

3.7 Comparison of AZ-101 Initial Feed and Effluent Composite Composition

The analysis of the effluent composite (Table 3.6) can be compared with the initial feed analysis (Table 3.1) to determine the effect, if any, on the analytes in solution upon passing through the resin bed. Table 3.7 compares the initial feed characterization with the final effluent composition. Note that the effluent composite contains the column effluent and the samples collected to monitor the lead and lag loading. The table also shows the percent change in concentration from the initial feed composition. A negative percent change indicates that the effluent composition was lower than the initial feed composition. Changes of less than or equal to 10% are likely insignificant since this is the stated uncertainty of the individual reported results. For those samples that are reported to be within ten times the detection limit (indicated with brackets in the table), an observed change of less than or equal to 15% is not significant since this is the reported analytical uncertainty of the individual reported results. Analytes that exceeded the percent change criteria stated above are discussed in the following paragraph.

Table 3.7. Comparison of Initial Feed and Effluent Composite Composition

Analyte	Feed	Effluent	Percent Change ^(a)	Analyte	Feed	Effluent	Percent Change ^(a)
Radiochemical Results in $\mu\text{Ci/mL}$							
⁹⁹ Tc ^(b)	3.75E-01	1.83E-03	-99.5%	TcO ₄ ⁻	3.78E-01	9.33E-04	-99.8%
Inorganic Analysis Results in $\mu\text{g/mL}$							
Al	5070	5280	4%	Pb	[5.7]	[6.7]	17% ^(c)
As	[12]	[14]	13% ^(c)	Si	[76]	[53]	-31% ^(c)
B	16.5	[7.8]	-53%	Sn	[40]	[44]	10%
Cr	541	570	5%	Ti	[2.5]	[2.6]	2% ^(c)
Fe	< 0.5	[0.58]	16%	V	[1.2]	[1.4]	13% ^(c)
K	3640	3800	4%	W	[52]	[51]	-2%
Mo	81.2	85.3	5%	Zn	[1.6]	[1.7]	3% ^(c)
Na	97,900	99,000	1%	Zr	[1.8]	[2.0]	8% ^(c)
P	456	482	6%	U	< 0.0022 ^(d)	< 40	--
Inorganic and Organic Anion Results in $\mu\text{g/mL}$, <u>M</u> is moles/L							
Hydroxide	0.63 <u>M</u>	0.66 <u>M</u>	7%	NO ₂	62,800	61,900	-1%
Cl	[270]	< 130	-52%	oxalate	1,050	1,000	-5%
F	2,000	1,900	-5%	PO ₄	1,600	1,600	0%
NO ₃	52,600	52,950	1%	SO ₄	16,900	16,500	-2%
<p>(a) The percent change was determined by taking $\{(\text{effluent value} - \text{feed value}) / (\text{effluent value})\} * 100\%$</p> <p>(b) Uncertainty, including standards bias, ranged from -10% to +30%</p> <p>(c) The reported data is reduced in significant figures, hence, there may be rounding errors for the % change values</p> <p>(d) The initial feed analysis for U is reported in Table 3.1 as 0.0022 $\mu\text{g/mL}$ by KPA. The ICP-AES result, which is not reported in Table 3.1 was < 40 $\mu\text{g/mL}$.</p> <p>[] Signifies that the analyte was detected within 10 times the instrument detection limit and the uncertainty is estimated to exceed $\pm 15\%$.</p> <p>-- Indicates that the % change could not be calculated.</p> <p>< Indicates the analyte was not observed above the instrument detection limit</p>							

Concentration changes of greater than 15% were observed for boron, chloride, iron, lead, and silicon. The presence of boron and silicon were likely due to leaching from the glass storage vials; thus, the apparent concentration changes are an artifact. The chloride feed value was performed in duplicate: the primary response was 270 mg/L, and the duplicate response was <130 mg/L. Thus, it is likely that the reported value of 270 mg/L is high, indicating that the observed change in concentration is an artifact. The iron result is a combination of a less-than-detection value and a near-the-detection-limit value. Thus, the percent change of 16% for iron is within the stated analytical uncertainty. The percent change of 17% for lead is just outside the lower estimate of the analytical uncertainty and is likely within the error of the analytical measurement.

3.8 Technetium Recovery

All stages of the column run were monitored with a $^{95m}\text{TcO}_4^-$ tracer. Multiple samples were collected during each process step: loading, feed displacement, water rinse, elution, and regeneration. Approximately one-half of these collected samples were also analyzed for ^{99}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 ^{99}Tc by ICP-MS. Since overlapping analytical data are available for each processing stage, multiple mass balances are shown. Section 3.8.1 details the mass balance of the column system based upon the analysis of the composite solutions for ^{99}Tc by ICP-MS. Section 3.8.2 details the mass balance for column loading and column elution using ^{99}Tc by ICP-MS data. Section 3.8.3 calculates the estimated technetium retained on the resin.

3.8.1 System Recovery Using Composite Solution ^{99}Tc Data

Table 3.8 shows the system recovery for ^{99}Tc . The recovery is based upon the analysis of each of the process samples (feed displacement, water rinse, regeneration) and the final effluent and eluate composite samples. These solutions account for all of the material collected during the column run. The effluent composite solution contains the ion exchange effluent and the loading samples taken during processing. The eluate composite was simply a collection of each of the lead column elution fractions. All values are based upon ICP-MS analysis of ^{99}Tc .

Table 3.8. Recovery for Technetium During Processing Using Composite Solution Data ^(a)

Column Processing Step	Technetium Activity, μCi (mg)		Percent of ^{99}Tc in Feed
Feed	596.6	(35.2)	100
Effluent composite ^(b)	2.92	(0.17)	0.5
Lead column feed displacement ^(c)	0.74	(0.043)	0.1
Lead column rinse ^(d)	2.25	(0.13)	0.4
Lead column eluate	582.6	(34.3)	97.7
Lead column regeneration ^(d)	0.025	(0.0015)	0.004
Total technetium recovered	588.5	(34.68)	98.6

(a) Based upon ^{99}Tc analysis of final solutions, except where noted.
 (b) Effluent composite contains collected effluent and loading samples.
 (c) Value (0.74 μCi) is based upon analysis of $\frac{1}{2}$ the collected samples, and interpolation of the remaining $\frac{1}{2}$.
 (d) Rinse and regeneration sample values both are a sum of the analysis of two solutions.

A total of 596.6 μCi of ^{99}Tc was processed through the column system. The data reported in Table 3.8 do not account for the technetium loaded on the lag column. The lead-column recovery was 98.7%. Section 3.8.3 includes the lag-column loading and the retention of technetium.

3.8.2 Recovery for Lead Column Loading and Elution Using Individual ^{99}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. Table 3.9 shows a recovery for the lead column loading and the elution of AZ-101 tank-waste feed. 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 = the ratio of Tc in each sample versus the Tc present in the initial feed
- C_0 = the initial feed technetium concentration
- ΔV = the volume of feed processed between each sample (including sample volume)
- N = the 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 = the ratio of Tc in each elution sample versus the Tc present in the initial feed
- C_0 = the initial feed technetium concentration
- ΔV = the volume of each elution sample
- N = the number of samples collected during elution.

Table 3.9 shows the lead column loading and elution recovery calculated for total technetium.

Table 3.9. Recovery for Lead Column Loading and Elution of Technetium Using Sum of Individual Sample ⁹⁹Tc Analyses

Column Processing Step	Technetium Activity, μCi (mg)	Percent of ⁹⁹ Tc in Total Feed, %
Total Technetium Processed	596.6 (35.2)	100
Loaded on Lead Column	550.4 (32.4)	92.3
Eluted from Lead Column ^(a)	551.3 (32.5)	92.4
Fraction Eluted from Lead Column	100%	
(a) Based upon the sum of μCi eluted during elution (548.3), feed displacement (0.74), water rinse (2.25), and regeneration stages (0.025)		

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 recovery of 102% indicates that it is likely that technetium was retained on the resin from the previous ion exchange run (AN-102/C-104). This is not unexpected, since the lag column from the AN-102/C-104 run was not eluted and was switched to the lead column position for the AZ-101 ion exchange run. However, 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. Further discussion of technetium retention is provided in the next section.

3.8.3 Estimate of Technetium Retained on Lead and Lag Columns

Table 3.10 estimates the amount of technetium retained on the lead and lag columns. 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 analysis of the eluate composite solution combined with the analysis of the regeneration samples, feed-displacement samples, and water-rinse sample.

Equation 3.3 was used to calculate the amount of technetium passing through the lag column during processing:

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

where

C/C_0 = the ratio of Tc exiting the lead column with respect to the initial technetium feed concentration

C_0 = the initial feed technetium concentration

ΔV_L = the volume of feed processed by the lag column

N = the 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 eluted from the lag column during elution.

The data indicate that a significant amount of technetium was loaded and retained on the lag column (approximately 6%). The lead-column retention value may reflect analytical uncertainties rather than the actual amount of technetium retained on the columns. However, as stated in Section 3.8.2, it is likely that technetium was retained on the resin from the previous ion exchange run (AN-102/C-104), and the eluted value, which is greater than the loaded value, is correct. 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 3.10. Estimate of Technetium Retained on Lead and Lag Columns During Processing of AZ-101

Column Processing Step	Technetium Activity, μCi (mg)	Percent of ⁹⁹ Tc in Total Feed, %
Loaded on Lead Column	550.4 (32.4)	92.3
Eluted from Lead Column ^(a)	551.3 (32.5)	92.4
Estimated Retained on Lead Column	-0.93 (-0.05)	-0.2
Technetium Entering Lag Column	37.7 (2.22)	6.3
Eluted from Lag Column	0.42 (0.025)	0.1
Estimate Retained on Lag Column	36.9 (2.17)	6.2
Estimate Retained on Both Columns	36.0 (2.12)	6.0
(a) Based upon the sum of μCi eluted during elution (548.3), feed displacement (0.74), water rinse (2.25), and regeneration stages (0.031)		

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 ⁹⁹Tc decontamination of an Envelope B material (Tank AZ-101); remove at least 98% of technetium within 250 bed volumes (BVs) of processed feed
- elute technetium from the resin to a target technetium C/C₀<1%, within 22 BV of eluant
- provide a technetium-decontaminated sample for downstream process testing (i.e., corrosion testing, LAW melter feed testing, and vitrification)
- 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 AZ-101 waste feed and SL-639 resin progressed without difficulties. A total of 318 BVs of feed (1590 mL) was processed through the dual-column system. The

lead-column breakthrough was 31%, and the lag-column breakthrough was 0.1%, based upon ^{99}Tc analytical data.

Feed displacement progressed only slightly during passage of 0.10 M NaOH through the system; there was no sharp drop in the concentration of ^{99}Tc in the lead column. The feed-displacement samples collected after 6 BV showed that technetium was eluting from the lead column; the C/C_0 increased from 18% to 40%. The water rinse of the first column continued the elution of technetium, resulting in a final C/C_0 of 67%. The specified rinse volume is one-half an apparatus volume, which correlates to 2 BVs (10 mL) of water in this system.

The elution of the technetium-loaded columns with DI water at 65°C progressed quickly. The concentration of ^{99}Tc peaked in the 2nd BV, giving a $C/C_0 = 81$. The elution peak flattened out until the 7th BV and then decreased sharply, with tailing starting near the 10th BV. The C/C_0 ratio was already quite low, ~ 0.04 , at this point. The target C/C_0 value of <0.01 was reached at the 17th BV. Continuing the elution for an additional 9 BVs only succeeded in reducing the C/C_0 ratio to 0.003.

The resin was regenerated with 2 apparatus volumes (8 BVs) of 0.25 M NaOH. The regeneration effluent was collected in two samples, in which the ^{99}Tc concentrations were found to be 1.37 $\mu\text{Ci/L}$ and 0.023 $\mu\text{Ci/L}$.

Investigate the potential for resin and/or column fouling

No anomalies were 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 ^{99}Tc decontamination of an Envelope B material (Tank AZ-101); remove at least 98% of technetium within 250 bed volumes (BV) of processed feed

Waste feed from Tank AZ-101 was successfully processed through a dual 5.0-mL resin-bed column system that had previously been loaded and eluted with AW-101 simulant, an Envelope A waste feed, and an Envelope C waste feed. The AZ-101 feed contained 375 $\mu\text{Ci/L}$ ^{99}Tc , and 1590 mL of feed (318 BV) were processed through the column system. The total amount of technetium processed through the two columns was 596.6 μCi . The final effluent composite ^{99}Tc concentration was 1.83 $\mu\text{Ci/L}$, which correlates to 99.5% removal of technetium. An overall ^{99}Tc DF value of 205 was obtained using the two 5.0-mL beds in series.

The ^{99}Tc concentration in the effluent from this test meets the plant target of removing ^{99}Tc to a level that would permit production of an LAW glass with a concentration of $^{99}\text{Tc} < 0.1 \text{ Ci/m}^3$. Assuming 14% sodium oxide loading and a sodium molarity of 4.3, the calculated technetium concentration in the LAW glass is 0.005 $\text{Ci } ^{99}\text{Tc/m}^3$ LAW glass.

Elute technetium from the resin to a target technetium $C/C_0 < 1\%$, within 22 BV of eluant

The elution reached the target level of technetium, $C/C_0 < 1\%$, within 17 bed volumes of eluant. This met the goal of reducing the technetium concentration to less than $1\% C/C_0$ within 22 bed volumes.

Provide a technetium-decontaminated sample for downstream process testing (i.e., LAW melter feed testing and vitrification)

The column effluent was combined with the collected loading samples, providing approximately 1595 mL of solution. This solution is the LAW vitrification feed and contains $1.83 \mu\text{Ci/L } ^{99}\text{Tc}$ and $0.036 \mu\text{Ci/L } ^{137}\text{Cs}$. The eluate samples were composited to provide 127 mL of solution. This solution is the HLW vitrification feed and contains $4578 \mu\text{Ci/L } ^{99}\text{Tc}$.

Provide batch-distribution measurements on actual tank waste feed

Batch distributions were measured with AZ-101 waste feed using SL-639 resin batch #010227CTC-9-23. The SL-639 resin demonstrated excellent removal of technetium: the $^{99}\text{Tc } K_d$ value at the initial feed conditions was ~ 920 , providing 92% removal of technetium. Extrapolation of the isotherm data provides an estimated $^{99}\text{Tc } K_d$ value of 920 mL/g at the initial feed conditions. The batch λ value derived from the $^{99}\text{Tc } K_d$ extrapolated to the feed conditions is ~ 460 BV. This value is significantly higher than the extrapolated column λ value (360 BV).

4.0 Conclusions

All objectives of the testing were met.

- All process steps for ^{99}Tc decontamination of AZ-101 waste feed were successfully demonstrated.
- No resin or column fouling was observed.
- Elution of the lead column reached the target C/C_0 value of <0.01 in 17 BV. This exceeds the baseline requirement that C/C_0 be reduced to 1% within 22 BV.
- The ^{99}Tc decontamination of an Envelope B waste (AZ-101) was successfully demonstrated. A total of 318 BV (1590 mL, 375 $\mu\text{Ci } ^{99}\text{Tc/L}$) of feed was processed, resulting in 99.5% removal of ^{99}Tc . This exceeds the design basis: removal of 98% of technetium after processing 250 BV of feed.
- The Tc-decontaminated AZ-101 feed (1590 mL, 1.83 $\mu\text{Ci } ^{99}\text{Tc/L}$) and the Tc eluate (127 mL, 4578 $\mu\text{Ci } ^{99}\text{Tc/L}$) were provided for downstream LAW and HLW vitrification feed, respectively.
- Batch-distribution measurements were made on the AZ-101 feed. The resulting batch λ value compares adequately with the column λ value determined by extrapolating the observed breakthrough to 50%.
- There were no results from this testing which would require additional testing.

5.0 References

- Bechtel National, Inc. (BNI). 2002. *Research and Technology Plan*, 24590-WTP-PL-RT-01-002, Rev 1, U.S. Department of Energy, Office of River Protection, Richland, WA.
- Blanchard Jr. DL, DE Kurath, and BM Rapko. 2000a. *Small Column Testing of Superlig® 639 for Removal of ⁹⁹Tc from Hanford Tank Waste Envelope C (Tank 241-AN-107)*, PNWD-3028, Battelle-Pacific Northwest Division, Richland, WA.
- Blanchard Jr. DL, DE Kurath, and JR Bontha. 2000b. *Small Column Testing of Superlig® 639 for Removal of ⁹⁹Tc from Hanford Tank Waste Envelope A (Tank 241-AW-101)*, PNWD-3004, Battelle-Pacific Northwest Division, Richland, WA.
- Burgeson IE, JR Deschane, and DL Blanchard Jr. 2002. *Small Column Testing of Superlig® 639 for Removal of ⁹⁹Tc from Hanford Tank Waste Envelope A (241-AP-101)*, PNWD-3222, Battelle-Pacific Northwest Division, Richland, Washington.
- Burgeson IE, JR Deschane, and DL Blanchard Jr. 2003. *Small Column Testing of Superlig® 639 for Removal of ⁹⁹Tc from Hanford Tank Waste 241-AN-102 Supernate (Envelope C) Mixed with Tank 241-C-104 Solids (Envelope D) Wash and Permeate Solutions*, PNWD-3252, Battelle-Pacific Northwest Division, Richland, WA.
- Fiskum SK, DL Blanchard Jr., BM Rapko, and ST Arm. 2003. *Small Column Testing of Superlig® 644 for Removal of ¹³⁷Cs from Hanford Tank Waste Envelope B (Tank 241-AZ-101)*. PNWD-3266, Battelle-Pacific Northwest Division, Richland, WA.
- Geeting JGH, RT Hallen, LK Jagoda, AP Poloski, RD Scheele, and DR Weier. 2002. *Filtration, Washing, and Caustic Leaching of Hanford Tank AZ-101 Sludge*. PNWD-3206, Battelle-Pacific Northwest Division, Richland, WA.
- Hamm LL, F. G. Smith, III, and D. J. McCabe. 2000. *Preliminary Ion Exchange Modeling for Removal of Technetium from Hanford Waste Using SuperLig® 639 Resin*, WSRC-TR-2000-00305, Westinghouse Savannah River Company, Aiken, SC.
- Rapko BM, KJ Carson, and DL Blanchard Jr. 2003. *Equilibrium Batch Contact Testing of SuperLig 639*, PNWD-3251, Battelle—Pacific Northwest Division, Richland, WA.
- Toth JJ. 2001. *Tank 241-AZ-101 and 241-AZ-102 Ion Exchange Test Specification*, 24590-PTF-TSP-RT-01-002, Rev 1, Bechtel National, Inc, Richland, WA.
- Urie, MW, PR Bredt, JA Campbell, OT Farmer, SK Fiskum, LR Greenwood, EW Hoppe, LK Jagoda, GM Mong, AP Poloski, RD Scheele, CZ Soderquist, RG Swoboda, MP Thomas, and JJ Wagner. 2002. *Chemical Analysis and Physical Property Testing of 241-AZ-101 Tank Waste-Supernatant and Centrifuged Solids*, PNWD-3215, Battelle-Pacific Northwest Division, Richland, WA.

Appendix A

Sample Identification

Appendix A: Sample Identification

Sample ID	Sample #	Description
AZ1-Tc-0	02-2546 and 02-2787	Initial feed sample, analyzed twice for technetium content
AZ1-Tc-PL1 through 6	--	Lead and lag column reservoir collected during initial loading
AZ1-Tc-LL1 through LL27	02-2547 to 02-2559	Loading samples from lead column (½ submitted for analysis)
AZ1-Tc-LP1 through LP13	02-2560 to 02-2567	Loading samples from lag column (½ submitted for analysis)
AZ1-Tc-FD-P1 through P8	02-2570 to 02-2576	Feed displacement samples from lag column
AZ1-Tc-FD-L5 through L8	02-2580 to 02-2583	Feed displacement samples from lead column
AZ1-Tc-RL1 and RL2	02-2578 and 02-2579	DI water rinse sample
AZ1-Tc-E1-1 through E1-38	02-2584 to 02-2604	Lead column eluate samples
AZ1-Tc-Regen 1	02-2605	Lead column regeneration sample
Tc-AZ1-EFF-Copm	02-2789	Effluent composite sample (contains feed displacement and rinse solutions)
Tc-AZ1-Eluate	02-2788	Eluate composite sample
-- indicates samples were not submitted for analytical characterization		

Appendix B

Column-Run Spreadsheets

Appendix B: Column-Run Spreadsheets

Pre-Loading

Feed: AZ-101 supernate with Cs-137 removed by ion exchange (SL644)
 Resin: SuperLig 639, batch #010227CTC-9-23
 Tracer: Tc-95m
 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 (w/o/hole) = 298.5 g
 Bed volumes diverted to waste = none
 Supernate Density = 1.2290
 Feed analyses: Tc-99, µCi/mL
 Flow Rate: approx 15 mL/hr or ~3.0 CV/hr
 Start Date and Time: 4/24/2002 8:30 AM
 End Date and Time: 4/29/2002 7:40 PM

-- indicates the table cell is empty, the lack of data is intentional

Table 1. Column Loading Initial Sample Data Sheet

Sample	Source	# CV	Elapsed Time at Start, h:m	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 Bottle	--	--	--	--	--	--	--	--	--	--	--
AZ1-Tc-0-C	Feed Bot	--	0:00	--	2	17.0127	--	--	--	--	--
AZ1-Tc-0b-C	Feed Bot	--	0:00	--	10	16.8871	--	--	--	--	--
AZ1-Tc-PL1	Col 2	1	0:00	4/24/2002 8:30 AM	5	16.9043	8:30 AM	8:50 AM	--	20.6075	20.2533
AZ1-Tc-PL2	Col 2	2	0:20	4/24/2002 8:50 AM	5	16.9714	8:50 AM	9:10 AM	--	21.4294	21.0613
AZ1-Tc-PL3	Col 2	3	0:40	4/24/2002 9:10 AM	5	16.8187	9:10 AM	9:30 AM	--	20.9507	20.5688
AZ1-Tc-PL4	Col 2	4	1:00	4/24/2002 9:30 AM	5	16.9697	9:30 AM	9:50 AM	--	21.9894	21.5936
AZ1-Tc-PL5	Col 2	5	1:20	4/24/2002 9:50 AM	5	16.9169	9:50 AM	10:10 AM	--	21.8365	21.4268
AZ1-Tc-PL6	Col 2	6	1:40	4/24/2002 10:10 AM	5	17.0120	10:10 AM	10:30 AM	--	22.2657	21.8421

Bed Height of Column 1 @ begin. 3.2 cm
 Bed Height of Column 2 @ begin. 3.2 cm

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

Bed Height of Column 1 @ end 3.2 cm
 Bed Height of Column 2 @ end 3.2 cm

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

B.1

Loading

Feed: AZ-101 supernate with Cs-137 removed by ion exchange (SL644)
 Resin: SuperLig 639, batch #010227CTC-9-23
 Tracer: Tc-95m
 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/hole) = 298.5 g
 Bed volumes diverted to waste: none

Supernate Density = 1.2290
 Flow Rate: approx 15 mL/hr or ~3.0 CV/
 Start Date and Time: 4/24/2002 8:30 AM
 End Date and Time: 4/29/2002 7:40 PM

-- indicates the table cell is empty, the lack of data is intentional

Table 2. Data Collection for Tc Loading of Lead Column in AZ-101 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)	Mass Processed, g	Est. Effluent Vol	Est. Flow Rate
AZ1-Tc-0-C	Feed bo	--	--	--	10	17.0127	--	--	--	19.3640	2.3513	--	--	--
Effluent bottle, 2 L	--	--	--	--	--	--	--	--	298.5	--	--	0.0	--	--
AZ1-Tc-LL1	Col 1	1	0:00	4/24/2002 10:30 AM	2	17.0468	10:30 AM	10:38 AM	--	19.2097	2.1629	2.2	1.8	0.00
AZ1-Tc-LL2	Col 1	5	1:40	4/24/2002 12:10 PM	2	16.9181	12:10 PM	12:18 PM	322.3	19.0004	2.0823	30.1	24.5	14.67
AZ1-Tc-LL3	Col 1	15	5:00	4/24/2002 3:30 PM	2	16.8882	3:30 PM	3:38 PM	373.5	18.9955	2.1073	85.6	69.6	13.92
AZ1-Tc-LL4	Col 1	25	8:20	4/24/2002 6:50 PM	2	16.9775	6:50 PM	6:58 PM	428.04	19.0388	2.0613	142.1	115.6	13.88
AZ1-Tc-LL5	Col 1	35	11:40	4/24/2002 10:10 PM	2	16.9496	10:10 PM	10:18 PM	479.9	19.0515	2.1019	198.3	161.3	13.83
AZ1-Tc-LL6	Col 1	45	15:00	4/25/2002 1:30 AM	2	17.0095	1:30 AM	1:38 AM	534.0	19.1223	2.1128	254.5	207.1	13.80
AZ1-Tc-LL7	Col 1	55	18:20	4/25/2002 4:50 AM	2	16.8728	4:50 AM	4:58 AM	585.34	19.1025	2.2297	310.2	252.4	13.77
AZ1-Tc-LL8	Col 1	65	21:40	4/25/2002 8:10 AM	2	16.8958	8:10 AM	8:18 AM	640.2	18.9483	2.0525	367.1	298.7	13.78
AZ1-Tc-LL9	Col 1	75	25:00	4/25/2002 11:30 AM	2	16.9222	11:30 AM	11:38 AM	696.5	19.4912	2.5690	428.1	348.3	13.93
AZ1-Tc-LL10	Col 1	85	28:20	4/25/2002 2:50 PM	2	17.0163	2:50 PM	2:58 PM	764.1	19.6872	2.6709	498.3	405.5	14.31
AZ1-Tc-LL11	Col 1	95	31:40	4/25/2002 6:10 PM	2	16.9941	6:10 PM	6:18 PM	823.8	19.3839	2.3898	563.1	458.2	14.47
AZ1-Tc-LL12	Col 1	105	35:00	4/25/2002 9:30 PM	2	16.9562	9:30 PM	9:38 PM	885.4	19.3730	2.4168	627.1	510.3	14.58
AZ1-Tc-LL13	Col 1	115	38:20	4/26/2002 12:50 AM	2	16.9137	12:50 AM	12:58 AM	928.6	19.2148	2.3011	674.7	549.0	14.32
AZ1-Tc-LL14	Col 1	125	41:40	4/26/2002 4:10 AM	2	17.0496	4:10 AM	4:18 AM	987.7	19.3243	2.2747	736.1	599.0	14.49
AZ1-Tc-LL15	Col 1	130	47:13	4/26/2002 9:43 AM	2	16.9453	9:43 AM	9:51 AM	1084.8	19.2721	2.3268	837.9	681.8	14.65
AZ1-Tc-LL16	Col 1	140	50:30	4/26/2002 2:10 PM	2	17.0754	2:10 PM	2:18 PM	1163.4	19.3681	2.2927	918.8	747.6	14.82
AZ1-Tc-LL17	Col 1	150	53:50	4/26/2002 5:30 PM	2	17.0143	5:30 PM	5:38 PM	1232.5	19.3228	2.3085	992.5	807.6	15.14
AZ1-Tc-LL18	Col 1	160	57:10	4/26/2002 8:50 PM	2	17.0575	8:50 PM	8:58 PM	1289.8	19.3722	2.3147	1052.1	856.1	15.20
AZ1-Tc-LL19	Col 1	170	60:30	4/27/2002 12:10 AM	2	17.0725	12:10 AM	12:18 AM	1347.1	19.5912	2.5187	1114.3	906.6	15.28
AZ1-Tc-LL20	Col 1	180	63:50	4/27/2002 3:30 AM	2	16.9880	3:00 AM	3:08 AM	1408	19.4730	2.4850	1177.6	958.2	15.50
AZ1-Tc-LL21	Col 1	190	67:10	4/27/2002 6:50 AM	2	17.0527	6:50 AM	6:58 AM	1453.7	19.4548	2.4021	1228.1	999.3	15.41
AZ1-Tc-LL22	Col 1	200	70:30	4/27/2002 10:10 AM	2	17.0803	10:10 AM	10:18 AM	1529.7	19.4922	2.4119	1306.5	1063.1	15.79
AZ1-Tc-LL23	Col 1	220	77:10	4/27/2002 4:50 PM	2	16.9905	4:50 PM	4:58 PM	1654.2	19.4990	2.5085	1436.0	1168.4	15.86
AZ1-Tc-LL24	Col 1	240	83:50	4/27/2002 11:30 PM	2	16.9459	11:30 PM	11:38 PM	1768	19.4196	2.4737	1552.3	1263.0	15.85
AZ1-Tc-LL25	Col 1	260	90:30	4/28/2002 6:10 AM	2	16.8542	6:10 AM	6:18 AM	1904.7	19.3363	2.4821	1694.0	1378.3	16.09
AZ1-Tc-LL26	Col 1	280	97:10	4/28/2002 12:50 PM	2	16.8789	1:20 PM	1:28 PM	2031.5	19.3993	2.5204	1823.3	1483.6	16.10
AZ1-Tc-LL27	Col 1	300	103:50	4/28/2002 7:30 PM	2	16.9403	7:30 PM	7:38 PM	2160.8	19.4824	2.5421	1955.2	1590.8	16.21

B.2

-- indicates the table cell is empty, the lack of data is intentional

Table 2 (continued) Data Collection for Tc Loading of Lead Column in AZ-101 Run

Sample	Sampling Time	#CV	Count date	Count start time	file name	Mass Counted, g	count time, sec	gross counts	Net counts	back ground	Tot cts-bck	AZ1-Tc-0-C	counts of sample/gram
AZ1-Tc-0-C	--	--	4/25/2002	7:41 AM	AZ10C01	2.3513	900	61307	55514	-101	55514	55514	23610
--	--	--	--	--	--	--	--	--	--	--	--	--	--
AZ1-Tc-LL1	10:38 AM	0.4	4/25/2002	8:00 AM	AZ1LL01a	2.1629	900	4278	1225	-101	1225	55514	566
AZ1-Tc-LL2	12:18 PM	4.9	4/25/2002	10:07 AM	AZ1LL02	2.0823	900	4425	1489	-101	1489	55514	715
AZ1-Tc-LL3	3:38 PM	13.9	4/25/2002	10:42 AM	AZ1LL03	2.1073	900	4834	1476	-101	1476	55514	700
AZ1-Tc-LL4	6:58 PM	23.1	4/25/2002	11:16 AM	AZ1LL04	2.0613	900	4798	1719	-101	1719	55514	834
AZ1-Tc-LL5	10:18 PM	32.3	4/25/2002	11:55 AM	AZ1LL05	2.1019	900	4767	1530	-101	1530	55514	728
AZ1-Tc-LL6	1:38 AM	41.4	4/25/2002	12:43 PM	AZ1LL06	2.1128	900	4827	1681	-101	1681	55514	796
AZ1-Tc-LL7	4:58 AM	50.5	4/25/2002	9:30 AM	AZ1LL07	2.2297	900	4784	1510	-101	1510	55514	677
AZ1-Tc-LL8	8:18 AM	59.7	4/25/2002	1:50 PM	AZ1LL8	2.0525	900	4377	1536	186	1350	55514	658
AZ1-Tc-LL9	11:38 AM	69.7	4/25/2002	1:15 AM	AZ1LL09	2.5690	900	5412	2029	-101	2029	55514	790
AZ1-Tc-LL10	2:58 PM	81.1	4/26/2002	8:49 AM	AZ1LL10	2.6709	900	5270	1876	53	1823	51406	683
AZ1-Tc-LL11	6:18 PM	91.6	4/26/2002	10:23 AM	AZ1LL11	2.3898	900	5336	1935	53	1882	51406	788
AZ1-Tc-LL12	9:38 PM	102.1	4/26/2002	10:44 AM	AZ1LL12	2.4168	900	5482	2047	53	1994	51406	825
AZ1-Tc-LL13	12:58 AM	109.8	4/26/2002	7:27 AM	AZ1LL13	2.3011	900	5491	1921	53	1868	51406	812
AZ1-Tc-LL14	4:18 AM	119.8	4/26/2002	7:10 AM	AZ1LL14	2.2747	900	5329	2061	53	2008	51406	883
AZ1-Tc-LL15	9:51 AM	136.4	4/26/2002	10:03 AM	AZ1LL15	2.3268	900	5689	2305	53	2252	55514	968
AZ1-Tc-LL16	2:18 PM	149.5	4/27/2002	2:40 AM	AZ1LL16	2.2927	900	5671	2460	-8	2460	60744	1073
AZ1-Tc-LL17	5:38 PM	161.5	4/27/2002	12:58 AM	AZ1LL17	2.3085	900	5746	2436	-8	2436	54992	1055
AZ1-Tc-LL18	8:58 PM	171.2	4/27/2002	1:32 AM	AZ1LL18	2.3147	900	6016	2787	-8	2787	54992	1204
AZ1-Tc-LL19	12:18 AM	181.3	4/27/2002	2:20 AM	AZ1LL19	2.5187	900	7351	3597	-8	3597	54992	1428
AZ1-Tc-LL20	3:08 AM	191.6	4/27/2002	3:45 AM	AZ1LL20	2.4850	900	8126	4361	71	4290	51242	1726
AZ1-Tc-LL21	6:58 AM	199.9	4/27/2002	5:44 PM	AZ1LL21	2.4021	900	7698	4260	19	4241	51825	1766
AZ1-Tc-LL22	10:18 AM	212.6	4/27/2002	5:26 PM	AZ1LL22	2.4119	900	9109	5439	19	5420	51825	2247
AZ1-Tc-LL23	4:58 PM	233.7	4/27/2002	5:02 PM	AZ1LL23	2.5085	900	10610	6944	-38	6944	51825	2768
AZ1-Tc-LL24	11:38 PM	252.6	4/28/2002	10:20 AM	AZ1LL24	2.4737	900	12575	8945	-38	8945	49628	3616
AZ1-Tc-LL25	6:18 AM	275.7	4/28/2002	9:45 AM	AZ1LL24	2.4821	900	15112	11423	-38	11423	49628	4602
AZ1-Tc-LL26	1:28 PM	296.7	4/30/2002	8:31 AM	AZ1LL26	2.5204	900	20867	16706	43	16663	56303	6611
AZ1-Tc-LL27	7:38 PM	318.2	4/28/2002	8:08 PM	AZ1LL27	2.5421	900	22766	18601	-44	18601	48828	7317

-- indicates the table cell is empty, the lack of data is intentional

Data used in spreadsheet calculations

TcO ₄ ⁻ initial feed	0.378	uCi/mL
Tc-99 initial feed	0.375	uCi/mL

Table 2 (continued) Data Collection for Tc Loading of Lead Column in AZ-101 Run

Sample	control counts/ g	C/Co, Tc-95m	Tc-99 Data μ Ci/L	Dilution Factor	Tc-99 μ Ci/L	Tc-99 % C/Co	uCi/mL	Volume	uCi	uCi/mL	Volume	uCi
AZ1-Tc-0-C	23,610	--	5.77E-03	65015	3.75E+02	--	^{99m} TcO ₄ Mass Balance		Tc-99 Mass Balance			
--	--	--	--	--	--	--	--	--	--	--	--	--
AZ1-Tc-LL1	23,610	2.4%	3.67E-04	1047	3.84E-01	0.10%	0.36893	1.8	0.649	0.375	1.8	1
AZ1-Tc-LL2	23,610	3.0%	3.65E-04	1046	3.82E-01	0.10%	0.36655	22.7	8.319	0.375	22.7	9
AZ1-Tc-LL3	23,610	3.0%	--	--	--	0.10%	0.36679	45.2	16.562	0.375	45.2	17
AZ1-Tc-LL4	23,610	3.5%	--	--	--	0.10%	0.36465	46.0	16.782	0.375	46.0	17
AZ1-Tc-LL5	23,610	3.1%	3.62E-04	1038	3.76E-01	0.10%	0.36635	45.7	16.735	0.375	45.7	17
AZ1-Tc-LL6	23,610	3.4%	--	--	--	0.10%	0.36526	45.7	16.707	0.375	45.7	17
AZ1-Tc-LL7	23,610	2.9%	3.62E-04	1026	3.72E-01	0.10%	0.36716	45.3	16.635	0.375	45.3	17
AZ1-Tc-LL8	23,610	2.8%	--	--	--	0.13%	0.36747	46.3	17.017	0.375	46.3	17
AZ1-Tc-LL9	23,610	3.3%	5.45E-04	1056	5.75E-01	0.15%	0.36536	49.7	18.143	0.374	49.7	19
AZ1-Tc-LL10	21,863	3.1%	--	--	--	0.23%	0.3662	57.1	20.926	0.374	57.1	21
AZ1-Tc-LL11	21,863	3.6%	--	--	--	0.30%	0.36438	52.7	19.209	0.374	52.7	20
AZ1-Tc-LL12	21,863	3.8%	1.38E-03	1013	1.39E+00	0.37%	0.36374	52.1	18.946	0.374	52.1	19
AZ1-Tc-LL13	21,863	3.7%	--	--	--	0.59%	0.36396	38.7	14.099	0.372	38.7	14
AZ1-Tc-LL14	21,863	4.0%	--	--	--	0.80%	0.36274	49.9	18.115	0.371	49.9	19
AZ1-Tc-LL15	23,610	4.1%	1.97E-03	2288	4.50E+00	1.2%	0.3625	82.8	30.027	0.370	82.8	30.7
AZ1-Tc-LL16	25,834	4.2%	--	--	--	1.8%	0.3623	65.8	23.847	0.368	65.8	24
AZ1-Tc-LL17	23,388	4.5%	3.95E-03	2202	8.69E+00	2.3%	0.36095	60.0	21.639	0.366	60.0	22
AZ1-Tc-LL18	23,388	5.1%	--	--	--	2.9%	0.35854	48.5	17.392	0.364	48.5	18
AZ1-Tc-LL19	23,388	6.1%	6.09E-03	2128	1.30E+01	3.5%	0.35492	50.6	17.951	0.362	50.6	18
AZ1-Tc-LL20	21,793	7.9%	--	--	--	4.6%	0.34806	51.6	17.951	0.358	51.6	18
AZ1-Tc-LL21	22,041	8.0%	1.00E-02	2147	2.16E+01	5.8%	0.34772	41.1	14.281	0.353	41.1	15
AZ1-Tc-LL22	22,041	10.2%	--	--	--	8.0%	0.33946	63.8	21.658	0.345	63.8	22
AZ1-Tc-LL23	22,041	12.6%	1.79E-02	2149	3.84E+01	10.2%	0.33053	105.3	34.818	0.337	105.3	35
AZ1-Tc-LL24	21,107	17.1%	--	--	--	14.5%	0.31324	94.6	29.635	0.321	94.6	30
AZ1-Tc-LL25	21,107	21.8%	3.24E-02	2162	7.01E+01	18.7%	0.29558	115.3	34.083	0.305	115.3	35
AZ1-Tc-LL26	23,945	27.6%	--	--	--	24.7%	0.27364	105.2	28.793	0.283	105.2	30
AZ1-Tc-LL27	20,766	35.2%	5.59E-02	2054	1.15E+02	30.6%	0.24481	107.3	26.262	0.260	107.3	28
							Sum	1591 mL	537 uCi	Sum	1591 mL	550 uCi

B.4

Table 3. Data Collection for Tc Loading of Lag Column in AZ-101 Run

Sample	Source	# CV	Elapsed Time at Start, h:m	Projected Date/Time	2	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Eff. Bottle Mass	Sample+ Vial+ Cap Mass (g)	Sample Mass (g)	Effluent Mass, g	Est. Effluent Vol	Est. Flow Rate
AZ1-Tc-LP0	Col 2	2	0:45	4/24/2002 10:46 PM	2	16.7114	10:46 AM	10:54 AM	--	18.7230	2.0116	--	--	--
AZ1-Tc-LP1	Col 2	5	1:40	4/24/2002 12:10 PM	2	16.9348	12:18 PM	12:26 PM	322.3	19.0836	2.1488	28.0	22.8	12.64
AZ1-Tc-LP2	Col 2	25	8:20	4/24/2002 6:50 PM	2	16.9762	6:58 PM	7:06 PM	428.04	19.1163	2.1401	133.8	108.9	12.86
AZ1-Tc-LP3	Col 2	45	15:00	4/25/2002 1:30 AM	2	17.0997	1:38 AM	1:46 AM	534.0	19.2516	2.1519	242.0	196.9	13.01
AZ1-Tc-LP4	Col 2	65	21:40	4/25/2002 8:10 AM	2	16.9451	8:18 AM	8:26 AM	640.2	19.0651	2.1200	350.3	285.0	13.07
AZ1-Tc-LP5	Col 2	85	28:20	4/25/2002 2:50 PM	2	17.0304	2:58 PM	3:06 PM	764.1	19.7302	2.6998	476.9	388.0	13.63
AZ1-Tc-LP6	Col 2	105	35:00	4/25/2002 9:30 PM	2	17.0032	9:38 PM	9:46 PM	885.4	19.1109	2.1077	600.3	488.4	13.90
AZ1-Tc-LP7	Col 2	125	41:40	4/26/2002 4:10 AM	2	17.0137	4:18 AM	4:26 AM	987.7	19.3871	2.3734	704.9	573.6	13.72
AZ1-Tc-LP8	Col 2	140	50:30	4/26/2002 2:10 PM	2	17.0948	2:18 PM	2:26 PM	1163.4	19.3660	2.2712	882.9	718.4	13.87
AZ1-Tc-LP9	Col 2	160	57:10	4/26/2002 8:50 PM	2	17.1273	8:58 PM	9:06 PM	1289.8	19.4671	2.3398	1011.7	823.2	14.08
AZ1-Tc-LP10	Col 2	180	63:50	4/27/2002 3:30 AM	2	17.0981	3:08 AM	3:16 AM	1408	19.4731	2.3750	1132.2	921.3	14.25
AZ1-Tc-LP11	Col 2	200	70:30	4/27/2002 10:10 AM	2	16.9780	10:18 AM	10:26 AM	1529.7	19.4349	2.4569	1256.4	1022.3	14.24
AZ1-Tc-LP12	Col 2	240	83:50	4/27/2002 11:30 PM	2	17.0610	11:38 PM	11:46 PM	1768	19.5954	2.5344	1497.2	1218.2	14.31
AZ1-Tc-LP13	Col 2	280	97:10	4/28/2002 12:50 PM	2	16.9213	1:28 PM	1:36 PM	2031.5	19.4760	2.5547	1763.3	1434.7	16.59

-- indicates the table cell is empty, the lack of data is intentional

Table 3 (continued) Data Collection for Tc Loading of Lag Column in AZ-101 Run

Sample	Sampling Time	#CV	Count date	Count start time	file name	Mass Counted, g	count time, sec	gross counts	Net counts	back ground	Tot cts-bck	AZ1-Tc-0-C	counts of sample/gram
AZ1-Tc-LP0	10:54 AM	--	4/25/2002	9:47 AM	AZ1LP00	2.0116	900	4419	1357	-101	1357	55514	675
AZ1-Tc-LP1	12:26 PM	5	4/25/2002	10:24 AM	AZ1LP01	2.1488	900	4479	1521	-101	1521	55514	708
AZ1-Tc-LP2	7:06 PM	22	4/25/2002	11:36 AM	AZ1LP02	2.1401	900	4797	1908	-101	1908	55514	892
AZ1-Tc-LP3	1:46 AM	39	4/26/2002	8:02 AM	AZ1LP03	2.1519	900	4814	1775	53	1722	51406	800
AZ1-Tc-LP4	8:26 AM	57	4/25/2002	1:32 PM	AZ1LP4	2.1200	900	4596	1439	-101	1439	55514	679
AZ1-Tc-LP5	3:06 PM	78	4/26/2002	9:06 AM	AZ1LP05	2.6998	900	5278	1799	53	1746	51406	647
AZ1-Tc-LP6	9:46 PM	98	4/26/2002	11:02 AM	AZ1LP06	2.1077	900	4612	1511	53	1458	51406	692
AZ1-Tc-LP7	4:26 AM	115	4/26/2002	7:45 AM	AZ1LP07	2.3734	900	5160	1714	53	1661	51406	700
AZ1-Tc-LP8	2:26 PM	144	4/30/2002	9:12 AM	AZ1LP08	2.2712	900	5581	2005	43	1962	56303	864
AZ1-Tc-LP9	9:06 PM	165	4/27/2002	1:52 AM	AZ1LP9	2.3398	900	4808	1659	-23	1659	54992	709
AZ1-Tc-LP10	3:16 AM	184	4/27/2002	4:02 AM	AZ1LP10	2.3750	900	4898	1562	71	1491	51242	628
AZ1-Tc-LP11	10:26 AM	204	4/30/2002	9:29 AM	AZ1LP11	2.4569	900	5853	2223	43	2180	56303	887
AZ1-Tc-LP12	11:46 PM	244	4/28/2002	10:40 AM	AZ1LP12	2.5344	900	5149	1739	-38	1739	49628	686
AZ1-Tc-LP13	1:36 PM	287	4/30/2002	9:49 AM	AZ1LP13	2.5547	900	5945	2066	43	2023	56303	792

Table 3 (continued) Data Collection for Tc Loading of Lag Column in AZ-101 Run

Sample	control counts/ g	C/Co, Tc-95m	Tc-99 Data $\mu\text{Ci/L}$	Dilution Factor	Tc-99 $\mu\text{Ci/L}$	Tc-99 % C/Co	C/Co Out of Lag	C/Co Into Lag	[Tc] into lag, uCi/mL	Vol into lag column, mL	uCi into lag	uCi out of lag
AZ1-Tc-LP0	23,610	2.9%	2.16E-04	1132	0.24	0.07%	0.07%	0.10%	0.000	2.0	0.001	0.00
AZ1-Tc-LP1	23,610	3.0%	2.33E-04	1096	0.25	0.07%	0.07%	0.10%	0.000	22.8	0.01	0.01
AZ1-Tc-LP2	23,610	3.8%	--	--	--	--	0.07%	0.10%	0.000	86.1	0.03	0.02
AZ1-Tc-LP3	21,863	3.7%	2.25E-04	1059	0.24	0.06%	0.06%	0.10%	0.000	88.0	0.03	0.02
AZ1-Tc-LP4	23,610	2.9%	--	--	--	--	0.06%	0.13%	0.000	88.1	0.04	0.02
AZ1-Tc-LP5	21,863	3.0%	2.24E-04	1074	0.24	0.06%	0.06%	0.23%	0.001	103.0	0.09	0.02
AZ1-Tc-LP6	21,863	3.2%	--	--	--	--	0.06%	0.37%	0.001	100.4	0.14	0.02
AZ1-Tc-LP7	21,863	3.2%	2.28E-04	1072	0.24	0.07%	0.07%	0.80%	0.003	85.2	0.26	0.02
AZ1-Tc-LP8	23,945	3.6%	--	--	--	--	0.07%	1.76%	0.007	144.8	1.0	0.04
AZ1-Tc-LP9	23,388	3.0%	2.48E-04	1060	0.26	0.07%	0.07%	2.89%	0.011	104.8	1.1	0.03
AZ1-Tc-LP10	21,793	2.9%	--	--	--	--	0.07%	4.61%	0.017	98.1	1.7	0.02
AZ1-Tc-LP11	23,945	3.7%	2.30E-04	1062	0.24	0.07%	0.07%	8.00%	0.030	101.0	2.7	0.02
AZ1-Tc-LP12	21,107	3.3%	--	--	--	--	0.09%	14.47%	0.054	196.0	11	0.07
AZ1-Tc-LP13	23,945	3.3%	4.32E-04	1061	0.46	0.12%	0.12%	24.66%	0.092	216.5	20	0.10
										Sum	37.7	0.42

-- indicates the table cell is empty, the lack of data is intentional

Feed Displacement

Feed: AZ-101 supernate with Cs-137 removed by ion exchange (SL644)
 Resin: SuperLig 639, batch #010227CTC-9-23
 Tracer: Tc-95m
 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 (w/o/hole) = 298.5 g
 NaOH Density = 1.008
 Flow Rate: approx 15 mL/hr or ~3.0 CV/hr
 Start Date and Time: 4/24/2002 8:30 AM
 End Date and Time: 4/29/2002 7:40 PM
 mass AZ101 control, g 2.3513

-- indicates the table cell is empty, the lack of data is intentional
 ~ indicates the #CV is an estimate, the value is slightly larger than the number following (e.g. ~4 is slighter greater than 4 CV)

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

Sample	Source	# CV	Elapsed Time	Projected Date/Time	Vol. (mL)	MT Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial+ Cap Mass (g)	Sample Mass (g)	Approximate Volume mL
0.10 M NaOH Feed Displacement Solution											
AZ1-Tc-FD-L1	Col 1	1	0:00	4/29/2002 8:00 AM	5	16.9009	8:00 AM	8:20 AM	23.0313	6.1304	4.99
AZ1-Tc-FD-L2	Col 1	2	0:20	4/29/2002 8:20 AM	5	16.9650	8:20 AM	8:40 AM	22.9600	5.9950	4.88
AZ1-Tc-FD-L3	Col 1	3	0:40	4/29/2002 8:40 AM	5	16.9917	8:40 AM	9:00 AM	22.4423	5.4506	4.43
AZ1-Tc-FD-P4	Col 1//Eff	4	1:00	4/29/2002 9:00 AM	5	16.9979	9:00 AM	9:20 AM	22.2917	5.2938	4.31
AZ1-Tc-FD-L5	Col 1	~4	--	--	1	16.7208	9:41 AM	9:45 AM	17.7222	1.0014	0.81
AZ1-Tc-FD-P5	Eff	5	1:20	4/29/2002 9:20 AM	5	16.9510	9:45 AM	10:01 AM	21.9126	4.9616	4.04
AZ1-Tc-FD-L6	Col 1	~5	--	--	1	16.6946	10:01 AM	10:05 AM	17.6336	0.9390	0.93
AZ1-Tc-FD-P6	Eff	6	1:40	4/29/2002 9:40 AM	5	16.9724	10:05 AM	10:21 AM	21.4151	4.4427	4.41
AZ1-Tc-FD-L7	Col 1	~6	--	--	1	16.5725	10:21 AM	10:25 AM	17.5350	0.9625	0.95
AZ1-Tc-FD-P7	Eff	7	2:00	4/29/2002 10:00 AM	5	16.9145	10:25 AM	10:41 AM	21.0996	1.3434	1.33
AZ1-Tc-FD-L8	Col 1	~7	--	--	1	16.7266	10:41 AM	10:45 AM	17.6903	0.9637	0.96
AZ1-Tc-FD-P8	Eff	8	2:20	4/29/2002 10:20 AM	5	16.9697	10:45 AM	10:50 AM	18.3045	1.2058	1.20

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

Sample	Calculated # CV's	Count date	Count start time	file name	Mass Counted, g	count time, sec	gross counts	Net counts	back ground
AZ1-Tc-FD-L1	1.0	5/3/2002	8:34 AM	AZ1FDL01	6.1304	925.82	52633	43852	98
AZ1-Tc-FD-L2	2.0	5/3/2002	8:54 AM	AZ1FDL02	5.9950	922.38	42995	35377	98
AZ1-Tc-FD-L3	2.9	5/3/2002	9:49 AM	AZ1FDL03	5.4506	915.82	45054	39505	98
AZ1-Tc-FD-P4	3.7	5/3/2002	10:06 AM	AZ1FD04	5.2938	916.08	16048	10153	98
AZ1-Tc-FD-L5	3.9	4/29/2002	9:45 AM	AZ1FDL5	1.0014	904.74	6835	5179	26
AZ1-Tc-FD-P5	4.7	5/3/2002	10:25 AM	AZ1FD05	4.9616	915.48	9508	3924	98
AZ1-Tc-FD-L6	4.88	4/29/2002	10:05 AM	AZ1FDL6	0.9390	904.54	6218	4598	26
AZ1-Tc-FD-P6	5.8	5/3/2002	10:42 AM	AZ1FD06	4.4427	907.56	4313	1472	98
AZ1-Tc-FD-L7	5.95	4/29/2002	10:25 AM	AZ1FDL7	0.9625	904.66	8549	6819	26
AZ1-Tc-FD-P7	6.2	5/3/2002	10:59 AM	AZ1FD07	4.1851	904.84	2264	433	98
AZ1-Tc-FD-L8	6.41	4/29/2002	10:48 AM	AZ1FDL8	0.9637	905.22	12161	10335	26
AZ1-Tc-FD-P8	6.6	5/3/2002	11:18 AM	AZ1FD08	1.3348	903.94	1619	138	98

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

Sample	Tot cts-bck	AZ1-Tc-0-C	counts/g of sample	control counts/ g	C/Co, Tc-95m	Tc-99 Data $\mu\text{Ci/L}$	Dilution Factor	Tc-99 $\mu\text{Ci/L}$	Tc-99 % C/Co	99Tc Mass Balance
AZ1-Tc-FD-L1	43754	56656	7137	24,096	29.6%	9.59E-02	1022	9.80E+01	26%	feed displacement occurs through FD-L6 and then begins eluting The FD plot is curved, with the elution being polynomial. The area under the curve was hand calculated and determined using graphics program area under curve calculation
AZ1-Tc-FD-L2	35279	56656	5885	24,096	24.4%	7.95E-02	1046	8.32E+01	22%	
AZ1-Tc-FD-L3	39407	56656	7230	24,096	30.0%	9.35E-02	1101	1.03E+02	27%	
AZ1-Tc-FD-P4	10055	56656	1899	24,096	7.9%	2.02E-02	1050	2.12E+01	5.6%	
AZ1-Tc-FD-L5	5153	48509	5146	20,631	24.9%	3.60E-02	2628	9.45E+01	25%	
AZ1-Tc-FD-P5	3826	56656	771	24,096	3.2%	4.54E-04	1065	4.83E-01	0.13%	
AZ1-Tc-FD-L6	4572	48509	4869	20,631	23.6%	3.26E-02	2116	6.89E+01	18.4%	
AZ1-Tc-FD-P6	1374	56656	309	24,096	1.3%	4.65E-04	928	4.32E-01	0.12%	
AZ1-Tc-FD-L7	6793	48509	7058	20,631	34.2%	4.64E-02	2160	1.00E+02	26.7%	
AZ1-Tc-FD-P7	335	56656	80	24,096	0.3%	3.01E-04	991	2.98E-01	0.08%	
AZ1-Tc-FD-L8	10309	48509	10697	20,631	51.9%	7.12E-02	2097	1.49E+02	39.8%	
AZ1-Tc-FD-P8	40	56656	30	24,096	0.1%	1.77E-04	1069	1.89E-01	0.05%	Sum area under curve = 0.739 uCi

B.10

feed displacement continues from AZ1-Tc-FD-L1 through AZ1-Tc-FD-L4 and then elution begins starting at AZ1-Tc-FD-L6

See calculation detail for hand calculation of area under curve- using two triangles and two rectangles to determine the area

Rinse Feed: AZ-101 supernate with Cs-137 removed by ion exchange (SL644)

Resin: SuperLig 639, batch #010227CTC-9-23

Tracer: Tc-95m

Bed volume, Col 1 = 5.0 mL (3.3 cm)

Bed volume, Col 2 = 5.0 mL (3.4 cm)

Flow Rate: approx 15 mL/hr or ~3.0 CV/hr

Start Date and Time: 4/29/2002 11:00 AM

End Date and Time: 4/29/2002 11:40 AM

-- indicates the table cell is empty, the lack of data is intentional

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)	Sample Mass (g)	Count date
AZ1-Tc-RL1	Col 1	1	0:20	4/29/2002 11:00 AM	5	16.9524	11:00 AM	11:20 AM	21.9974	5.045	5/3/2002
AZ1-Tc-RL2	Col 1	1	0:40	4/29/2002 11:20 AM	5	17.0041	11:20 AM	11:40 AM	21.9859	4.9818	5/3/2002

Sample	Count start time	file name	Mass Counted, g	count time, sec	gross counts	Net counts	back ground	Tot cts-bck	AZ1-Tc-0-C	counts/g of sample	control counts/ g
AZ1-Tc-RL1	7:36 AM	AZ1RL1	5.0450	914.76	82603	77379	98	77281	56656	15318	24,096
AZ1-Tc-RL2	7:53 AM	AZ1RL2	5.0335	916.98	99228	93215	98	93117	56656	18499	24,096

Sample	C/Co, Tc-95m	Tc-99 Data uCi/L	Dilution Factor	Tc-99 mCi/L	Tc-99 % C/Co	uCi/mL	Volume, mL	uCi	uCi/mL	Volume, mL	uCi
--	--	--	--	--	--	^{95m} TcO ₄ Mass Balance			⁹⁹ Tc Mass Balance (a)		
AZ1-Tc-RL1	63.6%	0.1898	1040	1.97E+02	52.64%	no data			1.97E-01	5.05	1.0
AZ1-Tc-RL2	76.8%	0.2415	1045	2.52E+02	67.30%	no data			2.52E-01	4.98	1.3
									uCi total rinse	2.3	

(a) rinse collected in a single solution therefore can calculate the Tc eluted directly

data used in spreadsheet calculations

0.375 uCi/mL	Tc-99 control
2.3513	g sample for control

ElutionFeed: H₂O 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)

Flow Rate: 5 mL/hr or 1 CV/hr

Start Date and Time: 4/29/2002 12:30 PM

End Date and Time: 4/30/2002 2:30 PM

Table 6. Data Collection for Tc Elution of Column 1 (lead column) in AZ-101 Run @ 65C

Sample	Source	# CV	Elapsed Time	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial+ Cap Mass (g)	Sample Mass (g)	Count date	Count start time
--------	--------	------	--------------	---------------------	-----------	---------------------	---------------------	-------------------	---------------------------	-----------------	------------	------------------

AZ1-Tc-E1-1	Col 1	0.5	0:00	4/29/2002 12:30 PM	2.5	16.7603	12:30 PM	1:00 PM	19.4481	2.6878	5/2/2002	12:21 PM
AZ1-Tc-E1-2	Col 1	1	0:30	4/29/2002 1:00 PM	2.5	17.0655	1:00 PM	1:30 PM	19.3665	2.3010	5/7/2002	7:22 AM
AZ1-Tc-E1-3	Col 1	1.5	1:00	4/29/2002 1:30 PM	2.5	16.9238	1:30 PM	2:00 PM	19.1857	2.2619	5/2/2002	12:52 PM
AZ1-Tc-E1-4	Col 1	2	1:30	4/29/2002 2:00 PM	2.5	16.8933	2:00 PM	2:30 PM	19.3108	2.4175	5/7/2002	7:34 AM
AZ1-Tc-E1-5	Col 1	2.5	2:00	4/29/2002 2:30 PM	2.5	16.9509	2:30 PM	3:00 PM	19.5185	2.5676	5/2/2002	1:06 PM
AZ1-Tc-E1-6	Col 1	3	2:30	4/29/2002 3:00 PM	2.5	16.9680	3:00 PM	3:30 PM	19.5393	2.5713	5/7/2002	7:42 AM
AZ1-Tc-E1-7	Col 1	3.5	3:00	4/29/2002 3:30 PM	2.5	16.9383	3:30 PM	4:00 PM	19.5446	2.6063	5/2/2002	1:13 PM
AZ1-Tc-E1-8	Col 1	4	3:30	4/29/2002 4:00 PM	2.5	17.0024	4:00 PM	4:30 PM	19.5707	2.5683	5/7/2002	8:05 AM
AZ1-Tc-E1-9	Col 1	4.5	4:00	4/29/2002 4:30 PM	2.5	16.8964	4:30 PM	5:00 PM	19.5368	2.6404	5/2/2002	1:20 AM
AZ1-Tc-E1-10	Col 1	5	4:30	4/29/2002 5:00 PM	2.5	16.9061	5:00 PM	5:32 PM	19.6789	2.7728	5/7/2002	8:22 AM
AZ1-Tc-E1-11	Col 1	5.5	5:00	4/29/2002 5:30 PM	2.5	16.9767	5:32 PM	6:00 PM	19.4868	2.5101	5/2/2002	1:30 PM
AZ1-Tc-E1-12	Col 1	6	5:30	4/29/2002 6:00 PM	2.5	16.9904	6:00 PM	6:30 PM	19.6118	2.6214	5/7/2002	8:56 AM
AZ1-Tc-E1-13	Col 1	6.5	6:00	4/29/2002 6:30 PM	2.5	16.9278	6:30 PM	7:00 PM	19.4704	2.5426	5/2/2002	1:38 PM
AZ1-Tc-E1-14	Col 1	7	6:30	4/29/2002 7:00 PM	2.5	17.0450	7:00 PM	7:30 PM	19.3984	2.3534	5/7/2002	9:05 AM

B.12

Table 6 (continued). Data Collection for Tc Elution of Column 1 (lead column) in AZ-101 Run @ 65C

Sample	file name	Volume Counted	Counting aliquot, mL	Diluent: Water, mL	Dilution Factor	count time, sec	gross counts	Net counts	back ground	Tot cts-bck	AZ1-Tc-0-C	final counts	counts/ mL of sample
Counting Dilution Data													
AZ1-Tc-E1-1	AZ1E01d	50 µL	0.0496	1.9856	41	301	2848	2221	-1	5971	18658	244,987	120375
AZ1-Tc-E1-2	AZ1E2DA	50 µL	0.0433	1.9402	46	303	15957	14813	35	14778	17562	676,955	341293
AZ1-Tc-E1-3	AZ1E3DA	50 µL	0.0498	1.9856	41	307	42095	39650	-1	39651	18658	1,620,595	796205
AZ1-Tc-E1-4	AZ1E4DA	50 µL	0.0472	1.9392	42	307	42761	40580	35	40545	17562	1,706,326	859004
AZ1-Tc-E1-5	AZ1E5DA	50 µL	0.0491	1.9426	41	304	20684	19372	-1	19373	18658	785,849	394562
AZ1-Tc-E1-6	AZ1E6DA	50 µL	0.0474	1.9393	42	307	42761	40580	35	40545	17562	1,699,383	855380
AZ1-Tc-E1-7	AZ1EDA	50 µL	0.0495	1.9158	40	303	17054	15918	-1	15919	18658	632,033	321596
AZ1-Tc-E1-8	AZ1E8DA	50 µL	0.0450	1.9397	44	304	17824	16589	35	16554	17562	730,105	367867
AZ1-Tc-E1-9	AZ1E9DA	50 µL	0.0480	1.9412	41	304	18590	17414	-1	17415	18658	721,707	362813
AZ1-Tc-E1-10	AZ1E10DA	50 µL	0.0459	1.9367	43	303	17317	16124	35	16089	17562	694,947	350523
AZ1-Tc-E1-11	AZ1E11DA	50 µL	0.0501	1.9293	40	304	21333	19999	-1	20000	18658	790,180	399202
AZ1-Tc-E1-12	AZ1E12DA	50 µL	0.0470	1.9361	42	304	18960	17699	35	17664	17562	745,308	375830
AZ1-Tc-E1-13	AZ1E13DA	50 µL	0.0496	1.9464	40	306	18344	17201	-1	17202	18658	692,242	346815
AZ1-Tc-E1-14	AZ1E14DA	50 µL	0.0452	1.9422	44	303	10503	9693	35	9658	17562	424,653	213673

B.13

Data used in spreadsheet calculations

TcO4- initial feed	0.378	uCi/mL
Tc-99 initial feed	0.375	uCi/mL

Table 6 (continued). Data Collection for Tc Elution of Column 1 (lead column) in AZ-101 Run @ 65C

Sample	control counts/mL	%C/Co Tc-95m	C/Co Tc-95m	Tc-99 Data μ Ci/L	Dilution Factor	Tc-99 μ Ci/L	Tc-99 C/Co	uCi/mL	Volume, mL	uCi	uCi/mL	Volume, mL	uCi
								^{95m} TcO ₄ Mass Balance			⁹⁹ Tc Mass Balance		
AZ1-Tc-E1-1	9,752	1234%	12	1.79E+00	1055	1.88E+03	5	4.67E+00	2.69	1.25E+01	1.88E+00	2.69	5
AZ1-Tc-E1-2	9,179	3718%	37	--	--	--	43	1.41E+01	2.30	3.23E+01	1.61E+01	2.30	37
AZ1-Tc-E1-3	9,752	8164%	82	2.82E+01	1077	3.04E+04	81	3.09E+01	2.26	6.98E+01	3.04E+01	2.26	68.7
AZ1-Tc-E1-4	9,179	9358%	94	--	--	--	65	3.54E+01	2.42	8.55E+01	2.43E+01	2.42	59
AZ1-Tc-E1-5	9,752	4046%	40	1.70E+01	1076	1.83E+04	49	1.53E+01	2.57	3.93E+01	1.83E+01	2.57	47
AZ1-Tc-E1-6	9,179	9318%	93	--	--	--	42	3.52E+01	2.57	9.06E+01	1.57E+01	2.57	40
AZ1-Tc-E1-7	9,752	3298%	33	1.30E+01	1011	1.32E+04	35	1.25E+01	2.61	3.25E+01	1.32E+01	2.61	34
AZ1-Tc-E1-8	9,179	4007%	40	--	--	--	36	1.51E+01	2.57	3.89E+01	1.35E+01	2.57	35
AZ1-Tc-E1-9	9,752	3720%	37	1.34E+01	1030	1.38E+04	37	1.41E+01	2.64	3.71E+01	1.38E+01	2.64	37
AZ1-Tc-E1-10	9,179	3819%	38	--	--	--	39	1.44E+01	2.77	4.00E+01	1.47E+01	2.77	41
AZ1-Tc-E1-11	9,752	4093%	41	1.52E+01	1024	1.56E+04	42	1.55E+01	2.51	3.88E+01	1.56E+01	2.51	39
AZ1-Tc-E1-12	9,179	4094%	41	--	--	--	39	1.55E+01	2.62	4.06E+01	1.45E+01	2.62	38
AZ1-Tc-E1-13	9,752	3556%	36	1.32E+01	1011	1.34E+04	36	1.34E+01	2.54	3.42E+01	1.34E+01	2.54	34
AZ1-Tc-E1-14	9,179	2328%	23	--	--	--	23	8.80E+00	2.35	2.07E+01	8.45E+00	2.35	20

-- indicates the table cell is empty, the lack of data is intentional

Table 6. Data Collection for Tc Elution of Column 1 (lead column) in AZ-101 Run @ 65C

Sample	Source	# CV	Elapsed Time	Projected Date/Time	Vol. (mL)	Vial + Cap Mass (g)	Sampling Start Time	Sampling End Time	Sample+Vial+ Cap Mass (g)	Sample Mass (g)	Count date	Count start time
AZ1-Tc-E1-15	Col 1	7.5	7:00	4/29/2002 7:30 PM	2.5	17.1084	7:30 PM	8:00 PM	19.3607	2.2523	5/2/2002	1:46 PM
AZ1-Tc-E1-16	Col 1	8	7:30	4/29/2002 8:00 PM	2.5	16.9839	8:00 PM	8:30 PM	19.3734	2.3895	4/29/2002	2:25 AM
AZ1-Tc-E1-17	Col 1	8.5	8:00	4/29/2002 8:30 PM	2.5	16.9740	8:30 PM	9:00 PM	19.3913	2.4173	5/1/2002	9:10 AM
AZ1-Tc-E1-18	Col 1	9	8:30	4/29/2002 9:00 PM	2.5	16.7726	9:00 PM	9:30 PM	19.1320	2.3594	5/1/2002	8:46 AM
AZ1-Tc-E1-19	Col 1	9.5	9:00	4/29/2002 9:30 PM	2.5	16.8726	9:30 PM	10:00 PM	19.2901	2.4175	5/1/2002	8:29 AM
AZ1-Tc-E1-20	Col 1	10	9:30	4/29/2002 10:00 PM	2.5	16.9351	10:00 PM	11:00 PM	22.1265	5.1914	5/1/2002	8:13 AM
AZ1-Tc-E1-21	Col 1	11	10:30	4/29/2002 11:00 PM	5	16.9798	11:00 PM	12:00 PM	22.0000	5.0202	5/1/2002	7:55 AM
AZ1-Tc-E1-22	Col 1	12	11:30	4/30/2002 12:00 AM	5	17.0626	12:00 PM	1:00 AM	22.3759	5.3133	5/1/2002	7:38 AM
AZ1-Tc-E1-23	Col 1	13	12:30	4/30/2002 1:00 AM	5	17.0867	1:00AM	2:00 AM	22.3349	5.2482	4/30/2002	12:12 PM
AZ1-Tc-E1-24	Col 1	14	13:30	4/30/2002 2:00 AM	5	16.9632	2:00 AM	3:00 AM	22.0308	5.0676	4/30/2002	11:56 AM
AZ1-Tc-E1-25	Col 1	15	14:30	4/30/2002 3:00 AM	5	17.0113	3:00 AM	4:00 AM	22.1878	5.1765	4/30/2002	11:04 AM
AZ1-Tc-E1-26	Col 1	16	15:30	4/30/2002 4:00 AM	5	16.9437	4:00 AM	5:00 AM	22.0463	5.1026	4/30/2002	10:45 AM
AZ1-Tc-E1-27	Col 1	17	16:30	4/30/2002 5:00 AM	5	17.0831	5:00 AM	6:00 AM	22.0306	4.9475	4/30/2002	10:27 AM
AZ1-Tc-E1-28	Col 1	18	17:30	4/30/2002 6:00 AM	5	17.0925	6:00 AM	7:00 AM	21.8566	4.7641	4/30/2002	7:48 AM
AZ1-Tc-E1-29	Col 1	19	18:30	4/30/2002 7:00 AM	5	16.7806	7:00 AM	8:00 AM	21.5367	4.7561	4/30/2002	10:09 AM
AZ1-Tc-E1-30	Col 1	20	19:30	4/30/2002 8:00 AM	5	16.8831	8:00 AM	9:00 AM	21.5036	4.6205	4/30/2002	1:53 PM
AZ1-Tc-E1-31	Col 1	21	20:30	4/30/2002 9:00 AM	5	16.9334	9:00 AM	10:00 AM	21.5489	4.6155	4/30/2002	1:03 PM
AZ1-Tc-E1-32	Col 1	22	21:30	4/30/2002 10:00 AM	5	16.9918	10:00 AM	11:00 AM	21.5898	4.5980	4/30/2002	12:46 PM
AZ1-Tc-E1-33	Col 1	23	22:30	4/30/2002 11:00 AM	5	17.0025	11:00 AM	12:00 PM	21.6510	4.6485	4/30/2002	12:29 PM
AZ1-Tc-E1-34	Col 1	24	23:30	4/30/2002 12:00 PM	5	17.0433	12:00 PM	1:00 PM	21.6864	4.6431	4/30/2002	1:19 PM
AZ1-Tc-E1-35	Col 1	25	24:30	4/30/2002 1:00 PM	5	16.9297	1:00 PM	1:30 PM	19.2257	2.2960	4/30/2002	1:37 PM
AZ1-Tc-E1-36	Col 1	26	25:00	4/30/2002 1:30 PM	5	16.9771	1:30 PM	2:00 PM	19.3898	2.4127	4/30/2002	2:10 PM
AZ1-Tc-E1-37	Col 1	27	25:30	4/30/2002 2:00 PM	5	16.9987	2:00 PM	2:30 PM	19.4655	2.4668	4/30/2002	2:33 PM
AZ1-Tc-E1-38	Col 1	28	26:00	4/30/2002 2:30 PM	5	16.9681	2:30 PM	3:00 PM	19.3450	2.3769	4/30/2002	3:02 PM

Table 6 (continued). Data Collection for Tc Elution of Column 1 (lead column) in AZ-101 Run @ 65C

Sample	file name	Volume Counted	Counting aliquot, mL	Diluent: Water, mL	Dilution Factor	count time, sec	gross counts	Net counts	back ground	Tot cts-bck	AZ1-Tc-0-C	final counts	counts/ mL of sample
AZ1-Tc-E1-15	AZ1E15DA	50 µL	0.0496	1.9321	40	302	5008	4367	-1	4368	18658	174,517	88065
AZ1-Tc-E1-16	AZ1E16	full sample	--	--	--	926	160981	160981	40	160941	55593	160,941	67353
AZ1-Tc-E1-17	AZ1E17	full sample	--	--	--	909	39327	36223	11	36212	51246	36,212	14980
AZ1-Tc-E1-18	AZ1E18	full sample	--	--	--	905	12501	10533	11	10522	51246	10,522	4460
AZ1-Tc-E1-19	AZ1E19	full sample	--	--	--	904	6185	4400	11	4389	51246	4,389	1816
AZ1-Tc-E1-20	AZ1E20	full sample	--	--	--	905	7422	5699	11	5688	51246	5,688	1096
AZ1-Tc-E1-21	AZ1E21	full sample	--	--	--	904	5556	3834	11	3823	51246	3,823	762
AZ1-Tc-E1-22	AZ1E22	full sample	--	--	--	904	4227	2581	11	2570	51246	2,570	484
AZ1-Tc-E1-23	AZ1E23	full sample	--	--	--	904	4353	2598	43	2555	56303	2,555	487
AZ1-Tc-E1-24	AZ1E24	full sample	--	--	--	904	3743	2116	43	2073	56303	2,073	409
AZ1-Tc-E1-25	AZ1E25	full sample	--	--	--	904	3487	1874	43	1831	56303	1,831	354
AZ1-Tc-E1-26	AZ1E26	full sample	--	--	--	907	3276	1821	43	1778	56303	1,778	348
AZ1-Tc-E1-27	AZ1E27	full sample	--	--	--	904	2883	1402	43	1359	56303	1,359	275
AZ1-Tc-E1-28	AZ1E28	full sample	--	--	--	904	2701	1151	43	1108	56303	1,108	233
AZ1-Tc-E1-29	AZ1E29	full sample	--	--	--	904	2546	1113	43	1070	56303	1,070	225
AZ1-Tc-E1-30	AZ1E30	full sample	--	--	--	904	2715	1073	43	1030	56303	1,030	223
AZ1-Tc-E1-31	AZ1E31	full sample	--	--	--	904	2245	753	43	710	56303	710	154
AZ1-Tc-E1-32	AZ1E32	full sample	--	--	--	904	2198	842	43	799	56303	799	174
AZ1-Tc-E1-33	AZ1E33	full sample	--	--	--	904	2036	541	43	498	56303	498	107
AZ1-Tc-E1-34	AZ1E34	full sample	--	--	--	904	2205	749	43	706	56303	706	152
AZ1-Tc-E1-35	AZ1E35	full sample	--	--	--	904	1823	390	43	347	56303	347	151
AZ1-Tc-E1-36	AZ1E36	full sample	--	--	--	904	1874	331	43	288	56303	288	119
AZ1-Tc-E1-37	AZ1E37	full sample	--	--	--	904	1905	450	43	407	56303	407	165
AZ1-Tc-E1-38	AZ1E38	full sample	--	--	--	904	1709	294	43	251	56303	251	106

-- indicates the table cell is empty, the lack of data is intentional

Table 6 (continued). Data Collection for Tc Elution of Column 1 (lead column) in AZ-101 Run @ 65C

Sample	control counts/mL	%C/Co Tc-95m	C/Co Tc-95m	Tc-99 Data $\mu\text{Ci/L}$	Dilution Factor	Tc-99 $\mu\text{Ci/L}$	Tc-99 C/Co	uCi/mL	Volume, mL	uCi	uCi/mL	Volume, mL	uCi
AZ1-Tc-E1-15	9,752	903%	9.0	3.42E+00	1032	3.53E+03	9.4	3.41E+00	2.25	7.69E+00	3.53E+00	2.25	7.9
AZ1-Tc-E1-16	29,058	232%	2.3	--	--	--	5.0	8.76E-01	2.39	2.09E+00	1.87E+00	2.39	4.5
AZ1-Tc-E1-17	26,786	56%	0.6	2.06E-01	1039	2.14E+02	0.57	2.11E-01	2.42	5.11E-01	2.14E-01	2.42	0.5
AZ1-Tc-E1-18	26,786	17%	0.2	--	--	--	0.32	6.29E-02	2.36	1.48E-01	1.19E-01	2.36	0.280
AZ1-Tc-E1-19	26,786	6.8%	0.07	2.30E-02	1026	2.36E+01	0.06	2.56E-02	2.42	6.19E-02	2.36E-02	2.42	0.057
AZ1-Tc-E1-20	26,786	4.1%	0.04	--	--	--	0.04	1.55E-02	5.19	8.03E-02	1.65E-02	5.19	0.086
AZ1-Tc-E1-21	26,786	2.8%	0.03	9.40E-03	1011	9.50E+00	0.03	1.07E-02	5.02	5.40E-02	9.50E-03	5.02	0.048
AZ1-Tc-E1-22	26,786	1.8%	0.02	--	--	--	0.02	6.83E-03	5.31	3.63E-02	7.72E-03	5.31	0.041
AZ1-Tc-E1-23	29,429	1.7%	0.02	5.88E-03	1010	5.94E+00	0.02	6.25E-03	5.25	3.28E-02	5.94E-03	5.25	0.031
AZ1-Tc-E1-24	29,429	1.4%	0.01	--	--	--	0.01	5.25E-03	5.07	2.66E-02	5.26E-03	5.07	0.027
AZ1-Tc-E1-25	29,429	1.2%	0.01	4.50E-03	1019	4.58E+00	0.01	4.54E-03	5.18	2.35E-02	4.58E-03	5.18	0.024
AZ1-Tc-E1-26	29,429	1.2%	0.01	--	--	--	0.01	4.48E-03	5.10	2.28E-02	3.97E-03	5.10	0.020
AZ1-Tc-E1-27	29,429	0.9%	0.01	3.22E-03	1044	3.36E+00	0.01	3.53E-03	4.95	1.75E-02	3.36E-03	4.95	0.017
AZ1-Tc-E1-28	29,429	0.8%	0.01	--	--	--	0.01	2.99E-03	4.76	1.42E-02	3.03E-03	4.76	0.014
AZ1-Tc-E1-29	29,429	0.8%	0.01	2.61E-03	1035	2.70E+00	0.01	2.89E-03	4.76	1.37E-02	2.70E-03	4.76	0.013
AZ1-Tc-E1-30	29,429	0.8%	0.01	--	--	--	0.01	2.86E-03	4.62	1.32E-02	2.47E-03	4.62	0.011
AZ1-Tc-E1-31	29,429	0.5%	0.01	2.22E-03	1011	2.24E+00	0.01	1.98E-03	4.62	9.12E-03	2.24E-03	4.62	0.010
AZ1-Tc-E1-32	29,429	0.6%	0.01	--	--	--	0.01	2.23E-03	4.60	1.03E-02	2.01E-03	4.60	0.009
AZ1-Tc-E1-33	29,429	0.4%	0.004	1.72E-03	1038	1.78E+00	0.005	1.38E-03	4.65	6.40E-03	1.78E-03	4.65	0.008
AZ1-Tc-E1-34	29,429	0.5%	0.01	--	--	--	0.005	1.95E-03	4.64	9.07E-03	1.81E-03	4.64	0.008
AZ1-Tc-E1-35	29,429	0.5%	0.01	1.84E-03	997	1.83E+00	0.005	1.94E-03	2.30	4.46E-03	1.83E-03	2.30	0.004
AZ1-Tc-E1-36	29,429	0.4%	0.004	1.46E-03	1023	1.50E+00	0.004	1.53E-03	2.41	3.70E-03	1.50E-03	2.41	0.004
AZ1-Tc-E1-37	29,429	0.6%	0.01	2.05E-03	1002	2.05E+00	0.005	2.12E-03	2.47	5.23E-03	2.05E-03	2.47	0.005
AZ1-Tc-E1-38	29,429	0.4%	0.004	1.23E-03	976	1.20E+00	0.003	1.36E-03	2.38	3.22E-03	1.20E-03	2.38	0.003
								Sum	131 mL	624 uCi	Sum	131 mL	548 uCi
													32 mg

B.17

-- indicates the table cell is empty, the lack of data is intentional

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)

Flow Rate: 5 mL/hr or 1 CV/hr

Start Date and Time: 5/3/2002 7:10 AM

End Date and Time: 5/3/2002 2:30 PM

Information used in spreadsheet calculations

Mass of feed control counted	2.3513	g
density 0.25 M NaOH	1.014	g/mL
Tc-99 initial feed	0.0745	uCi/mL
Tc-99 initial feed (mass balance)	0.375	uCi/mL

Table 6. Data Collection for Regeneration of Column 1 (lead column) in AZ-101 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)	Sample Mass (g)	Count date
AZ1-Tc-Regen-1	Feed bot.	4	4 hrs	5/3/2002 7:10 AM	20	16.9065	7:10 AM	11:10 AM	34.9320	18.0255	5/6/2002
AZ1-Tc-Regen-2	Feed bot.	4	4 hrs	5/3/2002 11:10 AM	20	16.9547	11:10 AM	2:30 PM	35.1675	18.2128	5/6/2002

Count start time	file name	Mass Counted, g	count time, sec	gross counts	Net counts	back ground	Tot cts-bck	AZ1-Tc-0-C	counts/g of sample	control counts/ g	C/Co, Tc-95m
8:59 AM	AZ1REG1	18.0255	904.98	4326	2537	45	2492	47790	138	20,325	0.68%
2:00 PM	AZ1REG2	18.2128	904.68	1815	-43	45	-88	47790	-5	20,325	-0.02%

Sample	Tc-99 Data $\mu\text{Ci/L}$	Dilution Factor	Tc-99 $\mu\text{Ci/L}$	Tc-99 % C/Co	uCi/mL	Volume, mL	uCi	uCi/mL	Volume, mL	uCi
--	--	--	--	--	$^{95\text{m}}\text{TcO}_4$ Mass Balance			^{99}Tc Mass Balance		
AZ1-Tc-Regen-1	0.00142	968	1.38	0.37%				1.38E-03	17.78	0.0245
AZ1-Tc-Regen-2	0.00385	6	0.02	0.01%				2.31E-05	17.96	0.0004
								Sum	0.0249 uCi	

-- indicates the table cell is empty, the lack of data is intentional

Distribution

**No. of
Copies**

**No. of
Copies**

OFFSITE

ONSITE

5 Savannah River Technology Center

Larry Hamm
PO Box 616, Road 1
Building 773-42A
Aiken, South Carolina 29808

Neguib Hassan
PO Box 616, Road 1
Building 773-A
Aiken, South Carolina 29808

Jim Marra
PO Box 616, Road 1
Building 773-43A
Aiken, South Carolina 29808

Charles Nash
PO Box 616, Road 1
Building 773-42A
Aiken, South Carolina 29808

Harold Sturm
PO Box 616, Road 1
Building 773-A
Aiken, South Carolina 29808

14 Battelle, Pacific Northwest Division

S. T. Arm P7-25
I. E. Burgeson (3) P7-25
D.L. Blanchard, Jr. P7-25
J. R. Deschane P7-25
O. B. Egorov P7-22
S. K. Fiskum P7-25
D.E. Kurath P7-28
B. M. Rapko P7-25
Project Office (2) P7-28
Information Release (2) K1-06

2 Bechtel National, Inc.

J. F. Doyle H4-02
R. A. Peterson H4-02