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WTP-RPT-026, Rev 0

Equilibrium Batch Contact Testing of SuperLig® 639

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March 2003

Prepared for the Bechtel National, Inc.
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Equilibrium Batch Contact Testing Of SuperLig® 639:

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Test specification: N/A
Test plan: CHG-TP-41500-014, Rev. 0
Test exceptions: None
R&T Focus Area: Pretreatment
Test Scoping Statement(s): B-50

Battelle, Pacific Northwest Division
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by CHG-TP-41500-014, 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 Date
WTP R&T Support Project

_____ _____

G. Todd Wright, Manager Date
Research and Technology

Summary

Objectives

Battelle, Pacific Northwest Division (PNWD) is contracted to Bechtel National Inc. (BNI) on the River Protection Project – Waste Treatment Plant (RPP-WTP) project to perform research and development activities. Unit operations of the WTP process include the separation of ^{99}Tc by ion exchange from the liquid portion of the waste. The SuperLig® 639 (SL-639) ion exchange resin was selected by the project to perform this ^{99}Tc separation.

The RPP-WTP is evaluating the SL-639 resin to determine its behavior under various potential processing conditions. Under test scoping statements S-142 and S-143, a model is being developed at the Savannah River Technical Center (SRTC) that will be able to predict the response of SL-639 under various processing conditions. This investigation was conducted according to the test plan prepared by Rapko (2001) in response to the test requirements to investigate ion exchange resin degradation delineated by Barnes et al. (2002) in Section 3.7.2.1 of the Research and Technology Plan and test scoping statement B-50. The results from these tests will be incorporated into the afore-mentioned model. Objectives as noted in test scoping statement B-50 were to:

- evaluate the impact of the solution's nitrate to pertechnetate ratio on the SL-639 distribution ratio
- evaluate the impact of the solution's chloride to pertechnetate ratio on the SL-639 distribution ratio
- evaluate the impact of the solution's ionic strength on the SL-639 distribution ratio
- evaluate the impact of temperature and time on the SL-639 distribution ratio.

These results were achieved.

Conduct of Testing

A batch of SuperLig® 639 was preconditioned by contact with an alkaline solution at 5 M sodium. Excess hydroxide and sodium were removed by repeated contacts with deionized water, and the resin was dried to a constant weight. Approximately 0.1 g of resin was contacted with 10 mL of a pertechnetate (^{99}Tc)-containing test solution. Sample aliquots were taken before and after contact with the resin, and their activity was analyzed by liquid scintillation counting.

Results and Performance Against Objectives

The variables tested and a summary of key findings are given below:

1) Room-temperature kinetic measurements were performed under the experimental conditions used for subsequent batch contacts. The primary purpose behind this measurement was to verify that subsequent room-temperature batch contacts were performed under equilibrium conditions. It was determined from the results reported in Section 3.1 that under the experimental conditions employed for these batch contacts, in particular the speed (225 rpm) used with this rotary shaker, that 48 h would be sufficient to achieve equilibrium. Contact times of 72 h were used in subsequent measurements to assure that these measurements were made under equilibrium conditions.

2) Elevated (65°C) kinetic measurements were performed under the experimental conditions used for subsequent batch contacts. At high (5 M) sodium concentrations, the targeted concentration for treated Hanford tank solutions by the SuperLig® 639 resin, the results reported in Section 3.2 indicate that the system reaches equilibrium at 65°C, the temperature proposed for elution of the SuperLig® 639 resin, much more rapidly than at room temperature, with the system achieving a distribution value within 4 h that remained unchanged even if contacted for several additional days. For consistency's sake with the room-temperature measurements, 72 h was used for subsequent measurements made at 65°C. As described in Section 3.3, at 65°C and low, approximately 2 mM, sodium concentrations, the pertechnetate distribution values were so low that no decrease in pertechnetate concentration was observed (a K_d of effectively zero). Such low K_d values are a desirable feature for the resin under elution conditions.

3) Pertechnetate K_d measurements were made as a function of $[NO_3^-]/[TcO_4^-]$ at room temperature. In Section 3.4, the pertechnetate K_d is reported as a function of the equilibrium nitrate/pertechnetate ratio over a range of 10^6. In addition, pertechnetate measurements on an envelope A supernatant, AN-105, simulant, as well as selected perrhenate K_d measurements, were reported. The most striking features are: 1) the enhanced K_ds for perrhenate and pertechnetate observed in the AN-105 simulant versus the simple, 5 M $[Na^+]$ simulant and 2) the approximately 50% lower perrhenate K_d as compared to the pertechnetate K_d. The cause of the enhanced K_ds is unclear, but extraction of pertechnetate as an ion-pair coupled with the presence of a more tightly binding cation (such as potassium) in the AN-105 simulant or a slightly higher sodium concentration in the AN-105 simulant are possible explanations.

4) Pertechnetate K_d measurements were made as a function of $[NO_3^-]/[TcO_4^-]$ at elevated temperature (65°C). In Section 3.5, the same scope of measurements performed in Section 3.4 is repeated, but with the test samples at 65°C. The same trends noted above are also found at 65°C, but the distribution values are substantially decreased.

5) Pertechnetate K_d measurements were studied as a function of changing the solution's ionic strength. In Section 3.7, the impact of changing the salt concentration on the pertechnetate distribution value was examined. A strong linear correlation of the pertechnetate distribution value to both the total sodium concentration and to the solution's ionic strength was found. In this test, those two factors are so closely related that it cannot be said with certainty which

solution property is impacting the pertechnetate distribution value. If the ionic strength effect is indeed the controlling factor, the magnitude of the response is remarkable. However, the resin has been reported to operate by extracting a sodium pertechnetate ion pair. Consistent with that interpretation, these test results simply illustrate the resin affinity for sodium ion.

6) A measurement was made to evaluate the impact of varying the competing anions chloride and hydroxide on the pertechnetate K_d. In Section 3.6, the pertechnetate distribution value was measured as the chloride and hydroxide ratios were changed while keeping the solution's sodium concentration constant. The results indicate that hydroxide competes more effectively than chloride with pertechnetate for binding to SuperLig® 639.

For each set of test results, the distribution values are presented both in a table and graphically.

Quality Requirements

PNWD implemented the RPP-WTP quality requirements in a quality assurance project plan (QAPjP) as approved by the RPP-WTP QA organization. This work was conducted to the quality requirements in NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, Subpart 2.7, as instituted through PNWD's Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP).

PNWD addressed data-verification activities by conducting an independent technical review of the final data report in accordance with Procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the Test Plan objectives.

Issues

No WTP design or operational issues have been identified.

References

Barnes S, R Roosa, and R Peterson. 2002. *Research Technology Plan,* 24590-WTP-PL-RT-01-002, Rev. 1. River Protection Project, Richland, Washington.

Rapko BM. 2001. "Equilibrium Batch Contact Testing for CH2MHill Inc." CHG-TP-41500-014, Pacific Northwest National Laboratory, Richland, Washington.

Acronyms

DI	deionized water
HLW	high-level waste
ICP-MS	inductively-coupled plasma mass spectroscopy
LAW	low-activity waste
LSC	liquid scintillation counting
PNWD	Battelle Pacific Northwest Division
QAPjP	quality assurance project plan
RPP-WTP	River Protection Project – Waste Treatment Plant
SRTC	Savannah River Technology Center
WTPSP	Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual

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1.0 Introduction

1.1 Background

The U.S. Department of Energy plans to vitrify the waste from the 177 underground storage tanks at the Hanford Site. Vitrification will immobilize the tank waste for permanent disposal. In the proposed facility for immobilizing tank waste, the incoming tank waste will be initially split into a low-activity waste (LAW) solution stream and a high-level waste (HLW) solids steam for separate vitrification. To render the solution stream suitable for LAW vitrification, additional processing to remove radioactive cesium and technetium will be required (Barnes et al. 2002). Much recent work has been performed investigating the use of two ion exchange resins developed by IBC Advanced Technologies, Inc. for use in removing selected radionuclides from Hanford tank waste supernatants. Superlig® 639 has been and is being investigated for removing technetium (as pertechnetate) from alkaline, high sodium solutions, including actual Hanford tank supernatants (Hamm et al. 2000; Blanchard et al. 2000). Existing data to date using both simulants and actual tank wastes have been evaluated and included in an ion exchange process model under continuing development at Savannah River Technology Center (SRTC) (Hamm et al. 2000).

It has been shown that batch-contact distribution data collected from simple binary systems can be incorporated in such a process model for use in more complex systems (Mehablia et al. 1994). Incorporating such information from previous data is difficult because of changing conditions amongst prior testing with SuperLig® 639, such as differing resin batches and varying experimental designs (Hamm et al. 2000). A need for additional, equilibrium-batch-contact data has been identified in order to continue to develop and extend the modeling for use under likely process conditions (Hamm et al. 2000; Barnes et al. 2002). The work covered in this report measures a subset of the desired binary equilibrium-batch-contact data together with the appropriate controls (such as the batch-distribution kinetics) required to interpret the batch equilibrium data for Superlig® 639.

1.2 Objectives

Battelle, Pacific Northwest Division (PNWD) is contracted to Bechtel National Inc. (BNI) on the River Protection Project – Waste Treatment Plant (RPP-WTP) project to perform research and development activities. Unit operations of the WTP process include the separation of ^{99}Tc by ion exchange from the liquid portion of the waste. The SuperLig® 639 (SL-639) ion exchange resin was selected by the project to perform ^{99}Tc separations.

The RPP-WTP is evaluating the SL-639 resin to determine its behavior under various potential processing conditions. Under test scoping statements S-142 and S-143, a model is being developed at the Savannah River Technical Center (SRTC) that will be able to predict the response of SL-639 under various processing conditions. This investigation was conducted according to the test plan prepared by Rapko (2001) in response to the test requirements to investigate ion exchange resin degradation delineated by Barnes et al. (2002) in Section 3.7.2.1 of the Research and Technology Plan and test scoping statement B-50. The results from these tests will be incorporated into the afore-mentioned model.

Objectives as noted in test scoping statement B-50 were to:

- evaluate the impact of the solution's nitrate to pertechnetate ratio on the SL-639 distribution ratio
- evaluate the impact of the solution's chloride to pertechnetate ration on the SL-639 distribution ratio
- evaluate the impact of the solution's ionic strength on the SL-639 distribution ratio
- evaluate the impact of temperature and time on the SL-639 distribution ratio

1.3 Purpose

This report documents testing, results, and analysis associated with the measurement of kinetic and equilibrium batch contacts involving SL-639. The report is intended to aid the RPP-WTP project in developing the computer model to be used to predict performance of the Tc ion exchange system in the WTP over a wide variety of conditions.

1.4 Quality Assurance

PNWD implemented the RPP-WTP quality requirements in a quality assurance project plan (QAPjP) as approved by the RPP-WTP QA organization. This work was conducted to the quality requirements in NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, Subpart 2.7 as instituted through PNWD's Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP).

PNWD addressed data-verification activities by conducting an independent technical review of the final data report in accordance with Procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the Test Plan objectives.

2.0 Experimental

SuperLig® 639 was obtained from IBC Advanced Technologies, Inc., American Fork, Utah. The resin batch used for all testing in this report was # 010227 CTC-9-23. The material appears visually as beige beads of varying sizes. No attempt was made to sieve the material to a uniform particle size or to measure the particle size distribution of the resin.

2.1 Preparation of Non-Radioactive Stock Solutions

Chemicals were obtained from standard commercial sources unless indicated otherwise. Deionized (DI) water was purified by passing distilled water through a commercial water purifier and was stored in plastic containers until used. The hydroxide concentrations of the simple sodium hydroxide/sodium nitrate/sodium chloride and Hanford Tank 241-AN-105 simulant solutions used in this work were verified by titration by Analytical Chemistry Laboratory personnel in the 325 Building.

The AN-105 simulant (2 L) was prepared as follows:

1) 200 g of water were placed in a tared 2-L volumetric flask.
2) The following reagents (to a precision of at least ± 1 mg) were added as indicated in Table 2.1a:

Table 2.1a. Reagents Added to Simulant

Compound	Compound Formula	Target Mass (g)	Measured Mass (g)	Simulant Target M
Boric Acid	H_3BO_3	0.292	0.292	2.36E-03
Cadmium Nitrate	$Cd(NO_3)_2$-$4H_2O$	0.009	0.009	1.47E-05
Calcium Nitrate	$Ca(NO_3)_2$-$4H_2O$	0.236	0.236	4.99E-04
Lead Nitrate	$Pb(NO_3)_2$	0.085	0.086	2.18E-04
Potassium Nitrate	KNO_3	19.221	19.222	9.51E-02
Magnesium Nitrate	$Mg(NO_3)_2$-$6H_2O$	0.057	0.057	1.11E-04
Silver Nitrate	$AgNO_3$	0.026	0.026	7.56E-05
Zinc Nitrate	$Zn(NO_3)_2$-$6H_2O$	0.046	0.047	7.72E-05
Glycolic Acid	$HOCH_2COOH$, 70 wt. %	1.665	1.666	1.09E-02
Sodium Chloride	NaCl	14.984	14.985	1.28E-01
Sodium Fluoride	NaF	0.420	0.421	5.00E-03
Sodium Chromate	Na_2CrO_4-$4H_2O$	6.086	6.088	1.30E-02
Sodium Sulfate	Na_2SO_4	1.140	1.140	4.01E-03
Potassium Molydate	K_2MoO_4	0.204	0.206	4.27E-04
Ammonium Acetate	CH_3COONH_4	0.513	0.514	3.33E-03

3) In a separate container the following was prepared (Table 2.1b).

Table 2.1b. Solution Prepared in Separate Container

Compound	Compound Formula	Target Mass (g)	Measured Mass (g)	Simulant Target M
Sodium Aluminate	$NaAlO_2$	120.61	120.60	7.36E-01
Sodium Hydroxide	NaOH	137.83	137.84	1.72E+00
Selenium Dioxide	SeO_2	0.001	0.001	6.27E-06
Sodium meta-Silicate	Na_2SiO_3-$9H_2O$	2.135	2.135	3.76E-03
Sodium Acetate	CH_3COONa	2.330	2.330	1.42E-02
Sodium Formate	HCOONa	4.351	4.354	3.20E-02
Sodium Oxalate	$Na_2C_2O_4$	0.929	0.929	3.47E-03
Sodium Phosphate	Na_2HPO_4-$7H_2O$	1.608	1.608	3.00E-03

4) An additional 300 (± 1) g of water was added to the separate container, the solution was mixed thoroughly, and the contents of the separate container were added to the volumetric flask.
5) The following (Table 2.1c) was then added to the volumetric flask:

Table 2.1c. Compound Added to Volumetric Flask

Compound	Compound Formula	Target Mass (g)	Measured Mass (g)	Simulant Target M
Sodium Carbonate	Na_2CO_3	22.149	22.15	1.04E-01

6) The added material was mixed, and the following (Table 2.1d) was next added to the volumetric flask:

Table 2.1d. More Compounds Added to Volumetric Flask

Compound	Compound Formula	Target Mass (g)	Measured Mass (g)	Simulant Target M
Sodium Nitrate	$NaNO_3$	209.70	209.71	1.23E+00
Sodium Nitrite	$NaNO_2$	166.48	166.49	1.21E+00

7) The solution was again mixed thoroughly and then diluted to the mark with DI water. The final weight was measured and the density was calculated as 1.24 g/mL.

Once prepared the simulant was visually examined for precipitates; none were observed.

2.2 Preparation of Sodium Perrhenate Stock Solutions

To a 10-mL volumetric flask, 4.9204 g (18.0 mmol) of sodium perrhenate was added. The solution was diluted to 10 mL and agitated until all solids dissolved. A 100-fold diluted (by volume) sample, submitted in duplicate for analysis by inductively-coupled plasma mass spectroscopy (ICP-MS), gave Re concentrations in the stock solution of 0.0180 M and 0.0182 M, respectively, making for a stock-solution concentration of 1.8 M in Re. A second stock solution, 1.8 mM in Re, was generated by a 1000-fold dilution by volume of the initial stock solution.

2.3 Preparation of Pertechnetate Stock Solutions

(Procedure based on the information reported in Kolthoff and Elving [1964], p. 427)

A 2.3 g quantity of solid TcO_2 (obtained from in-house stores) was suspended in ca. 10 mL of concentrated ammonium hydroxide. Hydrogen peroxide (30% in water) was added until the bulk of the solids dissolved. The solution was evaporated using gentle heating to dryness; this procedure was repeated twice more. The final residue was taken up in ca. 35 mL of DI water and filtered through a 0.45-micron Nylon® syringe filter. This solution was diluted 1:1000 by volume with DI water by weight to generate a second stock solution. Assays of each solution were submitted to the Analytical Chemistry Laboratory at Battelle Pacific Northwest Division (PNWD) to measure the ^{99}Tc concentration. The results indicated a concentration of 0.0411 g Tc/mL or 0.415 M pertechnetate (1.55E+09 dpm/mL) for the first stock solution and 0.0000401 g Tc/mL or 0.405 mM pertechnetate (1.51E+06 dpm/mL) for the second stock solution.

2.4 Pretreatment of SuperLig® 639

The SuperLig® resin was pre-equilibrated starting with an initial contact with 5 M $NaNO_3$/0.1 M NaOH in approximately a 10:1 (mL liquid/g resin) ratio performed in a polypropylene bottle. For each contact, the suspension was agitated in a rotary shaker sufficient to achieve a vortex for at least 2 h. At the conclusion of each pre-equilibration contact, the shaking was stopped and the solids isolated by filtration through a 0.45-micron Nylon® filter. The solids were then sluiced back into the bottle with the next contact solution. Following the initial 5 M $NaNO_3$/0.1 M NaOH pre-equilibration contact, three further 5 M $NaNO_3$/0.1 M NaOH contacts were performed, followed by three contacts with DI water. Using broad-range pH paper as an indicator, the filtrate from the final DI water contact appeared identical to DI water itself, so further DI water washings were deemed unnecessary. The resin was then air dried for several days and placed in a plastic bottle until needed.

2.5 F-Factor Measurement for Pretreated SuperLig® 639

In duplicate, approximately 0.2-g samples of the pre-equilibrated SuperLig® 639 were placed in tared, 20-mL glass vials and reweighed. These open vials were placed in a vacuum oven and heated to 50 ± 2°C. The samples were dried until the observed weight change was less than 5% of the resin weight after about 24 h of drying. The F-factor is simply the ratio of the final 50°C/vacuum-dried resin weight to the air-dried resin weight. Table 2.2 shows the results of the F-factor experiment, which yielded a calculated average F-factor of 0.7643 ± 0.0041.

Table 2.2. Results from F-Factor Experiment

	1st sample	2nd sample
Tare wt. (g)	17.1494	17.1947
Tare wt. + resin (g)	17.3478	17.3963
Initial wt. resin (g)	0.1984	0.2016
Wt. after 24 hours drying (g)	17.3017	17.3485
Wt. after 48 hours drying (g)	17.3016	17.3482
Final wt. resin (g)	0.1522	0.1535
F-factor	0.7671	0.7614

2.6 Batch Contact Measurements – General Procedure

Step 1. Approximately 0.1 g of pre-treated SuperLig® 639 resin was placed in a tared 20-mL glass liquid scintillation counting (LSC) vial and reweighed.

Step 2. 10.1 mL of the test solution were placed in a second vial that was spiked with the appropriate Tc or Re stock solution. The spiked solution was agitated for at least 1 min, and a 0.1-mL aliquot was removed and added to a tared LSC vial containing 10 mL of Ultima Gold® LSC cocktail (Tc) or a tared 2-dram vial (Re), and reweighed.

Step 3. 10 mL of the spiked test solution was added to the vial containing the SuperLig® 639 resin, taking care not to disturb the resin so as to splash resin above the liquid. The vial was then sealed, placed in an orbital shaker, set to 25 ± 5°C unless indicated otherwise, and stirred at 225 rpm. The sample temperature was monitored using a thermocouple placed in a 20-mL LSC vial containing 10 mL DI water.

Step 4. At the conclusion of the shaking (generally 72 h unless indicated otherwise), the vial was removed, and the liquid was separated from the resin by filtration through a 0.2-micron Nylon® syringe filter.

Step 5. In the case of technetium, a 0.1-mL aliquot of the filtered solution was removed, added to a tared vial containing 10 mL of Ultima Gold® LSC cocktail, and reweighed. For the rhenium-spiked solution, the aliquot was placed in a tared 2-dram vial and reweighed.

Step 6. In the case of technetium, the sample's beta activities were measured on a Packard Instruments liquid scintillation counter using an 18-nanosecond coincidence time from the region of 200 to 800 KeV. In the case of rhenium, the concentration was determined by ICP-MS.

Step 7. K_d measurements were obtained according to the standard formula shown below (Brown et al. 1995):

$$K_d = \frac{C_0 - C_i}{C_i} * \frac{V}{(M * F)} \quad (1)$$

where:

K_d = distribution coefficient (in mL solution/g dried resin)
C_0 = initial metal concentration (Re) or activity (Tc)
C_i = final metal concentration (Re) or activity (Tc)
M = mass of resin (in grams) used in the batch contact experiment
V = volume of test solution (in mL) used in the batch contact experiment
F = F-factor.

Each experimental condition was examined in duplicate. Uncertainties presented in the tables and plots are presented as one standard deviation of the average K_d.

3.0 Results and Discussion

As noted earlier, both column and batch contact studies with SuperLig® 639 have been incorporated into a model (Hamm 2000). Because of either the importance of the data or because of conflicting results, a need for additional equilibrium data was noted. The goal of this study was to provide a large subset of the needed data. The set of binary measurements originally proposed (Test Plan CHG-TP-41500-014) were:

1) Room temperature kinetics under the experimental conditions used for subsequent batch contacts. This assures that later measurements are made under equilibrium conditions.
2) Elevated (65°C) kinetics under the experimental conditions used for subsequent batch contacts. This elevated temperature is the current choice for elution for column tests with both actual wastes and simulants.
3) Pertechnetate loading isotherm
4) Pertechnetate K_d measurements as a function of $[NO_3^-]/[TcO_4^-]$ at room temperature
5) Pertechnetate K_d measurements as a function of $[NO_3^-]/[TcO_4^-]$ at elevated temperature (65°C)
6) Pertechnetate K_d measurements as a function of changing the solution's ionic strength
7) Measurement of the impact of varying the competing anions (chloride and hydroxide) on the pertechnetate K_d
8) Measurement of pertechnetate capacity.

Several studies using SuperLig® 639 have used perrhenate as a substitute for pertechnetate. However, no study to date has done a side-by-side equilibrium batch contact comparison of perrhenate versus pertechnetate K_ds. For this reason, intermittent measurements of perrhenate K_ds were included. For each type of measurement, the general experimental conditions, a table providing the experimental results, an accompanying graph of the experimental results, and any needed commentary are provided. In general, aspects of the experimental design such as $[NO_3^-]/[MO_4^-]$ ratio, the solution ionic strength, and the solution sodium concentration have been kept constant to the extent practical to facilitate comparisons between sets of experiments.

3.1 Pertechnetate 25°C Kinetic Measurements

Samples of SuperLig® 639 were contacted with pertechnetate-spiked solutions that were 1.8 M in sodium nitrate, 0.1 M in sodium hydroxide, and 3.1 M in sodium chloride (for a total sodium concentration of 5 M) at 25 ± 0.5°C. The nitrate/pertechnetate ratio for these contacts was approximately 3400 (a 35 microliter spike of the 0.415 M pertechnetate stock solution into 10.1 mL of the 1.8 M nitrate test solution) in this experiment, and the volume of solution (mL) to grams resin was 100. Duplicate samples were agitated in a rotary shaker at 225 rpm and removed from the shaker at varying intervals out to almost 1 week in time, and the activity of each filtered solution was assayed. The results of this experiment are summarized in Table 3.1 and in Figure 3.1.

Table 3.1. Results of 25°C Pertechnetate Kinetic Tests with SuperLig® 639

Time (h)	K_d (Test 1)	K_d (Test 2)	Ave. K_d	SD Ave. K_d
2	289	298	293	6
4	399	425	412	18
21	580	632	606	37
28	597	658	627	43
47	656	643	649	9
72	629	673	651	31
94	645	632	638	9
166	602	475	538	90
SD = standard deviation				

These test results clearly show that contact times of 48 to 72 h are sufficient to achieve a steady and maximum K_d value. There is some suggestion of decreasing K_ds after extended contact times beyond 72 h, but it should be noted that such a conclusion is based primarily on the final data point that possesses a substantially larger experimental uncertainty than the other data points. This larger experimental uncertainty is because of the low K_d value measured in Test 2.

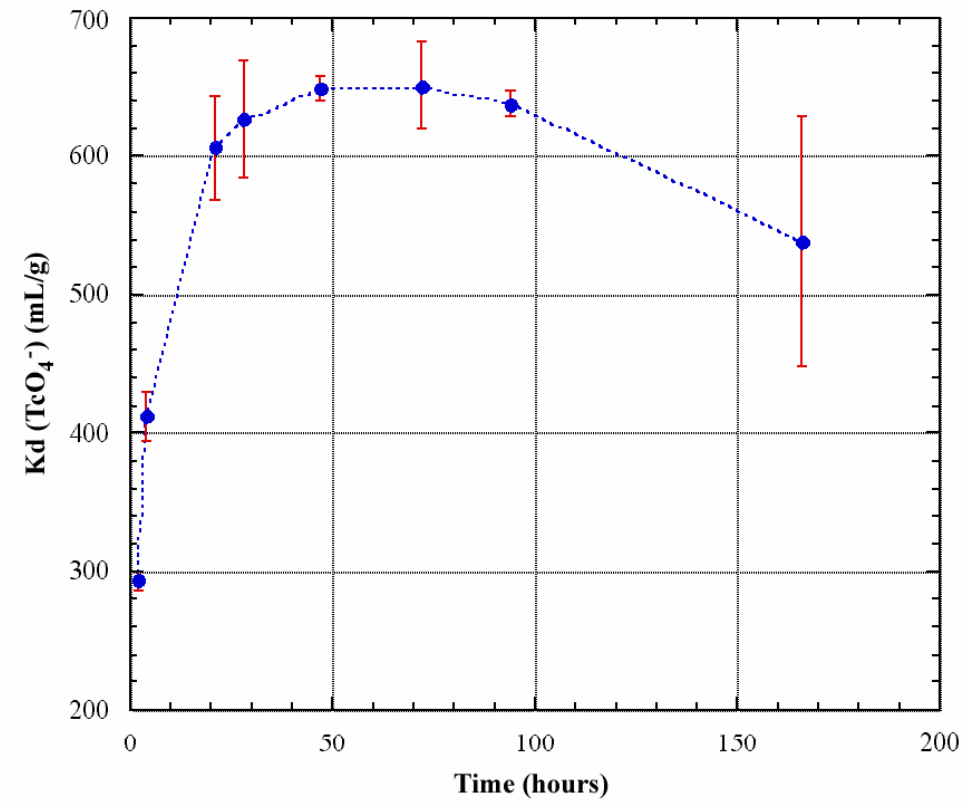


Figure 3.1. Plot of 25°C Pertechnetate Kinetic Tests with SuperLig® 639

3.2 Pertechnetate Kinetics Tests at 65°C Under Loading Conditions

The experimental procedure described in Part A was repeated except that the rotary shaker was heated such that the sample solution remained at 64.4 ± 0.5°C during shaking. This elevated temperature is expected during elution of the SuperLig® 639 resin. The results are shown in Table 3.2 and plotted in Figure 3.2.

A comparison of the 65°C data with the room-temperature data reveals several notable features. First, increasing the temperature causes a marked decrease in the pertechnetate K_d, with a decrease in these otherwise identical K_d measurements from about 650 at room temperature to approximately 140 at 65°C. Second, the system reaches a steady K_d value much more quickly than at room temperature, with essentially no change in the measured K_d from the initial 4-h measurement out to 168 h. Indeed, the average of all the average K_ds measured over the course of the experiment agree well, with an average K_d over all times of 142 ± 7.

Table 3.2. Results of 65°C Pertechnetate Kinetic Tests with SuperLig® 639 at 5 M Total Sodium

Time (h)	K_d (Test 1)	K_d (Test 2)	Ave. K_d	SD Ave. K_d
4	162	136	149	19
7	155	151	153	3
24	137	136	137	1
48	142	139	141	2
72	134	141	137	5
96	144	136	140	6
120	139	129	134	7
168	135	156	146	15
SD = standard deviation				

3.3 Pertechnetate Kinetics Tests at 65°C Under Stripping Conditions

The experimental approach described in Part B was repeated, but with two changes to the experimental conditions. First, the contact solution was composed of 0.005 M sodium nitrate and 0.001 M sodium hydroxide for a total [Na] = 0.006 M, instead of the total [Na] = 5M used in Part B. Second, the initial aqueous nitrate/pertechnetate ratio for these contacts was only about 15 (a 10-microliter spike of the 0.415 M pertechnetate stock solution into 10.1 mL of the 0.005 M nitrate test solution) in this experiment instead of the approximately 3400 in Part B. Contact temperatures were at 64.9 ± 1°C. Despite the lower initial nitrate-to-pertechnetate ratio in these test solutions, there was no detectable decrease in the aqueous pertechnetate activity in any of the solutions following contact with SuperLig® 639. This behavior, which implies a K_d of effectively zero, is desirable during elution.

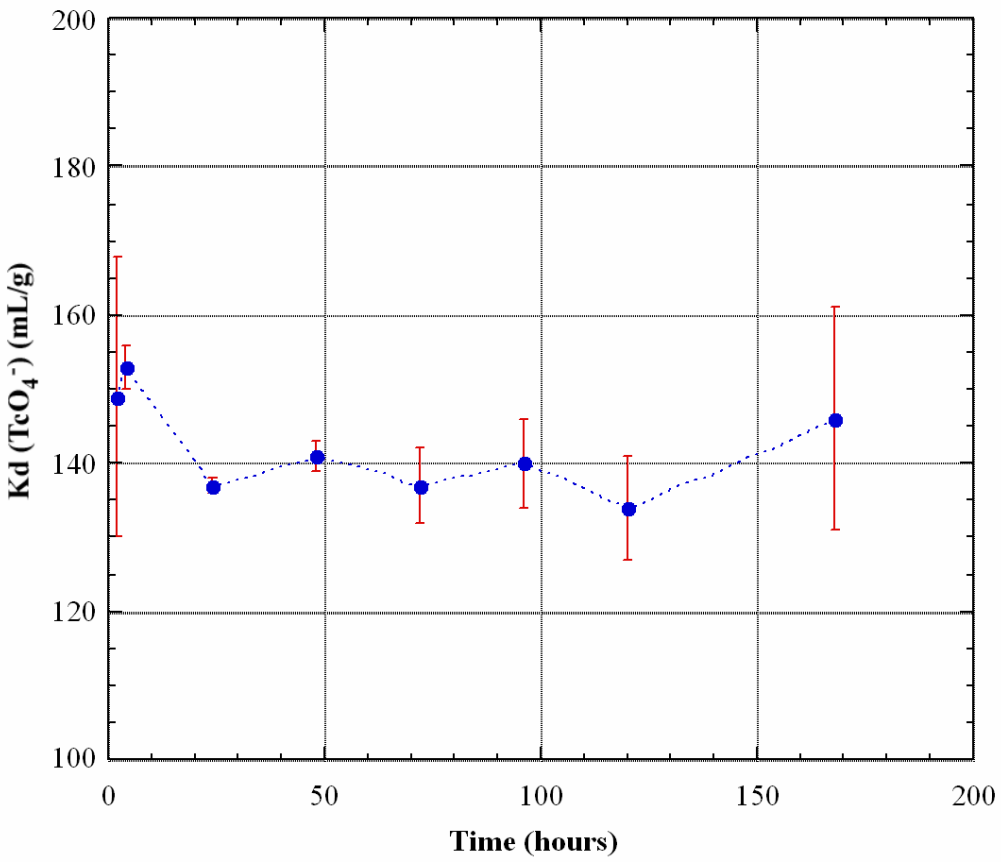


Figure 3.2. Plot of 65°C Pertechnetate Kinetic Tests with SuperLig® 639 at 5 M Total Sodium

3.4 Effect of Varying Nitrate/(Pertechnetate or Perrhenate) Ratios on SuperLig® 639 Distribution Values at 25°C

These experiments examined the impact of changing the nitrate to pertechnetate or perrhenate ratios at a constant sodium concentration of 5 M. The shaker temperature bath was held at 25.8 ± 0.3 °C over the course of the experiment. Two general types of test solutions were examined. The first test solution contained only sodium hydroxide (0.1 M), sodium chloride, and sodium nitrate. The second test solution was the AN-105 (Envelope A) simulant. The AN-105 simulant possesses a $[Na^+]$ of 5.34 M, a $[NO_3^-]$ of 1.33 M, a $[Cl^-]$ of 0.128 M, and a $[OH^-]$ of 1.72 M.

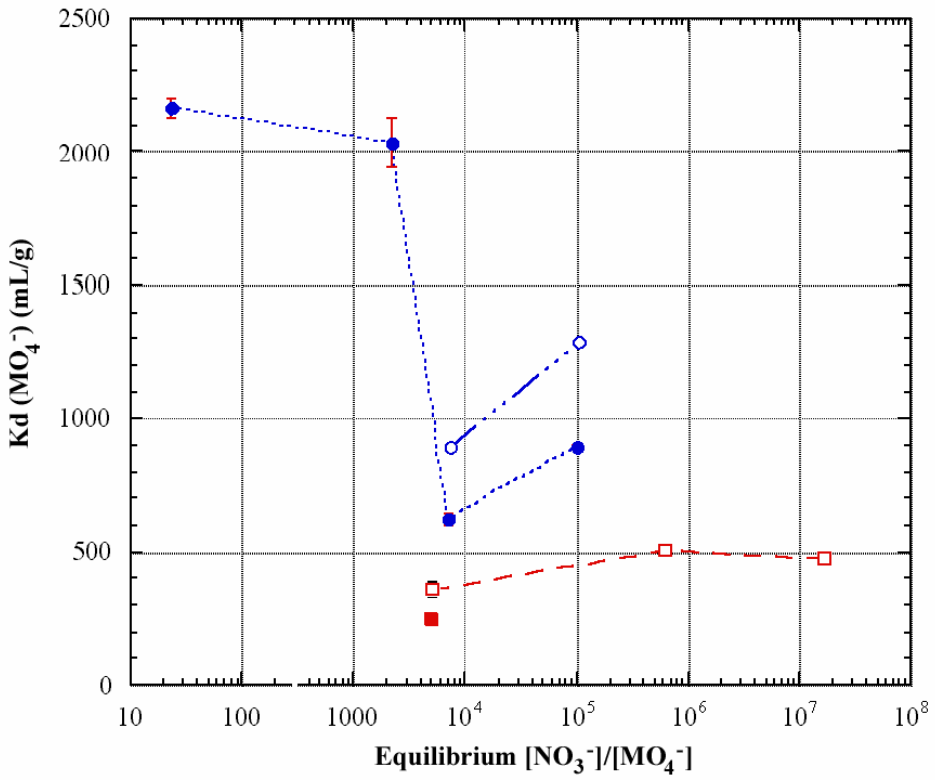
For the AN-105 simulant, the nitrate concentration is fixed, so the nitrate to perrhenate or pertechnetate ratios were varied only through changing the amount of added metal. For the simpler nitrate/hydroxide/chloride test solution, both the pertechnetate or perrhenate concentrations and the nitrate concentrations in the test solution were varied. With these test solutions, as the nitrate concentration was varied, the sodium concentration was maintained at 5 M by appropriately varying the sodium chloride concentrations. The results from this set of experiments are listed in Table 3.3 and illustrated in Figure 3.3.

Table 3.3. Impact of Varying Nitrate/(Pertechnetate or Perrhenate) Ratios on Metal K_d at 25°C

Equilibrium $[NO_3^-]/[MO_4^-]$	M in $[MO_4^-]$	Solution Type	K_d(Test 1)	K_d(Test 2)	Ave. K_d	SD of Ave. K_d
2.40E+01	Tc	A	2187	2138	2163	35
2.20E+03	Tc	A	2099	1972	2036	90
7.34E+03	Tc	A	636	608	622	19
1.03E+05	Tc	A	898	890	894	6
7.60E+03	Tc	B	881	898	889	12
1.06E+05	Tc	B	1288	1292	1290	2
4.92E+03	Re	A	254	251	253	3
5.18E+03	Re	B	380	339	360	29
6.30E+05	Re	B	490	522	506	23
1.71E+07	Re	B	476	469	473	5
Solution A = simple nitrate/chloride/hydroxide at 5 M total Na. Solution B = AN-105 simulant						

Several points are apparent from the data listed in Table 3.3 and plotted in Figure 3.3. First, the AN-105 simulant gives consistently higher K_ds for both Re and Tc than is found with the simple 0.1 M sodium hydroxide/sodium nitrate/sodium chloride test solution. The reason for these higher K_ds is unclear and may be difficult to say with certainty, especially given the unknown structure of the complexing agent in SuperLig® 639. All anions should be at least in competition with pertechnetate and perrhenate; it is difficult to imagine how an anion could be acting as a synergist. With respect to the relative type and concentration of cation in the AN-105 simulant versus the simple sodium hydroxide/sodium nitrate, sodium chloride simulant, if an ion-pair extraction mechanism were involved, the slightly higher sodium concentration (5.3 M versus 5 M in the simple simulant) and/or the presence of potassium (0.1 M versus 0.0 M in the simple simulant) in the AN-105 simulant may be in part responsible for the observed enhanced K_ds.

Two other features of note include the consistently lower K_ds observed for perrhenate when compared to pertechnetate and the trend in K_ds as the nitrate/perrhenate or pertechnetate ratio increases. Although the number of comparable data points are few, the perrhenate K_d appears to be consistently about half of the pertechnetate K_d under similar conditions. This is comparable to the approximately 70% higher values for pertechnetate versus perrhenate previously reported (Hamm 2000). The trend in Kds for both perrhenate and pertechnetate as the nitrate/metal ratio is varied appear similar. At low nitrate/metal ratios, the K_ds are at their highest. Between a ratio of 1000 and 10000, a marked drop in K_d is observed. It is unfortunate that more data were not collected in this region to better map these sharp changes in K_d. As the nitrate/metal ratio increases from 10^4 to 10^5, the K_d again increases. Data are only available for perrhenate above a ratio of 10^5, but it appears that little change in K_d occurs as the nitrate/metal ratios increase above 10^5. A simple model would have predicted decreasing K_ds as the ratio of a competing anion, nitrate, to the metal increases. The chemical behavior underlying the more complex behavior observed here is unknown.



Filled Circles = 0.1 M Sodium Hydroxide/Sodium Nitrate/Sodium Chloride Test Solution; M = Tc
Filled Square = 0.1 M Sodium Hydroxide/Sodium Nitrate/Sodium Chloride Test Solution; M = Re
Open Circles = AN-105 Simulant; M = Tc
Open Squares = AN-105 Simulant; M = Re

Figure 3.3. Plot of Varying Nitrate/(Pertechnetate or Perrhenate) Ratios on Metal K_d at 25°C

3.5 Effect of Varying Nitrate/(Pertechnetate or Perrhenate) Ratios on SuperLig® 639 Distribution Values at 65°C

A set of batch contacts analogous to those reported in Section D was performed, only here the shaker temperature was maintained at the elution temperature of 65.2°C. The results are listed in Table 3.4 and illustrated in Figure 3.4.

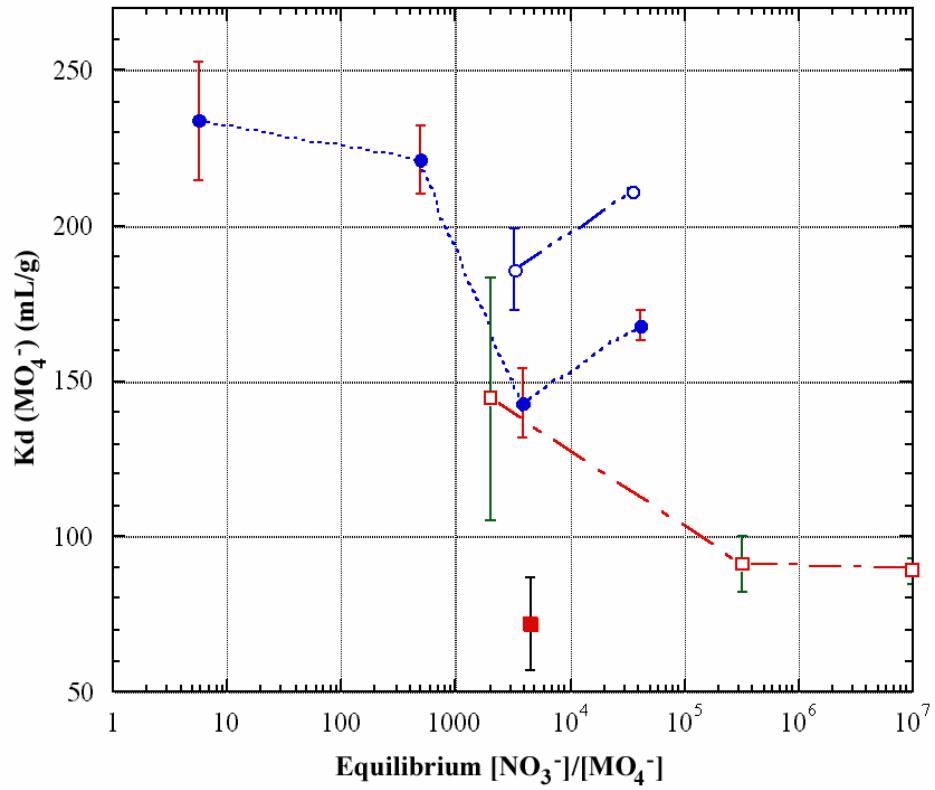
The trends observed in these 65°C batch contacts parallel the trends observed in the analogous room-temperature set of batch contacts.

- A sharp decrease in K_d is seen as the nitrate/pertechnetate ratio increases from approximately 10^3 to 10^4.
- The AN-105 simulant yields markedly higher K_ds than does the simple 5 M sodium, 0.1 M hydroxide nitrate/chloride test solution.
- In general, the perrhenate Kd appears to be about one-half the pertechnetate K_d.
- At approximately 10^4 to 10^5 nitrate/pertechnetate ratios, the pertechnetate K_ds increase.
- At very high (>10^5) nitrate/perrhenate ratios, little change in K_d is observed.

Table 3.4. Impact of Varying Nitrate/(Pertechnetate or Perrhenate) Ratios on Metal K_d at 65°C

Equilibrium $[NO_3^-]/[MO_4^-]$	M in $[MO_4^-]$	Solution Type	K_d(Test 1)	K_d(Test 2)	Ave. K_d	SD of Ave. K_d
3.90E+00	Tc	A	247	220	234	19
3.42E+02	Tc	A	228	213	221	11
2.64E+03	Tc	A	150	135	143	11
2.84E+04	Tc	A	171	165	168	5
2.29E+03	Tc	B	176	195	186	13
2.48E+04	Tc	B	212	210	211	1
4.50E+03	Re	A	83	62	72	15
2.09E+03	Re	B	172	117	144	39
3.34E+05	Re	B	85	98	91	9
9.97E+06	Re	B	83	62	72	15
Solution A = simple nitrate/chloride/hydroxide at 5 M total Na. Solution B = AN-105 simulant.						

Similar to the results seen in the batch-contact kinetics tests, increasing the temperature from 25°C to 65°C substantially decreases in K_d. The only unexpected feature is observed during the measurement at a ca. 10^3 nitrate/perrhenate ratio when contacted with the AN-105 simulant, which appears to have an unexpectedly high K_d. However, this point also exhibits poor reproducibility, which suggests that some problem is associated with the results of this particular batch contact. If the K_d(1) point is ignored, then the results fall more in line with the trends seen in the 25°C data set.



Filled Circles = Simple 0.1 M Sodium Hydroxide/Sodium Nitrate/Sodium Chloride Test Solution; M = Tc

Filled Square = Simple 0.1 M Sodium Hydroxide/Sodium Nitrate/Sodium Chloride Test Solution; M = Re

Open Circles = AN-105 Simulant; M = Tc

Open Squares = AN-105 Simulant; M = Re

Figure 3.4. Plot of Varying Nitrate/(Pertechnetate or Perrhenate) Ratios on Metal K_d at 65°C

3.6 Effect of Varying Chloride Versus Hydroxide as the Competing Anion on SuperLig® 639 Pertechnetate Distribution Values at 25°C

In this set of batch-contact experiments, with the shaker temperature maintained at 25.8 ± 0.3°C, the relative proportions of sodium hydroxide and sodium chloride were varied while keeping a constant initial nitrate to pertechnetate ratio (approximately 40) and a constant total sodium concentration of 5 M. The results of this set of batch contacts are listed in Table 3.5 and are illustrated in Figure 3.5. They indicate that hydroxide is a more effective competitor than chloride for pertechnetate.

Table 3.5. Effect of Varying Hydroxide Versus Chloride at 5 M Total Sodium, 0.018 M Nitrate, 25°C

$[Cl^-]$, M	$[OH^-]$, M	Equilibrium $[NO_3^-]/[TcO_4^-]$	K_d(Test 1)	K_d(Test 2)	Ave. K_d	SD of Ave. K_d
4.9	0.1	1.1E+03	2789	2864	2826	53
4	1	1.1E+03	2903	2970	2937	48
2.5	2.5	9.4E+02	2585	2672	2629	61
1	4	9.0E+02	2531	2250	2391	199
0.1	4.9	6.4E+02	1753	1508	1630	174

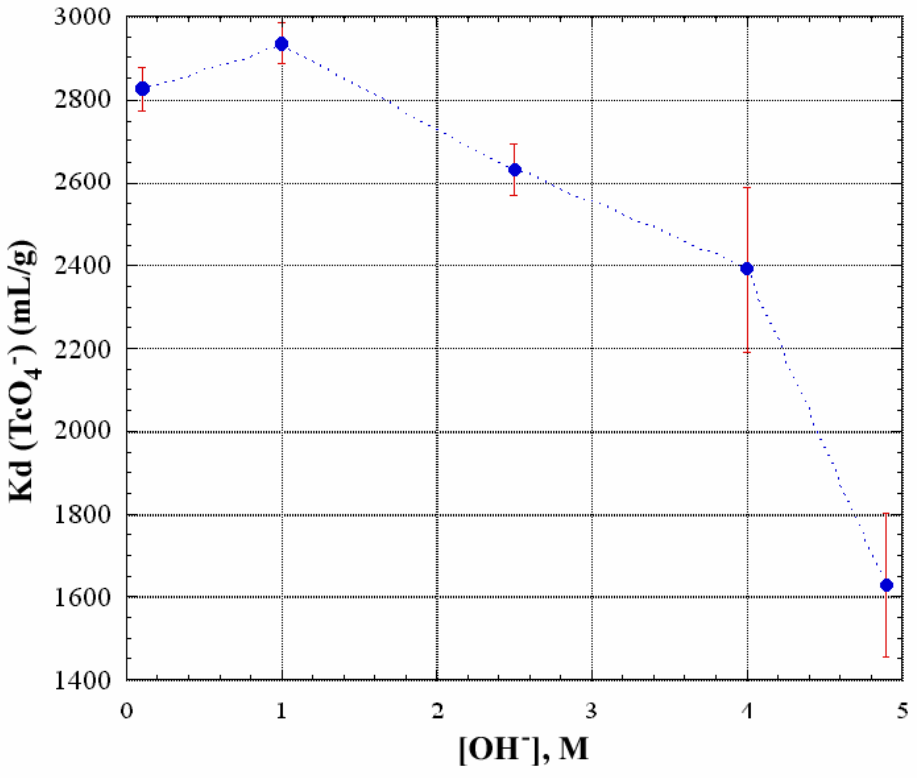


Figure 3.5. Plot of Effect of Varying Hydroxide Versus Chloride at 5 M Total Sodium, 0.018 M Nitrate, 25°C

3.7 Impact of the Sodium Concentration on SuperLig® 639 Pertechnetate Distribution Values at 25°C

In this set of batch-contact experiments, the hydroxide concentration was fixed at 0.01 M, the total sodium was adjusted from approximately 0.02 to 5 M sodium with sodium chloride, and the initial nitrate/pertechnetate ratio was kept constant at about 12. During these batch contacts, the shaker temperature was kept at 25.5 ± 0.2°C. The results of this set of batch contacts are listed in Table 3.6 and are illustrated in Figure 3.6.

Table 3.6. Impact of Varying Sodium Concentration on Pertechnetate K_d Values at 0.018 M Nitrate/0.01 M Hydroxide, 25°C

Total $[Na^+]$, M	Equilibrium $[NO_3^-]/[TcO_4^-]$	K_d(Test 1)	K_d(Test 2)	Ave. K_d	SD of Ave. K_d
5.028	2.23E+02	2069	1924	1997	103
1.028	4.94E+01	360	342	351	13
0.528	3.15E+01	217	188	203	20
0.128	1.92E+01	69	73	71	3
0.028	1.38E+01	15	10	12	3

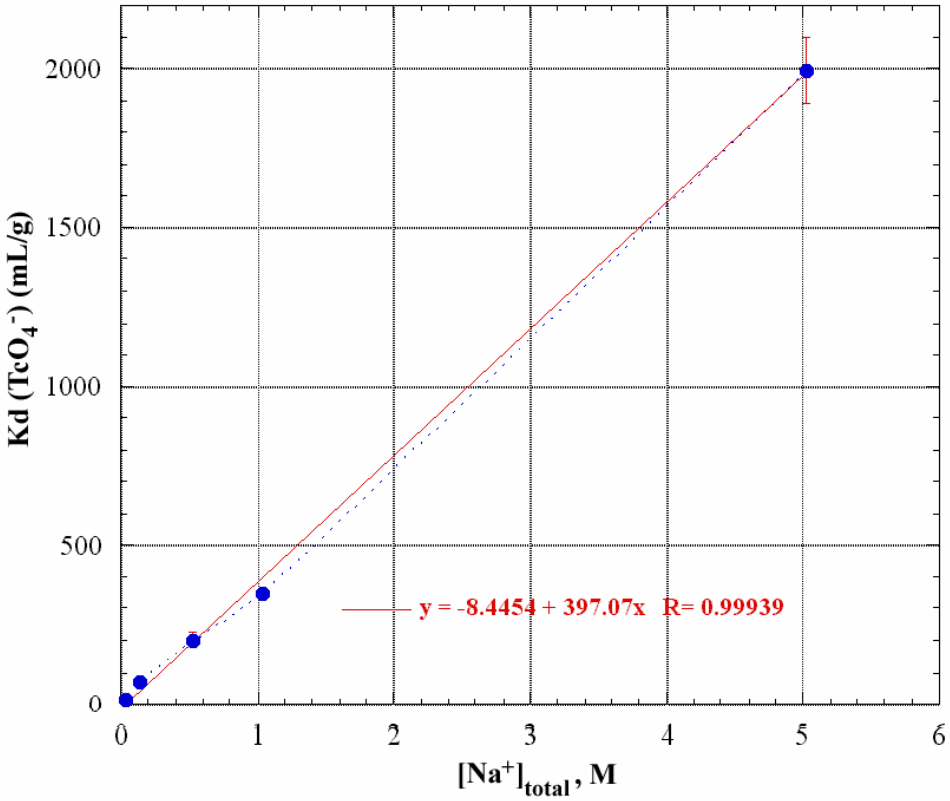


Figure 3.6. Plot of the Impact of Varying Sodium Concentration on Pertechnetate K_d Values at 0.018 M Nitrate/0.01 M Hydroxide, 25°C

These results show a striking linear correlation between the solution's sodium concentration and the pertechnetate K_d, and they are consistent with the previous statements that pertechnetate and the cation are extracted as an ion pair (Kurath et al. 1999, King et al. 2000). However, as shown in Table 3.7 and Figure 3.7, the agreement with solution molality, which for the 1:1 salts used in this test is equivalent to the test solution's ionic strength, is also excellent. In conclusion, it is impossible to eliminate either interpretation based on the experiments performed here.

Table 3.7. Impact of Varying Ionic Strength on Pertechnetate Distribution Values at 0.018 M Nitrate/0.01 M Hydroxide, 25°C

Total Ionic Strength, m	Equilibrium $[NO_3^-]/[TcO_4^-]$	K_d(Test 1)	K_d(Test 2)	Ave. K_d	SD of Ave. K_d
5.62E+00	2.23E+02	2069	1924	1997	103
1.04E+00	4.94E+01	360	342	351	13
5.34E-01	3.15E+01	217	188	203	20
1.29E-01	1.92E+01	69	73	71	3
2.80E-02	1.38E+01	15	10	12	3

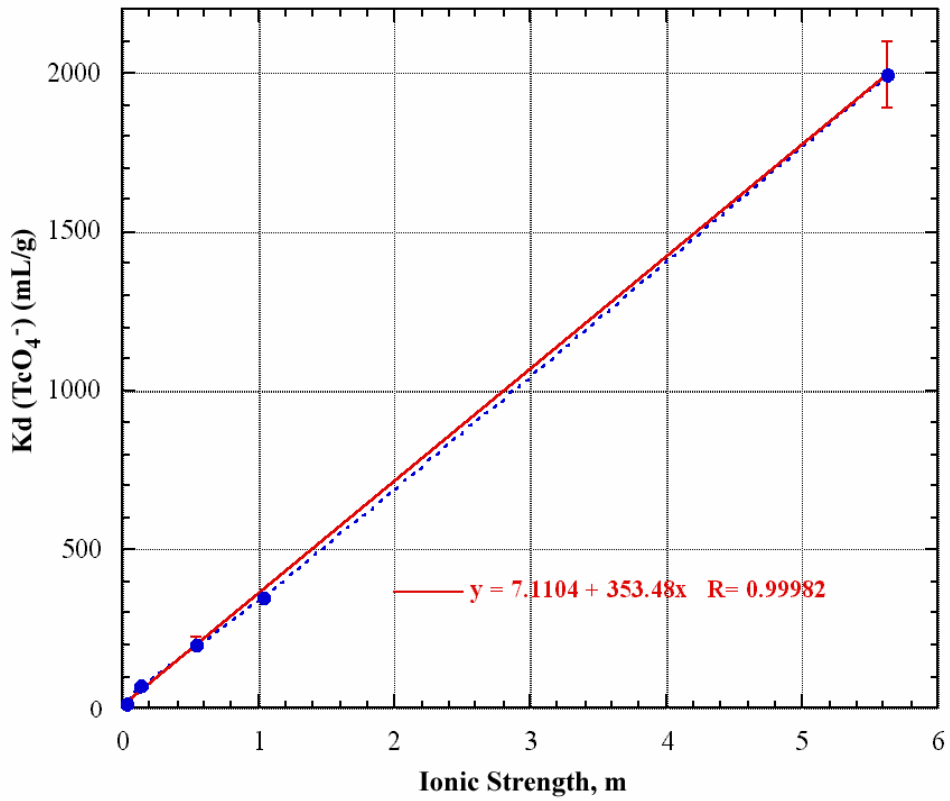


Figure 3.7. Plot of the Impact of Varying Solution Ionic Strength on Pertechnetate Distribution Values at 0.018 M Nitrate/0.01 M Hydroxide, 25°C

4.0 Conclusions

This report summarizes a series of batch contacts with SuperLig® 639 that was used to measure how various system perturbations alter pertechnetate and perrhenate distribution values. Several goals were described in the introduction. For the sake of convenience, they are repeated here along with the main conclusions from each set of tests.

1) Room-temperature kinetic measurements under the experimental conditions used for subsequent batch contacts. The primary purpose behind this measurement was to verify that subsequent room-temperature batch contacts were performed under equilibrium conditions. It was determined from the results reported in Section A that under the experimental conditions employed for these batch contacts, in particular the speed (225 rpm) used with this rotary shaker, that 48 h would be sufficient to achieve equilibrium. Contact times of 72 h were used in subsequent measurements to assure that these measurements were made under equilibrium conditions.

2) Elevated (65°C) kinetic measurements under the experimental conditions used for subsequent batch contacts. At high (5 M) sodium concentrations, the targeted concentration for treated Hanford tank solutions by the SuperLig® 639 resin, the results reported in Section B indicate that the system reaches equilibrium at 65°C much more rapidly than at room temperature, with the system achieving a distribution value within 4 h that remained unchanged even if contacted for several additional days. For consistency's sake with the room-temperature measurements, 72 h was used for subsequent measurements made at 65°C. In Part C, at 65°C and low, approximately 2 mM, sodium concentrations, the pertechnetate distribution values were so low that no decrease in pertechnetate concentration was observed (a K_d of effectively zero).

3) Pertechnetate K_d measurements as a function of $[NO_3^-]/[TcO_4^-]$ at room temperature. In Part D, the pertechnetate K_d is reported as a function of the equilibrium nitrate/pertechnetate ratio over a range of 10^6. In addition, pertechnetate measurements on a Envelope A supernatant, AN-105, simulant, as well as selected perrhenate K_d measurements, were reported. The most striking features are: 1) the enhanced K_ds for perrhenate and pertechnetate observed in the AN-105 simulant versus the simple, 5 M $[Na^+]$ simulant and 2) the approximately 50% lower perrhenate K_d as compared to the pertechnetate K_d. The cause of the enhanced K_ds is unclear, but extraction of pertechnetate as an ion-pair coupled with the presence of a more tightly binding cation (such as potassium) in the AN-105 simulant or a slightly higher sodium concentration in the AN-105 simulant are possible explanations.

4) Pertechnetate K_d measurements as a function of $[NO_3^-]/[TcO_4^-]$ at elevated temperature (65°C). In Part E, the same scope of measurements performed in Part D are repeated, but with the test samples at 65°C. The same trends noted above are also found at 65°C, but the distribution values are substantially decreased.

5) Pertechnetate K_d measurements as a function of changing the solution's ionic strength. In Part G, the impact of changing the salt concentration on the pertechnetate distribution value was examined. A strong linear correlation of the pertechnetate distribution value to both the total sodium concentration and to the solution's ionic strength was found. In this test, those two factors are so closely related that it cannot be said with certainty which solution property is impacting the pertechnetate distribution value. If the ionic strength effect is indeed the controlling factor, the magnitude of the response is remarkable. Alternatively, if the resin operates by extracting a sodium pertechnetate ion pair, the test results may be simply reflecting the resin affinity for sodium ion.

6) Measurement of the impact of varying the competing anions chloride and hydroxide on the pertechnetate K_d. In Part F, the pertechnetate distribution value was measured as the chloride and hydroxide ratios were changed while keeping the solution's sodium concentration constant. The results indicate that hydroxide competes more effectively than chloride with pertechnetate for binding to SuperLig® 639.

5.0 References

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Appendix A

Test Plan

CONTROLLED DOCUMENT
COPY NO. 2

PNNL Test Plan	Document No.: CHG-TP-41500-014 Rev. No.: 0
Title: Equilibrium Batch Contact Testing for CH2MHill Inc.	
Work Location: SAL, lab 410, 507, 511, 516	**Page** 1 of 14
Author: BM Rapko	**Effective Date: 1/22/01** **Supersedes Date: (New)**
Use Category Identification: Reference	
Identified Hazards: _X_ Radiological _X_ Hazardous Materials ___ Physical Hazards ___ Hazardous Environment ___ Other:	**Required Reviewers:** _X_ Author _X_ Technical Reviewer _X_ RPL Manager _X_ Project Manager _X_ RPG Quality Engineer _X_ CHG

Are One-Time Modifications Allowed to this Procedure? _X_ Yes ___ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? _____ Yes or _X_ No

FOR REVISIONS:
Is retraining to this procedure required? _____ Yes _X_ No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
_____ Yes _____ No _X_ N/A

PNNL Test Plan	Document No.: CHG-TP-41500-014 Rev. No.: 0

Title: Equilibrium Batch Contact Testing for CH2MHill Inc.

Approval	Signature	Date
Author	Brian Rapko	1-19-01 (02) ~~1-19-00~~
Technical Reviewer	Daniel McCloskey	1-19-01
RPL Manager	Dave E. Kurath	1/29/01
Project Manager	D E Kurath	1/29/01
RPG Quality Engineer	TJ Almeida	1/24/01
CHG	Michael J Johnson	1/30/01

Applicability

This test plan governs the measurement of distribution values (Kd) through batch contacts for technetium (as pertechnetate), cesium, and uranium (as uranyl ion) with various ion exchange materials in contact with a variety of solutions. Radioactive cesium, technetium and uranium tracers may be used in the solutions for measuring concentration changes and so allow Kd calculation. The ion exchange materials to be tested include Superlig® 644 (cesium and uranyl ion removal), and Superlig 639® (Tc removal). The tests will be conducted in fume hoods in lab 410, 507, 510, 511 and 516 of the 325 building as needed. The test plan applies to staff working on this task in the Radiological Processing Laboratory (RPL) and includes laboratory technicians, scientists and engineers.

DRD Reference

The work described in this test plan will be performed in partial fulfillment of task 8.2.12, Mathematical Modeling, of the River Protection Project Waste Treatment Plant (RPP-WTP) Development Requirements Document. The RPP-WTP Research and Technology schedule for fiscal year 2001 identifies this activity as R20500, Eluant Equilibrium Behavior PNNL. The corresponding activity on the PNNL schedule for fiscal year 2001 is BN.02.08.03.

Justification/Test Objectives/Success Criteria

The proposed facility for immobilization of Hanford underground storage tank waste will pretreat the waste to split it into a Low Activity Waste (LAW) and a high level waste (HLW) for separate vitrification. Unit processes are planned to remove radiocesium and radioactive technetium (in the pertechnetate form) by ion exchange from these strongly alkaline solutions. Currently, Savannah River Technology Center (SRTC) is attempting to develop a model for these ion exchange processes. Batch distribution coefficients with each ion exchange material are needed both to supply information about the behavior of these ion exchange resins under various processing conditions and to validate the developed model. The RPP-WTP contractor will then incorporate this SRTC process model into the process flow sheet.

The equilibrium batch data for technetium (Superlig 639®), cesium and uranyl (Superlig 644®) generated by this study will be incorporated by SRTC personnel into their ion exchange process models. Therefore, the RPP-WTP contractor success criteria for these tests are to accurately measure batch distribution coefficients (±10%) and understand the influences of contact duration and equilibrium concentrations on the batch distribution coefficients.

Background

Much recent work has been performed investigating the use of two ion exchange resins developed by IBC Advanced Technologies, Inc. for use in removing selected radionuclides from Hanford tank waste supernatants. Superlig 639® is being investigated for removal of technetium (as pertechnetate) and Superlig 644® (cesium) from alkaline, high sodium solutions including actual Hanford tank supernatants (Hamm 2000a, Hamm 2000b and Blanchard 2000). The existing data have been evaluated and included in an ion exchange process model currently under development at SRTC. However, a need for additional, equilibrium -batch -contact data has been identified in order to continue to develop and extend the modeling for use under likely process conditions. It has been shown that such batch contact distribution data collected from binary systems can be incorporated in such a model for use in more complex systems (Mehablia et. al. 1994). The work covered in this test plan will measure such binary equilibrium batch

contact data together with the appropriate controls (such as the batch distribution kinetics) required to interpret the batch equilibrium data for both Superlig® 644 and Superlig® 639.

Previous work in this area has generated information that impacts the experimental design described in this test plan. For instance, the capacity of SuperLig® 639 resin has been reported (Hamm 2000b) as 0.88 meq/g resin) and the capacity of SuperLig® 644 resin is uncertain but best estimates (Hamm, private communication) indicate a value around 0.6 meq/g resin. In general, batch distribution contacts should be designed so that the resin will not be fully loaded even under conditions that will generate very high Kds, i.e., effectively all of the binding species reports to the resin under the experimental conditions. A target of no more than 20% loading has been used in similar, prior, PNNL equilibrium batch contact tests. With a solution volume/resin mass ratio of 100 this would indicate that a solution should not be more concentrated than ca. 1.2 mM in the binding species for batch contacts with SuperLig® 644 and not more than ca. 1.8 mM in the binding species for contacts with SuperLig® 639.

Emergency Response

Spill containment will be provided where practical. In the event of a leak from the recirculating heating bath, the heater should be turned off or unplugged immediately.

Equipment Description

The test equipment required for the batch distribution tests are as follows:

- Aqueous test solutions - prepared as required in accordance with test instructions.
- Ion Exchange Materials - purchased as required or else obtained from existing stocks as needed.
- Appropriate tracers (^{99}Tc, ^{99m}Tc, ^{95m}Tc, ^{137}Cs) - the radiotracers may be obtained either through commercial sources (i.e., Brookhaven National Laboratory, Brookhaven NY; New England Nuclear, Boston, MA; or Amersham, Arlington, IL; or from stock solutions already in the laboratory). Technical data sheets accompanying the commercial shipments should list the assay of the isotope (i.e., chemical form, purity, assay date, specific activity, concentration, and volume). Because comparative measurements of tracers will be made, nominal activity concentrations of tracers may be used.
- Counting Equipment - gamma counting is performed by the use of a multichannel analyzer and suitable detector, such as a high purity germanium detector or NaI well detector. The equipment is user calibrated. Alpha and beta scintillation activities will be obtained by liquid scintillation counting using commercial instrumentation.
- Shaker/Temperature Control Systems - the shaker table will include a test chamber that holds the sample container. A calibrated thermocouple or other temperature measuring device is inserted in the shaker package along with the samples.
- Calibrated thermometer or thermocouple.
- Filters, 0.2µ pore size disposable.
- Syringe, 5 mL, plastic, Luer-Loc or equivalent.
- Vials, 20 mL glass scintillation type.
- Bottles, 20 to 4000 mL polyethylene or polypropylene.

- Pipets, adjustable, 10 to 10000 µL maximum capacity.

Safety Considerations

All activities conducted under this test plan will be performed in accordance with approved safety procedures, including but not limited to proper radiological control procedures. Personnel involved in the testing in the 325 Building will be familiar with specific safety procedures for that facility. All personnel performing work to this test plan shall be qualified to work in fume hood and to work with acids and bases.

Special safety considerations for these procedures include:

- Ion exchange materials are not to come in contact with nitric acid solutions of greater than 3 molar concentration.

Outline of test scope and work instructions

Note: The work covered in this test plan is governed by the RPL Work Practices document, RPL-OP-001, rev. 0, Routine Research Operations as well as those additional work practices described in the Radiochemical Processing Laboratory's Laboratory Handbook.

1. The ion exchange materials to be used in this test are SuperLig® 644 (Cs) and SuperLig® 639 (Tc) manufactured by IBC Advanced Technologies, Inc. The production lot number for each resin is to be recorded.

2. Preconditioning of the ion exchange materials is required before batch contacts will be performed for both SuperLig® 644 and SuperLig® 639.

(a) SuperLig® 644: a weighed portion of the resin will be contacted once with 5 M $NaNO_3$/0.1 M NaOH followed by 4 times with 0.5 M nitric acid at a V/m (mL to grams) ratio of 100. The solution and resin will remain in contact with agitation for at least 1 hour during each pre-equilibration. After each contact, the solid and liquid will be separated, either by centrifugation or settling, with the liquid removed and discarded. The resin will then be washed with DI water at a V/m (mL to grams) of 100 until the pH of the liquid is 5 or above, air dried, reweighed, and stored.

(b) SuperLig® 639: a weighed portion of this resin will be contacted 4 times with 5 M $NaNO_3$/0.1 M NaOH at a V/m (mL to grams) ratio of 100. The solution and resin will remain in contact for at least 1 hour during each pre-equilibration. After each contact, the solid and liquid will be separated either by centrifugation or settling, with the liquid removed and discarded. The resin will then be washed with DI water at a V/m (mL to grams) of 100 until the pH of the liquid is 8 or below, air dried, reweighed, and stored.

3. A portion of both the as-received and the conditioned materials will be dried to determine an F factor (relative water content of the resin).

(a) SuperLig® 644: the resin will be dried under reduced pressure at a temperature no greater than 50°C until the resin reaches a constant weight. A constant weight is assumed to be reached if there is no greater than a 5% change in the system weight after 24 hours of drying or at the discretion of the cognizant scientist. The temperature and duration of the drying will be recorded. A second portion will be similarly dried at 85°C under vacuum to allow for a comparison to earlier work.

(b) Superlig® 639: the resin will be dried under reduced pressure at no greater than 50°C until the resin reaches a constant weight. A constant weight is to be assumed if there is no greater than a 5% change in the system weight after 24 hours of drying or at the discretion of the cognizant scientist. The temperature and duration of the drying will be recorded. A second portion will be similarly dried at 85°C under vacuum to allow for a comparison to earlier work.

4. All batch contacts will be performed in duplicate.

5. Unless otherwise specified, the minimum equilibrium batch contact duration will be 72 hours.

6. The concentrations of the targeted elements will be measured for each batch contact before and after each batch contact. Either the solution activity for specific radionuclides used as tracers or the total solution activity are considered as an acceptable substitute for an actual elemental concentration measurement.

   (a) SuperLig® 644: cesium concentrations will be determined through gamma counting of a Cs-137 tracer using either a NaI or Ge detector.

   (b) SuperLig® 644: uranium concentrations will be determined by counting of the total alpha activity of a U-233 tracer using liquid scintillation counting or by a laser fluorimetric determination of the solution's total uranium concentration.

   (c) SuperLig® 639: technetium concentrations will be determined either by total beta activity of Tc-99 using liquid scintillation counting or by gamma counting of a Tc-95m tracer using either a NaI or Ge detector.

   (d) SuperLig® 639: rhenium concentrations in solutions will be determined by ICP-MS.

7. The batch contacts will be conducted at a specific recorded temperature or at ambient temperature as specified in the test instructions. The temperature of the temperature bath (or the surrounding air temperature) will be recorded during each batch contact test.

8. Two Tc-99 stock solutions will be prepared. The first, ca. 1.8 M Tc in water, will be prepared by taking 2.3 g of $^{99}TcO_2$ and dissolving it in ammonium hydroxide/hydrogen peroxide, evaporating to dryness at low temperature/atmospheric pressure and redissolving the solid to a total volume of 10 mL with DI water. The second Tc-99 stock solution will be ca. 1.8 mM in water and prepared by a 1:1000 volumetric dilution from the first Tc-99 stock solution. The specific activity will be measured by beta scintillation counting and the radiochemical purity of the material will be verified by ICP-MS before use.

9. A rhenium stock solution ca. 1.8 M Re in water, will be prepared by dissolving 4.92 grams of sodium perrhenate to a total volume of 10 mL with DI water.

10. To verify that the kinetics of the Superlig® 639 with pertechnetate are such that a 72 hr contact time will yield an equilibrium distribution value, a series of contacts will be performed at room temperature (25 ± 5°C) under the conditions described in Table 1. The temperature will be recorded during these batch contact tests. The sample's agitation will be stopped after 2, 4, 8, 24, 48, 72, 96 and 168 hr (1 week), respectively and the final solution activity measured.

Table 1. Room temperature kinetics under loading conditions. Contact conditions: ambient temperature, $[Na]_T = 5$ M

[Tc] stock solution.M	Stock sol. aliquot, µL	Test [Tc], M	$NaNO_3$, M	NaOH, M	NaCl, M	$[NO_3]/[TcO_4]$
1.8	10	1.8E-03	1.8 M	0.1	3.1	1E03

11. Chloride and hydroxide are believed to be spectator ions in pertechnetate absorption by SuperLig® 639. To test this, batch contacts at a constant $[NO_3]/[TcO_4]$ ratio and total [Na] will be performed with varying ratios of hydroxide and chloride as described in Table 2. The temperature will be recorded during these batch contact tests. If chloride is determined to have a significant impact on the pertechnetate distribution constant, then sodium nitrite will be used instead of sodium chloride for the technetium measurements indicated below.

Table 2. Tc loading isotherm. Contact conditions: room temperature (25 ± 5°C), 72 hours contact time, $[NO_3]/[TcO_4] = 1E03$, $[Na]_T = 5$ M

[Tc] stock solution.M	Stock sol. aliquot, µL	Test [Tc], M	$NaNO_3$, M	NaOH, M	NaCl, M	$[NO_3]/[TcO_4]$
0.0018	10	1.8E-06	0.018	0.1	4.9	1E04
0.0018	10	1.8E-06	0.018	1	4	1E04
0.0018	10	1.8E-06	0.018	2.5	2.5	1E04
0.0018	10	1.8E-06	0.018	4	1	1E04
0.0018	10	1.8E-06	0.018	4.9	0.1	1E04

12. To verify that the kinetics of the Superlig® 639 with pertechnetate are such that a 72 hr contact time will yield an equilibrium distribution value at the elevated temperatures proposed for elution, a series of contacts will be performed at 65°C under the conditions described in Table 3. The measured temperature will be recorded during these batch contact tests. The sample's agitation will be stopped after 2, 4, 8, 24, 48, 72, 96 and 168 hr (1 week), respectively and the final solution activity measured.

Table 3. Elevated temperature kinetics under stripping conditions. Contact conditions: 65 ± 5°C $[Na]_T = 0.006$ M, $[NO_3]/[TcO_4] = 3E00$

[Tc] stock solution.M	Stock sol. aliquot, µL	Test [Tc], M	$NaNO_3$, M	NaOH, M	NaCl, M	$[NO_3]/[TcO_4]$
1.8	10	1.8E-03	0.005 M	0.001	0	3E0

13. The impact of the NO_3^-/TcO_4^- ratio under loading conditions will be investigated. Additionally, the adsorption of Re and Tc by SL-639 resin will be compared to verify the suitability of Re as a surrogate for Tc. The batch contact will be performed at a V/m (mL/grams) of 100 unless otherwise specified. An initial aliquot will be removed for activity assay and a weighed amount of resin added. The sample will be agitated at ambient temperature (25 ± 5°C) for at least 72 hours, the resin removed by filtration through a 0.2 micron syringe filter, and another aliquot removed to

determine the final solution activity. The range of experimental conditions to be tested is outlined in Table 4 and the temperature will be recorded during these batch contact tests

Table 4: Contact conditions for the NO_3^-/TcO_4^- ratio variations under loading conditions; $[Na]_T$ = 5 M, ambient temperature (25 ± 5°C), 72 hour contact time

[Tc] stock solution.M	Stock sol. aliquot, µL	Initial [Tc], M	$NaNO_3$, M	NaOH, M	NaCl, M	Initial $[NO_3]/[TcO_4]$
1.8 M	10	0.0018	0.0018	0.1	4.9	1E00
1.8 M	10	0.0018	0.180	0.1	4.7	1E02
1.8 M	10	0.0018	1.80	0.1	3.1	1E03
1.8 M	1	1.8E-04	1.80	0.1	3.1	1E04
0.0018	100	1.8E-05	1.80	0.1	3.1	1E05
0.0018	10	1.8E-06	1.80	0.1	3.1	1E06
1.8 M	7	0.00125	Envelope A (AN-105) simulant(a)			1E03
1.8 M (Re)	7	0.00125 (Re)	Envelope A (AN-105) simulant(a)			1E03
0.0018 M	70	1.25E-05	Envelope A (AN-105) simulant(a)			1E05
0.0018 M (Re)	70	1.25E-05 (Re)	Envelope A (AN-105) simulant(a)			1E05
0.0018 M	7	1.25E-06	Envelope A (AN-105) simulant(a)			1E06
0.0018 M (Re)	7	1.25E-06 (Re)	Envelope A (AN-105) simulant(a)			1E06
1.8 M (Re)	10	0.0018 (Re)	1.80	0.1	3.1	1E03 (Re)

(a) Note that the 241-AN-105 simulant is approximately 1.25 M in nitrate.

14. The impact of temperature will be investigated by repeating the equilibrium batch contact experiments outlined in part 13 but the samples will be agitated while being kept in a 65°C± 5°C temperature bath for at least 72 hours. The temperature will be recorded during these batch contact tests. An aliquot will be removed and rapidly filtered through a 0.2 micron syringe filter to remove the SuperLig® 639 resin. An aliquot will then be removed to determine the final solution activity.

15. The effect of total ionic strength changes at a constant NO_3^-/TcO_4^- ratio of 1E04 will be evaluated as shown in Table 5. The temperature will be recorded during these batch contact tests. The density of each solution in the absence of added technetium or rhenium stock solution will be determined by three independent measurements of the weight of a 1 mL aliquot. The precision of the balance must be at least ± 1 mg or better.

Table 5. Evaluating the effect of total ionic strength with a constant NO_3^-/TcO_4^- ratio. Contact conditions: $[NO_3]/[TcO_4]$ = 1E04, 72 hr contact time, ambient temperature (25 ± 5°C).

[Tc] stock solution.M	Stock sol. aliquot, µL	Test [Tc], M	$NaNO_3$, M	NaOH, M	NaCl, M	App. Ionic strength
0.0018	10	1.8E-06	0.018	0.01	5	5
0.0018	10	1.8E-06	0.018	0.01	1	1
0.0018	10	1.8E-06	0.018	0.01	0.5	0.5
0.0018	10	1.8E-06	0.018	0.01	0.1	0.1
0.0018	10	1.8E-06	0.018	0.01	0	0.03
0.0018 (Re)	10	1.8E-06 (Re)	0.018	0.01	1	1

16. To verify the capacity of the SuperLig® 639 under the testing conditions, a Tc loading isotherm will be generated under high [Na] loading conditions. The temperature will be recorded during these batch contact tests. The conditions for these batch contacts are summarized in Table 6.

Table 6. Tc loading isotherm. Contact conditions: room temperature (25 ± 5°C), 72 hours contact time, $[NO_3]/[TcO_4]$ = 1E02, $[Na]_T$ = 5 M

[Tc] stock solution.M	Stock sol. aliquot, µL	Test [Tc], M	$NaNO_3$, M	NaOH, M	NaCl, M	% Theor. capacity
1.8	100	0.018	1.80	0.1	3.1	200
1.8	50	0.009	0.9	0.1	4	100
1.8	10	0.0018	0.18	0.1	4.7	20
0.0018	100	1.8E-05	0.0018	0.1	4.9	0.2
0.0018	10	1.8E-06	0.00018	0.1	4.9	0.02

17. Two Cs containing stock solutions will be prepared. The first, ca. 1 M in total cesium, will be prepared by dissolving 1.95 grams of cesium nitrate in water to a total volume of 10 mL followed by spiking of the solution with a small amount of ^{137}Cs. The second cesium stock solution will be 10 mM in total cesium and will be prepared by a 1:100 volumetric dilution from the first cesium stock solution. The specific activity and radiochemical purity will be evaluated by gamma counting and the radiochemical purity of the material will be verified by ICP-MS or graphite furnace AA before use.

18. Two uranyl ion-containing stock solutions will be prepared. For the uranyl ion batch contacts, a stock solution ca. 1 M in uranium will be prepared by dissolving 5.02 grams of uranyl nitrate hexahydrate in water to a total volume of 10 mL, followed by spiking of the solution with a small amount of ^{233}U. The specific activity and radiochemical purity of the stock solution will be determined by alpha energy analysis of a sample aliquot before use. The total uranium content will be determined by laser fluorimetry before use.

19. To verify that the kinetics of the Superlig® 644 with cesium ion are such that a 72 hr contact time will yield an equilibrium distribution value, a series of contacts will be performed at room temperature (25 ± 5°C) under the conditions described in Table 7. The temperature will be

recorded during these batch contact tests. The sample's agitation will be stopped after 2, 4, 8, 24, 48, 72, 96 and 168 hr (1 week), respectively and the final solution activity measured.

Table 7. Cs kinetics under loading conditions. Contact conditions: ambient temperature (25 ± 5°C), [Na]/[Cs] = 1E04

[Cs] stock solution.M	Stock sol. aliquot, µL	Test [Cs], M	NaOH, M	$NaNO_3$, M	Na/Cs
1	5	0.0005	0.25	4.75	1.0E04

20. Two methods will be employed to verify the capacity of the SuperLig® 644. First, a Cs loading isotherm will be generated under high $[Na]_T$ loading conditions. The conditions for these batch contacts are summarized in Table 8. Each batch contact will be conducted at 25 ± 5°C, with the actual temperature being recorded.

Table 8. Cs isotherm under loading conditions. Contact conditions: ambient temperature, 72 hours contact time, [Na]/[Cs] = 100

[Cs] stock solution.M	Stock sol. aliquot, µL	Test [Cs], M	NaOH, M	$NaNO_3$, M	Na/Cs	% Theor. capacity
1	500	0.05	0.25	4.75	100	830
1	250	0.025	0.25	2.25	100	415
1	100	0.01	0.25	0.75	100	166
1	50	0.005	0.25	0.25	100	83
1	25	0.0025	0.25	0	100	42

The second approach involves a pH titration of a solution containing 0.1 grams of SuperLig 644 in the acid form with a standardized CsOH solution.

21. Since the pH conditions change substantially from loading (high pH) to stripping (0.5 M nitric acid) the effect on changing pH on the equilibrium distribution values of Cs with SuperLig® 644 at a constant [Na]/[Cs] ratio will be measured as described in Table 9.

Table 9. Effect of changing pH. Contact conditions: ambient temperature (25 ± 5°C), 72 hours contact time, [Na]/[Cs] = 5000

[Cs] stock solution.M	Stock sol. aliquot, µL	Test [Cs], M	pH or NaOH, M or HNO_3, M	$NaNO_3$, M	Na/Cs
1	10	0.001	2.5 (NaOH)	2.5	5000
1	10	0.001	1 (NaOH)	4	5000
1	10	0.001	0.1 (NaOH)	4.9	5000
1	10	0.001	0.01 (NaOH)	5	5000
1	10	0.001	9	5	5000
1	10	0.001	7	5	5000
1	10	0.001	5	5	5000
1	10	0.001	2	5	5000
1	9	0.001	0.5 (HNO_3)	4.5	5000

22. The effect of total ionic strength with a constant [Na]/[Cs] ratio of 5000 will be evaluated over the conditions described in Table 10. The density of the cesium stock solution will be determined by three independent measurements of the weight of a 1 mL aliquot. The precision of the balance must be at least ± 1 mg or better.

Table 10. Effect of changing ionic strength. Contact conditions: ambient temperature (25 ± 5°C), 72 hours contact time

[Cs] stock solution.M	Stock sol. aliquot, µL	Test [Cs], M	NaOH, M	$NaNO_3$, M	Na/Cs	Appr. Ionic Strength
1	10	0.001	0.25	4.75	5000	5
1	5	0.0005	0.25	2.25	5000	2.5
1	2	0.0002	0.25	0.75	5000	1
0.01	50	5E-05	0.25	0.00	5000	0.25

23. The effect of changing [Na]/[Cs] ratios on the cesium equilibrium distribution value will be evaluated over the conditions outlined in Table 11.

Table 11. Effect of changing [Na]/[Cs]. Contact conditions: ambient temperature (25 ± 5°C), 72 hours contact time

[Cs] stock solution.M	Stock sol. aliquot, µL	Test [Cs], M	NaOH, M	$NaNO_3$, M	Na/Cs	Appr. Ionic Strength
1	10	0.001	0.25	4.75	5000	5
1	5	0.0005	0.25	4.75	10000	5
1	1	1E-04	0.25	4.75	50000	5
0.01	50	5E-05	0.25	4.75	100000	5
0.01	10	1E-05	0.25	4.75	500000	5
0.01	5	5E-06	0.25	4.75	1000000	5

24. The effect of changing [K]/[Cs] ratios on the cesium equilibrium distribution value will be evaluated over the conditions outlined in Table 12.

Table 12. Effect of changing [K]/[Cs]. Contact conditions: ambient temperature (25 ± 5°C), 72 hours contact time

[Cs] stock solution.M	Stock sol. aliquot, µL	Test [Cs], M	KOH, M	KNO_3, M	K/Cs	Appr. Ionic Strength
1	10	0.001	0.25	0.75	1000	1
1	2	0.0002	0.25	0.75	5000	1
1	1	1E-04	0.25	0.75	10000	1
0.01	20	5E-05	0.25	0.75	50000	1
0.01	10	1E-05	0.25	0.75	100000	1
0.01	2	5E-06	0.25	0.75	500000	1

25. To verify that the kinetics of the Superlig® 644 with uranyl ion are such that a 72 hr contact time will yield an equilibrium distribution value, a series of contacts will be performed at room temperature under the conditions described in Table 13. The sample's agitation will be stopped after 2, 4, 8, 24, 48, 72, 96 and 168 hr (1 week), respectively and the final solution activity measured.

Table 13. UO_2^{2+} kinetics. Contact conditions: ambient temperature (25 ± 5°C), $[Na]/[UO_2^{2+}]$ = 1E03

$[UO_2^{2+}]$ stock solution.M	Stock sol. aliquot, µL	Test $[UO_2^{2+}]$, M	NaOH, M	$NaNO_3$, M	$[Na]/[UO_2^{2+}]$
1	10	0.001	0.25	0.75	1000

26. The effect of changing the sodium to uranyl molar ratio on the uranyl distribution value with SuperLig® 644 will be investigated over the conditions described in Table 14.

Table 14. Effect of changing $[Na]/[UO_2^{2+}]$. Contact conditions: ambient temperature (25 ± 5°C), 72 hours contact time

$[UO_2^{2+}]$ stock soln. M	Stock sol. aliquot, µL	Test $[UO_2^{2+}]$, M	NaOH, M	$NaNO_3$, M	$[Na]/[UO_2^{2+}]$	Appr. Ionic Strength
1	10	0.001	0.25	0.75	1000	1
1	2	0.0002	0.25	0.75	5000	1
1	1	1E-04	0.25	0.75	10000	1
0.01	20	5E-05	0.25	0.75	50000	1
0.01	10	1E-05	0.25	0.75	100000	1
0.01	2	5E-06	0.25	0.75	500000	1

Quality Control

Personnel shall conduct this activity in accordance with the CH2MHill approved quality assurance plan that implements the applicable requirements of 10 CFR 830.120, "Technical Support to CHG for Phase 1B2 Quality Assurance Project Plan", CHG-QAPjP, Rev.0.

References

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Hamm LL, FG Smith III, and DJ McCabe. 2000a. "Preliminaray Ion Exchange Modeling for Removal of Cesium from Hanford Waste Using SuperLig® 644 Resin." BNF-003-98-0220, Savannah River Technology Center, Westinghouse Savannah River Company, Aiken SC.

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Appendix B

Test Instruction and Data Sheets

10-19-

F17179

	LABORATORY RECORD BOOK NUMBER BNW- 57652	This document consists of ..... pages, copy no. .....
	TITLE (INDICATE SUBJECT CONTENT)	
AUTHOR - DIVISION AND DEPARTMENT		
PERIOD COVERED FROM	TO	

Route To	P. R. No.	Location	Route Date	Signature & Date
BM Rapko	3G639	P7-25	10/19/00	

MAKE-UP 100 mL FOR BATCH CONTACT TESTING.

Attachment A: Test Instructions for NCAW Waste Preparation

Starting Date 12/15/00 Balance check 10 g = 10,0000 g, weight serial # 99-J50603-4

LRB # 57652, p. 1

Balance No. 362-06-01-043, Next Calibration Date 02/2001

Balance No. _____, Next Calibration Date _____

Objective: 1) to prepare a standard stock solution of NCAW Waste (5M Na^+).
FILTER SOLUTIONS IF NECESSARY.

This solution was used for tests: _____, _____, _____

1. Test Solution Make-Up, NCAW Stock Solution (Date _____)

Component	FW,g	M	g/L	~~40 liters,g~~ g/100 mL	Weighed,g	Date
$NaNO_3$	85	0.258	21.9	2.193877	2.1933g.	12/15/00
Na_2SO_4	142.05	0.15	21.3	2.1308852	2.1308g.	
KNO_3	101.11	0.14	14.3	1.4155580	1.4152g.	
Na_2CO_3	105.99	0.23	24.7	2.4378989	2.4377g.	
$NaNO_2$	69.0	0.43	29.9	2.9670 1196	2.9670 g.	
$Na_2HPO_4 \cdot 7H_2O$	268.07	0.025	6.7	0.6702 268	0.6704g.	
$Al(NO_3)_3 \cdot 9H_2O$	375.15	0.43	162.6	16.1315 6503	16.1344g.	
NaF	42.0	0.089	3.75	0.3738 150	0.3741g.	
NaOH	40.0	3.4	136	13.6000 5440	13.5653g.	
$CsNO_3$	194.9	_____	_____	_____		

Species	NCAW,M
Na	5.0
K	0.14
Al	0.43
SO_4	0.15
OH(free)	~1.6
CO_3	0.23
F	0.089
NO_2	0.43
NO_3	1.69
PO_4	0.025
SO_4	0.15

By K. J. Crum
Laboratory No. 511
Balance No. 362-06-01-043
Reviewed by _____
Date _____

Cesium nitrate will be added in different amounts to give various sodium:cesium mole ratios.

Project No. _____ Date of Work _____
Entered By K. J. Crum Date 12/19/00
Disclosed To and Understood By _____
Signed 1 _____ Date _____
2 _____ Date _____

BNW57652
p. 2

Make-up of Rhenium Stock Solution

Dissolve 4.92 g. sodium perrhenate to a total volume of 10 mL with DI water. 12/18/00 rgf

Balance: 362-06-01-043
Calibration expires: 02/2001

Balance check
10 g = 10.0000 g
Weight serial # 99-J50603-4

$NaReO_4$ 4.9204 g.

Prepare dilution for Re analysis by ICP-MS

Pipet Calibra # 025302 9 set at 100.0 µl
T = 21.4 °C

0.0975 g.
0.0993 g.
0.0994 g.
0.0997 g.
0.0994 g.

Re-1 10.0678 g. H_2O + 100 µl Re = 10.2055 g.

Re-2 10.0535 g. H_2O + 100 µl Re = 10.1912 g.

Re-1 and Re-2 submitted for analysis by ICP-MS 12/18/00.

see page 4 for analytical report. KJL

Project No. _____ Date of Work _____
Entered By Kathleen J. Carman Date 12/19/00
Disclosed To and Understood By _____
Signed 1. _____ Date _____

Chem Rec_61 Prep date: 12/18/2000

BKM 57652 p.3

Preparation of Standardized 5M, 0.1 M, and 0.01M NaOH

WP# F17605 Requester: K. Carson

Request: *Brain Rapko has given me the job of making up jillions of stock solutions for batch contact tests he is going to do for CH2MHill. In order to make up the solutions, I need some different NaOH solutions made up and validated by titration. Please make up: 0.01 M NaOH, 2 liters: 0.1 M NaOH, 2 liters: 5.0 M NaOH, 250 ml*
Charge to F17605. Thanks! ---Katharine Carson

Preparation: Use 50% NaOH (19.06N) Fisher stock solution (CMS # 52456) to prepare the 5M NaOH -- then prepare 0.1M NaOH from this stock and standardize this solution and adjust 5M NaOH accordingly. Then prepare the 0.01M solution and also standardize this 10X dilution.

Calculations : $\frac{250\ mL * 5M\ NaOH}{19.06M\ NaOH}$ = 65.6 mL conc NaOH --- to 250 mL with DI water.

$\frac{2000mL * 0.1M\ NaOH}{5\ M\ NaOH}$ = 40.0 mL of ~ 5M NaOH --- to 2000 mL with DI water.

Standardization : Use NIST SRM 84j, Potassium Acid Phthalate KHC8H4O4 (KAP) --CMS# 52232
Technique used will be via hand-titration to the phenopthalein endpint. A good titration would use about 20-25 mL of a 50 mL burrette. Hence. since KHC8H404 = 204.23 g/mole or mg/meq
20 mL * 0.1M NaOH = 2 meq. and 2 meq of KAP = 204.22 mg/meq * 2 = ~ 408 mg weigh on 5-place balance
Target wt for KAP to titrate 0.01M NaOH will be about 40 mg.

NaOH Molarity verification performed on ~ 0.1 M NaOH prep.

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.1M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.40351	20.95	0.09431	
2	0.39661	20.62	0.09418	
3	0.41712	21.65	0.09434	
		Ave=	**0.09428**	0.0001
			certified value	

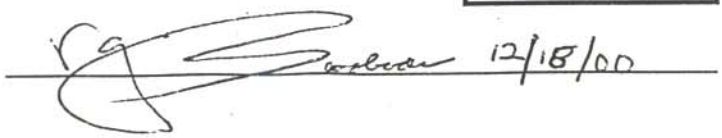
0.40351 g
0.39661 g
0.41712 g

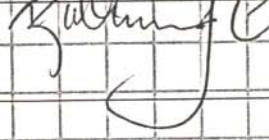
5M NaOH is revised as follows based on standardized 0.09428M NaOH
$\frac{2000\ mL * 0.09428\ M\ NaOH}{40.0mL}$ = **4.71M NaOH**
and:
$\frac{2000mL * 0.01M\ NaOH}{0.09428}$ = 212. 1 mL of 0.0943M to 2L with DI H2O

NaOH Molarity verification performed on 0.01 M NaOH dilution

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.01M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.04822	23.78	0.00993	
2	0.03937	17.82	0.01082	
3	0.02939	14.55	0.00989	
		Ave=	**0.01021**	0.0005
			certified value	

0.04822 g
0.03937 g
0.03572 g

Analyst/Date  12/18/00

Project No. Date of Work
Entered By  Date 12/19/00
Disclosed To and Understood By
Signed 1. Date
2. Date

ADVANCED INORGANIC ANALYSIS GROUP, NATIONAL SECURITY DIVISION
BATTELLE, PACIFIC NORTHWEST NATIONAL LABORATORY, RICHLAND, WA

BNW57652 p.4

RESULTS

Name: **Brian Rapko**
Work Package #: **F17605**
Report Date: 1/4/01

Reviewed By: S.Q. Slate (date) 1-5-01
Reference: on file 329/114, ICP Printout 01-03-01
Analyst: LMP Thomas samp.

Concentration: **as noted**

Sample Log-in #	Sample ID	Re			
		µg/ml	M $NaReO_4$	% RPD	
01-12-14	RE-1	3350	0.018_0	0.27% RPD	
01-12-14	RE-1 duplicate	3360	0.018_0		
01-12-14	RE-1 MS	101% spike recovery			
01-12-15	RE-2	3390	0.018_2	0.020% RPD	
01-12-15	RE-2 duplicate	3390	0.018_2		

Analytical Equipment: Inductively Coupled Argon Plasma Spectrometer (ICP), IRIS/AP Jarrell-Ash (WD25801)
Mettler AK160 balance (366-06-01-005), "S" class Troemner weights (22549-523)

Procedure(s): AIAL-01 Rev. 0

Sample Matrix: 2% HNO_3

Signed Date

Make-up of Technetium-99 Stock Solution

BNW 57652 p.5

Dissolve 2.3 g. technetium oxide in ammonium hydroxide/hydrogen peroxide and evaporate to dryness at room temperature. — done 12/18/00 K.J. Carson

Redissolve the solid ammonium pertechnetate to a total volume of ~~10~~ mL with DI water.

Balance: 384-06-01-008

Calibration expires: 08/2001

NH_4OH / H_2O_2 step repeated 2X.

Dissolve pertechnetate in ~ 35 ml H_2

3:30 pm → appears cloudy.

TcO_2 2.303 g.

Pipet # 0831412 T = 22.5°C 1/12/01 rcfe

10.0562 g.
10.0544 g.
10.0858 g.
10.0682 g.
10.0543 g.

used for preparing dilutions of Tc-stock.

~~Prepare 1:1000 dilution for second Tc-99 stock solution.~~ rcfe

Pipet # 033120

used to make up dilutions and counting samples

check Tc-99 stock

Filter with 0.45 μ filter

(stock dilution) 100 λ → 10.1 ml (DI water) →

(dilution #2) 100 λ → 10.1 ml (DI water) →

100 λ → 15 ml (Ultima Gold)

$$\left(\frac{10.1\ ml}{0.1\ ml}\right)\left(\frac{10.1\ ml}{0.1\ ml}\right)\left(\frac{15.0\ ml}{0.1\ ml}\right)\frac{\text{counts}}{15\ ml}$$

Blank

100 λ DI H_2O → 15 ml (Ultima Gold)

1/15/01 Filtered all Tc-99 stock using 0.45 μ filter, some solid material still undissolved. rcfe.

Signed 1. _____ Date _____

2. K.J. Carson Date 1/19/01

Tc-99 stock
(rough check)
1 minute count.

Time: 1.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0	40.0	0	0.0	0.00
Region B:	40.0	400	0	0.0	0.00
Region C:	2.0	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

0.1 ml ⇄ 10.1ml → (DI water)
0.1 ml ⇄ 10.1ml → (DI water)
0.1 ml → 15 ml (Ultima Gold)

Blank 0.1 ml H_2O → 15 ml (Ultima Gold)

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
blank 1	1.00	41.00	31.23	12.00	57.74	52.37	27.64	83.398	538.13	
stock #2	1.00	3715.22	3.28	10759.8	1.93	14445.8	1.66	180.65	543.67	
stock #3	1.00	3832.05	3.23	10615.0	1.94	14412.4	1.67	178.26	541.54	

BNW 57652
p.6

Disclosed To and Understood By
Signed 1. Date 1/19/01

Carson, Katharine J

From: Rapko, Brian M
Sent: Sunday, January 14, 2001 5:28 PM
To: Carson, Katharine J
Subject: RE: Rough cut Tc-99 counting data

BNWO 57652 p.7

Katharine: I would go ahead and filter and use as is - the current activity is plenty. Using the numbers you gave (and taking 1.07E04 cpm for the measured activity) and since the specific activi of Tc-99 is 0.017 Ci/g, the activity of the stock solution comes out to 4.8E-04 Ci/mL. This translate to about 2.8E-02g/mL in the stock solution or about 1 gram total Tc (35 mLs of stock solution). Wa that about the amount of Tc you weighed out?

- Brian

From: Carson, Katharine J
Sent: Friday, January 12, 2001 4:42 PM
To: Rapko, Brian M
Subject: Rough cut Tc-99 counting data

I got some numbers for you - at least enough to give you an idea.

The Tc stock is in about 35 mL water, no large chunks visible but it is still cloudy.

I filtered a small portion using a 0.45 micron filter, then made two successive 0.1 mL to 10 mL dilutions.

I took 0.1 mL of the second dilution into 15 ml Ultima Gold for counting. I also made up a blank of 0.1 mL water ir cocktail.

I did a one minute count of each sample.

Blank	12 CPM
Tc stock #1	10760 CPM
Tc stock #2	10615 CPM

Whipping out my calculator. . . I get 1.09E+09 counts/mL.

I can repeat all this with longer count times, and making dilutions by weight to give you something "official" for the records. Since the Tc stock hasn't cleared up yet, I thought you might want to have me dilute it out a little more, o else filter all of it to get a uniform solution.

Katharine Carson

katharine.carson@pnl.gov
509-376-4299

Project No. _____ Date of Work _____

Entered By Katharine J. Carson Date 1/19/01

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

Time: 10.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	40.0	0	0.0	0.00
Region B:	40.0 -	400	0	0.0	0.00
Region C:	2.0 -	2000	0	0.0	0.00

BNW5 7652 p.9

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
BLK 1	10.00	22.90	13.22	12.80	17.68	41.75	9.79	130.70	537.27	
3K1A 2	10.00	3611.99	1.05	10391.5	0.62	13973.4	0.54	177.97	537.12	
3K1B 3	10.00	3667.18	1.04	10417.8	0.62	14053.5	0.53	177.56	539.93	
3K2A 4	10.00	36373.5	0.33	106442	0.19	142451	0.17	180.21	541.54	
3K2B 5	10.00	37490.5	0.33	108336	0.19	145446	0.17	180.71	549.16	

Disclosed To and Understood By:
Signed 1. Date
2. Date 1/15/01

For Tc Stock 2

counts/mL =	1.086E+06	sample A
counts/mL =	1.073E+06	sample B
Average =	1.079E+06	

BNW 5 7652
p. 11

Entered By _____ Date _____

Disclosed To and Understood By [signature] Date 1/19/01

Signed 1. _____ Date _____

2. _____ Date _____

Assay of Tc-99 Stock Solutions for Equilibrium Batch Contact Testing

Balance 362-06-01-056
Mettler PM400
calibration expires 8/2001

BNW 57652 p.10

Make up a Tc Stock 2 by dilution of original Tc stock 1:1000 in water.

Tare weight, g	Vial & Water	Vial, water, Tc	Water, g	Tc, g	Dilution factor
13.209	23.372	23.38	10.163	0.008	1270.375

Make up two successive 100X dilutions of the original Tc stock.

Tare weight, g	Vial & Water	Vial, water, Tc	Water, g	Tc, g	Dilution factor	
13.438	23.577	23.679	10.139	0.102	99.40196	dilution #1
13.432	23.612	23.711	10.18	0.099	102.8283	dilution #2
				(used to prepare counting samples)		

Preparation of samples for LSC

Tare weight, g	Vial & Tc	Vial, Tc, cocktail	Tc, g	Cocktail, g	Dilution factor	
13.367	13.467	28.438	0.1	14.971	149.71	Tc stock #1 sample A
13.251	13.351	28.373	0.1	15.022	150.22	Tc stock #1 sample B
13.296	13.394	28.43	0.098	15.036	153.4286	Tc stock #2 sample A
13.11	13.211	28.228	0.101	15.017	148.6832	Tc stock #2 sample B

Blank counting sample made up with 0.1 mL DI water in 15 mL cocktail

Counting data

Sample	CPM
Blank	12.8
Stock 1 A	10391.5
Stock 1 B	10417.8
Stock 2 A	106442
Stock 2 B	108336

For Tc Stock 1

counts/mL =	1.061E+09	sample A
counts/mL =	1.064E+09	sample B
Average =	1.062E+09	

Prepared by Katharine Carson 1/18/01

Disclosed To and Understood By

Signed 1. Katharine Carson Date 1/19/01

2. Date

BMW 57652 p. 8

	(NO cap) Vial Tare Weight	Vial + water	Vial, water + Tc
# stock vial stir bar)	13.209 g.	23.372 g.	23.380 g.
il #1 vial stir bar)	13.438 g.	23.577 g.	23.679 g.
il. #2 vial stir bar)	13.432 g.	23.612 g.	23.711 g.

Calibra 10 ml pipet 0831428
" 10 ul pipet 028369
" 100 ul pipet 053081

	Tare wt.	Vial + ~~calculated~~ Tc	Vial, cocktail, Tc
stock 1 A	13.367 g.	13.467 g.	28.438 g.
stock 1 B	13.251 g.	13.351 g.	28.373 g.
stock 2 A	13.296 g.	13.394 g.	28.430 g.
stock 2 B	13.110 g.	13.211 g.	28.228 g.

Lab 516 1/16/01 KJ Cann

Balance 362-06-01-056
Mettler PM400
calibration expires 8/2001

Entered By _____ Date _____

Disclosed To and Understood By

Signed 1. Kathleen J. Cann Date 1/19/01

2. _____ Date _____

	Stock Solutions:							Density, gm/mL, 1 mL aliquots				
	NaCl, M	$NaNO_3$, M	NaOH, M	Total Vol, mL	Actual NaCl, g	Actual $NaNO_3$, g	Actual NaOH	Wt 1, gm	Wt 2, gm	Wt 3, gm	Average Density, g/mL	
FW	58.44	84.99	40									
a	3.1	1.8	0.1	500	90.5877	76.5012	0.0943 M					
b	3.1	0.018	0.01	250	45.2918	0.3827	0.0102 M					
d	4.7	0.18	0.1	100	27.4735	1.5272	0.0943 M					
e	4.9	0.0018	0.1	100	28.6408	0.0157	0.0943 M					
f	~~6~~	~~0.018~~	~~0.01~~	~~50~~	~~17.0766~~	~~0.0762~~	~~0.0102 M~~	will not dissolve				
g	5	0.018	0.01	50	14.6106	0.0773	0.0102 M	1.1818	1.1841	1.1845	1.1835	
h	1	0.018	0.01	50	2.9201	0.0779	0.0102 M	1.0356	1.0381	1.0383	1.0373	
l	0.5	0.018	0.01	50	1.4683	0.0763	0.0102 M	1.0130	1.0145	1.0169	1.0148	
j	0.1	0.018	0.01	50	0.2889	0.0766	0.0102 M	1.0016	1.0074	1.0053	1.0048	
k	4.9	0.00018	0.1	50	14.3403	0.0008	0.0943 M					
l	4.7	0.18	0.1	50	13.7286	0.7624	0.0943 M					
m	4	0.9	0.1	50	11.6974	3.8155	0.0943 M					
n	4.9	0.018	0.1	50	14.3210	0.0756	0.0943 M					
o	4	0.08	1	50	11.7022	0.3376	2.0604 g					
p	2.5	0.018	2.5	50	7.3115	0.0765	4.9981 g					
q	1	0.018	4	50	2.9244	0.0756	7.9850 g					
r	0.1	0.018	4.9	50	0.2926	0.0768	9.8709 g					
s	xx	5	0.1	500	xx	212.47	0.0943 g					
t	xx	5	0.01	500	xx	212.48	0.0102 M					
u	xx	0.5	0.01	50	dilute (t) with 0.01 M NaOH -			5	ml of (t)			
v	xx	0.1	0.01	50	dilute (t) with 0.01 M NaOH -			1	ml of (t)			
w	xx	0.01	0.01	50	dilute (t) with 0.01 M NaOH -			0.1	ml of (t)			
x	xx	0.001	0.01	50	dilute (t) with 0.01 M NaOH -			0.01	ml of (t)			
z	xx	5	xx	50	xx	21.26	xx	**pH 9**	adj with NaOH/HNO3		Final pH =	9.41
aa	xx	5	xx	50	xx	21.26	xx	**pH 7**	adj with NaOH/HNO3		Final pH =	7.18
ab	xx	5	xx	50	xx	21.26	xx	**pH 5**	adj with NaOH/HNO3		Final pH =	5.22
ac	xx	5	xx	50	xx	21.26	xx	**pH 2**	adj with NaOH/HNO3		Final pH =	2.14
ad	xx	5	0.5M HNO_3	50	xx	21.26	xx					
ae	xx	2.5	0.01	50	dilute (t) with 0.01 M NaOH -			25	ml of (t)			
af	xx	0.5	0.01	50	dilute (t) with 0.01 M NaOH -			5	ml of (t)			
ag	xx	0.04	0.01	50	dilute (t) with 0.01 M NaOH -			0.4	ml of (t)			
ah	xx	5M KNO_3	0.01M KOH	250								

Other	Value	Actual
con HNO3 M	15.77	1.585 mL
FW KNO_3	101.11	25.2910 g
FW KOH	56.11	0.1565 g

Balances	Calibraton Date
360-06-01-035	2/1/01
1113120467	2/9/00
360-06-01-009	8/1/01

pH Meter: Corning 345
Electrode: Corning Semi-micro combo

BW 57657 p.12

1/2/01 jrd, kjc

BNL 57652
p.13

Deschane, Jaquetta R

From: Rapko, Brian M
Sent: Thursday, December 28, 2000 9:27 AM
To: Deschane, Jaquetta R
Subject: RE: KNO3-KOH Solution

Change it from 5 M KNO3/0.01 M KOH to 1 M KNO3/0.01 M KOH. I just checked the KNO3 solubility and indeed we are about 30% over the maximum KOH solubility in room temperature wate Oops.

- Brian

From: Deschane, Jaquetta R
Sent: Thursday, December 28, 2000 7:48 AM
To: Rapko, Brian M
Subject: KNO3-KOH Solution

Project No. _____ Date of Work _____

Entered By _____ Date 1/19/01

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

Notes on solution make-up

Balance 362-06-01-043
Calibration expires 02/2001

Date 12/21/00
By [signature]

Balance check 10g = 10.0000g, weight serial # 99-J50603-4

BNW 57652 p.14

Pipet # Expandorf N32671
Weight checks
0.9980 g.
0.9981 g.
0.9973 g.
0.9961 g.
0.9962 g.
T = 21.4°C

Pipet # _____
Weight checks

Pipet # _____
Weight checks

Pipet # _____
Weight checks

Balance 360-06-01-009
expires 8/2001

~~Used~~ Did not use!

Balance check 100g = 100.001 g.
weight serial # 99-J50658-24

used for solution "t" - $NaNO_3$

[signature]

Balance 362-06-01-023 (Lab 510)
expires 8/2001

Disclosed To and Understood By
Signed 1. [signature] Date 1/9/01

Pipet weight checks

Balance 360-06-01-035
Calibration expires 2/01

Date 12/29/00
By J DesChane

Pipet # H30973
Weight checks
9.99914
10.00425
10.00762

Pipet # H30973
Weight checks
1.00011
0.99305
0.99991

Pipet # 4710
Weight checks
0.00947
0.00951
0.00923

Pipet # H30213
Weight checks
0.09832
0.10234
0.10200

Pipet # H30213
Weight checks
0.40045
0.39863
0.39939

Pipet # H30213
Weight checks
1.00183
1.00063
0.99938

Pipet # H30213
Weight checks
01/02/01 1.00246
1.00202
1.00167

Pipet #
Weight checks

Entered By _____ Date _____
Disclosed To and Understood By
Signed 1: _____ Date 1/19/01
2: _____ Date _____

TRACEABLE®

Certificate of Calibration for Temperature Probe

Certificate Number	Model Number	Serial Number
C374352	NEW 61220-604	20108266

This Temperature Probe was calibrated against National Institute of Standards and Technology Traceable Instrumentation. This calibration complies with the requirements of ISO 9000 Certification. All values are in accordance with ITS-90 (International Temperature Scale of 1990).

Calibration Test Information

Test Equipment	Serial Number	Calibration Due Date
HART PRECISION BATH, 7011	56063	06/23/00

NIST Traceable Test Number(s)

256495, ITS-90

Accuracy

Testing was performed on the unit as shown below. Test results are as follows:

Standard °C	Reading °C	Standard °F	Reading °F
25.00	25.1	77.00	77.2

BAW 57652 p.16

The maximum error of this Temperature Probe at the time of calibration did not exceed the specified accuracy of: ± 0.1°C

Test Conditions:	Temperature °C	Relative Humidity %	Barometric Pressure (inHg)
	23.00	41.00	29.91

Maintaining Accuracy

Once measured and calibrated your Temperature Probe should maintain it's accuracy. There is no exact way to determine how long calibration will be maintained. Electronics change little, if any at all, but can be affected by aging, temperature, and shock.

Calibration Dates

Factory Calibration Date	Next Calibration Due Date
06/05/00	06/05/02

We recommend that the unit's accuracy be recertified on an annual basis for those users with critical needs such as accreditation demands, government specifications, or ISO 9000 requirements.

Tester's Initials	Metrology Manager
PUL	William Berry

Recalibration

For factory calibration and recertification of this Temperature Probe contact:

Control Company • 308 West Edgewood • Friendswood, Texas 77546 • USA
Phone 281 482-1714 • Fax 281 482-9448

Control Company is an ISO 9001 Quality Certified Company.
ISO 9001 Certificate No. 98-HOU-AQ-1805

Signed 1. _____ Date _____

TRACEABLE®

Certificate of Calibration for Digital Thermometer

Certificate Number	Model Number	Serial Number
C373297	NEW 61220-601	20207313

This Electronic Digital Thermometer was calibrated against National Institute of Standards and Technology Traceable Instrumentation. This calibration complies with the requirements of ISO 9000 Certification. All values are in accordance with ITS-90 (International Temperature Scale of 1990).

Calibration Test Information

Test Equipment	Serial Number	Calibration Due Date
THERMISTOR CALIBRATION STD	98179306	08/25/00

NIST Traceable Test Number(s)

811/256331

Accuracy

Testing was performed on the unit as shown below. Test results are as follows:

Standard °C	Reading °C	Standard °F	Reading °F
5.00	5.0	41.00	41.0
25.00	25.0	77.00	77.0
45.00	45.0	113.00	113.0
100.00	100.0	212.00	212.0

BNW57652 p.17

The maximum error of this Electronic Digital Thermometer at the time of calibration did not exceed the specified accuracy of: ± 0.2°C

Test Conditions:	Temperature °C	Relative Humidity %	Barometric Pressure (inHg)
	29.00	41.00	29.83

Maintaining Accuracy

Once measured and calibrated your Electronic Digital Thermometer should maintain it's accuracy. There is no exact way to determine how long calibration will be maintained. Electronics change little, if any at all, but can be affected by aging, temperature, and shock.

Calibration Dates

Factory Calibration Date	Next Calibration Due Date
06/05/00	06/05/02

We recommend that the unit's accuracy be recertified on an annual basis for those users with critical needs such as accreditation demands, government specifications, or ISO 9000 requirements.

Tester's Initials	Metrology Manager
PUL	Wallace Berry

Recalibration

For factory calibration and recertification of this thermometer contact:

Control Company • 308 West Edgewood • Friendswood, Texas 77546 • USA
Phone 281 482-1714 • Fax 281 482-9448.

Control Company is an ISO 9001 Quality Certified Company.
ISO 9001 Certificate No. 97 177

Signed 1. _____ Date _____

2. _____ Date 1/30/01

Make-up of Cesium-137 Stock Solutions

Obtain vial of nominally 5 mCi Cs-137 in dilute HCl. Evaporate to dryness.
Dissolve Cs-137 residue in minimal amount of DI water.
Divide Cs-137 between 2 cesium stock solutions.

1 M Cs solution

RG Cinn 1/26/01

Weigh out 0.975 g. cesium nitrate into a 5 mL volumetric flask.
Dissolve using Cs-137 - DI water. Rinse Cs-137 residue with DI water, transfer washes to flask.

Balance: 362-06-01-043
Calibration expires: 2/01

10g = 10.0000 g,
weight serial # 99-J50603-4

0.9750 g.

0.01 M Cs solution

Weigh out 9.75 mg cesium nitrate into a 5 mL volumetric flask.
Dissolve using Cs-137 - DI water. Rinse Cs-137 residue with DI water, transfer washes to flask.

Balance: 362-06-01-040
Calibration expires: 2/01

100 mg = 0.09999 g.
weight std 812-86-02-026

00.00977 g.

Remove a 0.1 mL aliquot of each stock for gamma counting.

BNW 57652
p.18

Project No. _____ Date of Work _____
Entered By R Cinn Date 1/30/01
Disclosed To and Understood By _____
Signed 1. _____ Date _____

Instructions for the preparation of 137-Cs stock solutions.

BNW 57652 p. 19

1) Background.

We need two solutions. The first is 1 M in Cs and contains substantial activity so that counting of a 1 mL solution is straightforward, even if up to 99% of the total activity is removed. The second is 0.01 M in Cs and contains the same activity. Given that a 1 mL aliquot can be used, the assumption of a 100,000 dpm activity seem plenty. So a stock solution capable of delivering a 10 mL of a 100,000 dpm/mL 137Cs solution is needed. The typical spike solution is 10 microliters into 10 mL or a 1E03 dilution. This makes the required activity of the stock solution 1E06 dpm/mL multiplied by 1E03 or a 1E09 dpm/mL solution. Given that 1 Ci is about 1E12 dpm a ca. 1 mCi/mL concentration of Cs-137 is required.

The current estimate is that about 2.5 mL of each stock solution is needed. To be safe, prepare 5 mL of stock solution. Note that the specific activity of 5 mCi of 137Cs is only 5.76E-05 grams. To prepare 5 mL of each stock solution, ca. 5 mCi source of 137Cs should be evaporated to dryness at room temperature to minimize any aerosols that might spread 137Cs contamination.

2) Stock solution preparation:

To generate the 1 M total Cs stock solution, place 5 mmol (194.91 g/mol * 0.005 = 0.975 grams) of $CsNO_3$ or 5 mmol (168.36 grams/mol * 0.005 = 0.842 grams) of CsCl into a 5 mL volumetric flask. Rinse the ca. 5 mCi 137-Cs residue repeatedly with small (0.5-1mL) portions of DI water, transferring each wash into the 5 mL volumetric flask. Cap and agitate the volumetric flask, remove a 0.1 mL aliquot into a 2 dram vial containing 1 mL DI water, and measure the sample activity on a gamma counter.

Repeat the process for the 0.01 M Cs stock solution, only here the 5 mL volumetric flask will containing 9.75 mg of $CsNO_3$ or 8.42 mg of CsCl.

first counting samples!

Bal. 384-06-01-008 cal. exp 8/2

	Tare weight vial	Vial + H_2O	Vial, H_2O, Cs
stock #1	8.074 g.	9.086 g.	9.203 g.
stock #2	8.093 g.	9.098 g.	9.199 g.

mg Cs 1/26/01

Too hot for detector — make up more dilute samples for counting.

Entered By _____ Date _____

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Signed 1. _____ Date 1/30/01

2. _____ Date _____

1/29/01 [signature]

BNW57652
p. 20

make-up	Tare wt. Vial + stir bar	Vial, bar + H_2O	Vial, bar, H_2O + Cs
Cs stock #1 Dilution 1 (0.1 ml → 10 ml) stock	13.435 g.	23.620 g.	23.737 g.
Cs stock 1 Dilution 2 (0.1 ml → 10 ml) dil #1	13.409 g.	23.464 g.	23.565 g.
Cs stock 1 counting sample (10 λ → 1 ml) dil #2	~~8.045 g.~~ too dilute!	~~9.053 g.~~	~~9.063 g.~~
Cs stock #2 Dilution 1 (0.1 ml → 10 ml) stock	13.154 g.	23.248 g.	23.350 g.
Cs stock 2 Dilution 2 (0.1 ml → 10 ml) dil #1	13.485 g.	23.554 g.	23.658 g.
Cs ~~stock 2~~ counting sample (10 λ → 1 ml) dil #2	~~8.122 g.~~ too dilute!	~~9.135 g.~~	~~9.151 g.~~

Balance 384-06-01-008
exp. 8/2001

will take 1 ml each of dilutions 1 and 2 of both stocks for counting.

1/29/01 [signature]

BNW 57652 p. 21

	Tare wt.	Vial + Cs solution
Cs stock 1 Dilution #1	8.196 g.	9.194 g.
Cs stock 1 Dilution #2	8.146 g.	9.156 g.
Cs stock 2 Dilution #1	8.073 g.	9.084 g.
Cs stock 2 Dilution #2	8.145 g.	9.155 g.

Balance 384-06-01-008

cal. expires 8/2001

(1 ml each dilution for counting sample)

Project No. _____ Date of Work _____

Entered By [signature] Date 1/30/01

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1/29/01

Cs

10 λ stock → 10 ml total

count 1 ml of 10 ml dilution.

	Tare weight	Vial + H_2O	Balance 362-06-01-043 cal exp. 2/01
Cs stock 1 counting dil. (10 λ → 10 ml)	13.4471 g.	23.6539 g.	+ 10 λ stock 1
Cs stock 2 counting dil. (10 λ → 10 ml)	13.5100 g.	23.5093 g.	+ 10 λ stock 2

Counting samples	Tare weight	Vial + Cs solution	Balance 384-06-01-008 cal. exp. 8/01
Cs stock 1	8.074 g.	9.096 g.	
Cs stock 2	8.167 g.	9.182 g.	

Project No. _____ Date of Work _____

Entered By _____ Date 1/30/01

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Assay of Cs-137 Stock Solutions for Equilibrium Batch Contact Testing

Balance 384-06-01-008
calibration expires 8/2001

BNW 57652 p. 23

Make up two successive 100X dilutions of the original Cs stocks.

Tare weight, g	Vial & Water	Vial, water, Cs	Water, g	Cs, g	Dilution factor	
13.435	23.62	23.737	10.185	0.117	87.1	dilution #1 Cs stoc
13.409	23.464	23.565	10.055	0.101	99.6	dilution #2 Cs stoc
13.154	23.248	23.35	10.094	0.102	99.0	dilution #1 Cs stoc
13.485	23.554	23.658	10.069	0.104	96.8	dilution #2 Cs stoc

Take 1 mL of each dilution for gamma counting

Tare weight, g	Vial & Cs solution	Cs solution, g	
8.196	9.194	0.998	dilution #1 Cs stock 1
8.146	9.156	1.01	dilution #2 Cs stock 1
8.073	9.084	1.011	dilution #1 Cs stock 2
8.145	9.155	1.01	dilution #2 Cs stock 2

Gamma counting data All spectrum files are located on c:\mca\data\cesium

All counts were one minute

Sample ID	Net counts	Activity, Cs-137	Data file
IPL source	168	0.0396 uCi	130stnd.spc
Cs stock 1 dilution #2	316	0.1186 uCi	sk1dil2.spc
Cs stock 2 dilution #2	72	0.0309 uCi	sk2dil2.spc

Dilution #1 samples have too much activity to count.

Take 10 lambda of each stock solution, dilute into 10 mL, take 1 mL for gamma counting

Tare weight, g	Vial & water	Water, g	Stock, g.	Dilution factor	
13.4471	23.6539	10.2068	0.01	1021	Cs stock 1
13.51	23.5093	9.9993	0.01	1000	Cs stock 2

Tare weight, g	Vial & Cs solution	Cs solution, g	
8.074	9.096	1.022	Cs stock 1
8.167	9.182	1.015	Cs stock 2

Prepared by Katharine Carson 1/31/01

Signed 1. _____ Date _____

2. _____ Date 1/31/01

Gamma counting data All spectrum files are located on c:\mca\data\cesium

Sample ID	Net counts	Activity, Cs-137	Data file
IPL source	168	0.0396 uCi	130stnd.spc
Cs stock 1	698	0.6597 uCi	10lsk1.spc
Cs stock 2	270	0.4161 uCi	10lsk2.spc

All counts are one minute.

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p. 24

Prepared by Katharine Carson 1/31/01

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Signed 1. _____ Date 1/31/01

Note: ~~Activity~~ conc. of Tc stock solutions based on Activity

^{99}Tc mass = 98.906 Half life = 2.13 E05 years ✱ Chart of th nuclides, N

$$\frac{\text{\# disintegrations/min}}{g} = \frac{\ln(2)}{\epsilon\,(\text{min})} \times \frac{\text{molecules Tc}}{\text{mol Tc}} \div \frac{g\ Tc}{\text{mol Tc}} = \left(\frac{\ln(2)}{2.13E5 \times 365.25 \times 2}\right) \times \frac{6.022E23}{98.906}$$

$$= 3.767E10\ \frac{dpm}{g\ Tc}$$

since $2.22E12\ \frac{dpm}{Ci} \Rightarrow \frac{1.697E-02\ Ci}{g\ Tc}$

sec pg 10-11 Stock soln. #1 $1.062E09\ \frac{cpm}{mL} \times 1.062E09\ \frac{dpm}{mL}$

Stock soln #2 $1.079E06\ \frac{cpm}{mL} \times 1.079E06\ \frac{dpm}{mL}$

stock soln #1

$1.062E05\ \frac{dpm}{mL} \div 2.22E12\ \frac{dpm}{Ci} = 4.784E-04\ \frac{Ci}{mL} \div 1.697E-02\ \frac{Ci}{g\ Tc} = 2.819E$

Stock soln #2

$1.079E06\ \frac{dpm}{mL} \div 2.22E12\ \frac{dpm}{Ci} = 4.86E-07\ \frac{Ci}{mL} \times 1.697E-02\ \frac{Ci}{g\ Tc} = 2.864E-05\ \frac{g}{mL}$

soln #1 $2.819E-02\ \frac{g\ Tc}{mL} = 2.819E1\ \frac{g\ Tc}{L} \div 98.906\ \frac{g\ Tc}{\text{mol Tc}} = 0.285\ M$

soln #2 $2.864E-05\ \frac{g\ Tc}{mL} = 2.864E-02\ \frac{g\ Tc}{L} \div 98.906\ \frac{g\ Tc}{\text{mol Tc}} = 2.896 \times 10^{-4}\ M = 0.289$

Project No. _____ Date of Work _____

Entered By B. Rapko Date 2-1-04 (DC)

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

6

BNW 57652 p. 26

Chem Rec_61 Prep date: 12/18/2000

Preparation of Standardized 5M, 0.1 M, and 0.01M NaOH

WP# F17605 Requester: K. Carson

Request: *Brain Rapko has given me the job of making up jillions of stock solutions for batch contact tests he is going to do for CH2MHill. In order to make up the solutions, I need some different NaOH solutions made up and validated by titration. Please make up: 0.01 M NaOH, 2 liters: 0.1 M NaOH, 2 liters: 5.0 M NaOH, 250 ml*
Charge to F17605. Thanks! ---Katharine Carson

Preparation: Use 50% NaOH (19.06N) Fisher stock solution (CMS # 52456) to prepare the 5M NaOH -- then prepare 0.1M NaOH from this stock and standardize this solution and adjust 5M NaOH accordingly. Then prepare the 0.01M solution and also standardize this 10X dilution.

Calculations : $\frac{250\ mL * 5M\ NaOH}{19.06M\ NaOH}$ = 65.6 mL conc NaOH --- to 250 mL with DI water.

$\frac{2000mL * 0.1M\ NaOH}{5\ M\ NaOH}$ = 40.0 mL of ~ 5M NaOH --- to 2000 mL with DI water.

Standardization : Use NIST SRM 84j, Potassium Acid Phthalate KHC8H4O4 (KAP) --CMS# 52232
Technique used will be via hand-titration to the phenopthalein endpint. A good titration would use about 20-25 mL of a 50 mL burrette. Hence. since KHC8H404 = 204.23 g/mole or mg/meq
20 mL * 0.1M NaOH = 2 meq. and 2 meq of KAP = 204.22 mg/meq * 2 = ~ 408 mg weigh on 5-place balance
Target wt for KAP to titrate 0.01M NaOH will be about 40 mg.

NaOH Molarity verification performed on ~ 0.1 M NaOH prep.

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.1M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.40351	20.95	0.09431	
2	0.39661	20.62	0.09418	
3	0.41712	21.65	0.09434	
		Ave=	**0.09428**	0.0001
			certified value	

5M NaOH is revised as follows based on standardized 0.09428M NaOH

$\frac{2000\ mL * 0.09428\ M\ NaOH}{40.0mL}$ = **4.71M NaOH**

and:

$\frac{2000mL * 0.01M\ NaOH}{0.09428}$ = 212. 1 mL of 0.0943M to 2L with DI H2O

NaOH Molarity verification performed on 0.01 M NaOH dilution

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.01M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.04822	23.78	0.00993	
2	0.03937	17.82	0.01082	
3	0.02939	14.55	0.00989	
		Ave=	**0.01021**	0.0005
			certified value	

Analyst/Date 12/18/01

C-rec_61.XLS

02/26/2

Signed 1. Date

2. Date 2/26/01

Chem Rec_62

Prep date: 02/19/2001

BNW 57652 p. 27

Preparation of Standardized 0.25M NaOH

WP# F22698 Requester: K. Carson

Request: *I need some more NaOH solution made up and validated by titration. Please make up: 0.25 M NaOH, 250 ml ----Charge to F22698. Thanks! Katharine Carson*

Preparation: Prepare ~ 10M NaOH from reagent grade solid NaOH -- then prepare 500 mL of 0..25M NaOH from this stock and standardize this solution and adjust 10M NaOH accordingly. Then aliquot 250 mL for use and keep 250 for backup.

Calculations : 40g NaOH/mole *10M* .25L = 100g NaOH pellets dissolved to 250 mL with DI water.

$$\frac{500\text{mL} * 0.25\text{M NaOH}}{10\text{ M NaOH}} = 12.5\text{ mL (use 13mL) of ~ 10M NaOH --- to 500 mL with DI water.}$$

Standardization : Use NIST SRM 84j, Potassium Acid Phthalate KHC8H4O4 (KAP) --CMS# 52232

Technique used will be via hand-titration to the phenopthalein endpint. A good titration would use about 20-25 mL of a 50 mL burrette. Hence. since KHC8H404 = 204.23 g/mole or mg/meq

20 mL * 0.25 M NaOH = 5 meq. and 5 meq of KAP = 204.22 mg/meq * 5 = ~ 1021mg (~1g) weighed on 5-place balance

Hence, Target wt for KAP to titrate 0.01M NaOH will be about 1 g.

NaOH Molarity verification performed on ~ 0.25 M NaOH prep.

Verification Test #	(target = 1g) Wt. of KAP	Vol. Of ~ **0.25M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.90624	17.55	0.25284	
2	0.80848	15.60	0.25376	
3	0.88187	17.08	0.25281	
		Ave=	**0.25314**	0.0005
			certified value	

~10M NaOH is revised as follows based on standardized 0.09428M NaOH

$$\frac{500\text{ mL} * 0.25314\text{ M NaOH}}{13\text{ mL}} = \textbf{9.73 M NaOH}$$

Analyst/Date [signature] 2/16/2001

Signed 1. _____ Date _____

2. [signature] Date 2/26/01

Make-up of test solutions for Test H and Test J.

Test H kfl 2/12/01

BNL057652
p. 28

make up 175 ml of 4.75 M $NaNO_3$ + 0.25 M NaOH

166 ml 5 M $NaNO_3$ + 0.1 M NaOH
+ 5.8 ml 4.71 M NaOH
+ 3.2 ml DI water
175 ml

Test J kfl 2/16/01

solution A make up 30 ml of 2.5 M $NaNO_3$ + 2.5 M NaOH

15 ml 5 M $NaNO_3$ + 0.01 M NaOH
+ 15 ml 5 M NaOH
30 ml

solution B make up 30 ml 4 M $NaNO_3$ + 1 M NaOH

24 ml 5 M $NaNO_3$ + 0.01 M NaOH
6 ml 5 M NaOH
30 ml

solution C make up 30 ml 4.9 M $NaNO_3$ + 0.1 M NaOH

29.4 ml 5 M $NaNO_3$ + 0.1 M NaOH
0.6 ml 0.1 M NaOH
30 ml

solution D make up 30 ml 4.9 M $NaNO_3$ + 0.01 M NaOH

29.4 ml 5 M $NaNO_3$ + 0.01 M NaOH
0.6 ml 0.01 M NaOH
30 ml

solution I make up 30 ml 4.5 M $NaNO_3$ + 0.5 M HNO_3

27 ml 5 M $NaNO_3$ + 0.5 M HNO_3
+ 3 ml 0.5 M HNO_3
30 ml

Make-up of test solution for Test C.

Test C 175 ml of 0.005 M $NaNO_3$ + 0.001 M NaOH

17.2 ml 0.01 M NaOH
0.175 ml 5 M $NaNO_3$ + 0.1 M NaOH
157.625 ml DI water
175 ml

KJ Lynn 3/2/01

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Project No. _____ Date of Work _____

Entered By Kathryn J Lynn Date 3/6/01

Disclosed To and Understood By

Signed 1. _____ Date _____

2. _____ Date _____

filename 01-0544
3/12/2001

Battelle, Pacific Northwest National Laboratory
Richland, WA
Radiochemical Processing Group

Client: Carson

Cognizant Scientist: C. Soderquist 3-13-01

Concur: LR Greenwood 3-13-01

Sample	Lab ID	Results of Tc-99 Assay dpm/mL	Ci/mL	g/mL	± 1s
Tc Stock 1	01-0544	1.55E+9	6.97E-4	4.11E-2	±1%
Tc Stock 2	01-0545	1.51E+6	6.80E-7	4.01E-5	±1%
	Spike	1.41E+5 dpm/mL observed			
		1.39E+5 dpm/mL expected			
		101% spike yield			

BAW 57652
p. 30

Signed 1 Date

For Test A

BNWS 7652 p. 31

To make up 300 ml of 1.8 M $NaNO_3$
0.1 M NaOH
3.1 M NaCl

start w/ 5 M $NaNO_3$ + 0.1 M NaOH)

$$(X \text{ ml})(5 \text{ M } NaNO_3) = (300 \text{ ml})(1.8 \text{ M } NaNO_3)$$

$$\frac{5X}{5} = \frac{540}{5}$$

$$X = 108$$

108 ml of 5 M $NaNO_3$ + 0.1 M NaOH

NaCl 58.44 FW 181.164 g/1 liter for 3.1 M
need 54.3492 g into 300 ml

dilute to 300 ml w/ 0.1 M NaOH

Balance 362-06-01-043 weighed 54.3515 g, NaCl
cal. expires 2/2002

4/5/01

Project No. _____ Date of Work _____
Entered By _____ Date 4/8/01
Disclosed To and Understood By _____
Signed 1. _____ Date _____
2. _____ Date _____

BNW 5 7652
p. 32

Make-up of Rhenium Stock Solution, ca. 1.8 mM

Prepare 1:1000 dilution of initial Re stock, prepared 12/18/00, ca. 1.8 M Re.

Balance: 362-06-01-043
Calibration expires: Feb-02

N. Flinn 3/16/01

See BNW 5 7652
p. 2
for initial Re stock make up.

Tare weight	Weight vial & water	Weight vial, water, Re stock
16.7174	26.7077	26.7215

Water, g	Re, g	Dilution factor
9.9903	0.0138	723.9348

Project No. _____ Date of Work _____

Entered By N. Flinn Date 4/3/01

Disclosed To and Understood By _____

Signed 1. _____ Date _____

Chem Rec_64 Prep date: 12/18/2000

BND 57652 p. 33

Preparation of Standardized 5M, 0.1 M, and 0.01M NaOH

WP# W58010 Requester: K. Carson

Request: *I need more NaOH solutions made up and validated by titration. Please make up: 0.01 M NaOH, 2 liters: 0.1 M NaOH, 2 liters: Charge to W58010. Thanks! ---Katharine Carson*

Preparation: Restandardize some previous 0.1M NaOH from current stock and standardize this solution. Then prepare the 0.01M solution and also standardize this 10X dilution.

Calculations : See Chem Rec_60 for prep information on the ~ 0.1M NaOH.

$$\frac{2000\text{mL} * 0.01\text{M NaOH}}{0.1\text{M NaOH}} = 200.0\text{ mL of} \sim 0.1\text{M NaOH} \text{ --- to } 2000\text{ mL with DI water.}$$

Standardization : Use NIST SRM 84j, Potassium Acid Phthalate KHC8H4O4 (KAP) --CMS# 52232

Technique used will be via hand-titration to the phenopthalein endpint. A good titration would use about 20-25 mL of a 50 mL burrette. Hence. since KHC8H404 = 204.23 g/mole or mg/meq

20 mL * 0.1M NaOH = 2 meq. and 2 meq of KAP = 204.22 mg/meq * 2 = ~ 408 mg weigh on 5-place balance

Target wt for KAP to titrate 0.01M NaOH will be about 0.4 g.

NaOH Molarity verification performed on ~ 0.1 M NaOH prep.

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.1M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.40071	19.5	0.10062	
2	0.43252	21.1	0.10037	
3	0.41742	20.35	0.10044	
		Ave=	**0.10047**	0.00013
			certified value	0.13%

NaOH Molarity verification performed on 0.01 M NaOH dilution

Verification Test #	(target = .41g) Wt. of KAP	Vol. Of ~ **0.01M NaOH** to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.06842	33.52	0.00999	
2	0.07756	38.00	0.00999	
3	0.07141	34.94	0.01001	
		Ave=	**0.01000**	0.00001
			certified value	0.08%

Analyst/Date [signature] 3/28/01

Disclosed To and Understood By

Signed-1: [signature] Date 4/3/01

2: Date

3/28/01 [signature]

Balance 362-06-01-043

check wt 100 g = 100.0000 g

make up 100 ml of 4.75 M $NaNO_3$ in 0.25 M NaOH

40.3719 g. $NaNO_3$ - dissolve in 0.2531 M NaOH

3/29/01 [signature]

Balance 362-06-01-026 Cal. expires 8/01

~~Balance 360-06-01-004~~

check wt. 5 kg = 5000.0 kg

$NaNO_3$ 425.0 g, 5 M $NaNO_3$ in 0.1 M NaOH

dissolve and bring to 1 liter volume using

0.1005 M and 0.0943 M NaOH.

Project No. _____ Date of Work _____

Entered By [signature] Date 4/3/01

Disclosed To and Understood By _____

Signed 1. _____ Date _____

BATCH #6 (AN-105 simulant)

Envelope A Recipe	
Volume of Feed	2000 mL

In a tared 2-L Volumetric Flask add

	grams	Actual Wt, grams
Water	200	198.8 g

Tare weight flask + stopper = 438.4 g.
flask, stopper + water = 637.2 g.

Transition Metals and Complexing agents

Compounds	Formula	Mass Needed	Mass Measured	MW	M
Boric Acid	H3BO3	0.292	0.292 g.	61.83	2.36E-3
Cadmium Nitrate	Cd(NO3)2.4H2O	0.009	0.009 g.	308.47	1.47E-5
Calcium Nitrate	Ca(NO3)2.4H2O	0.236	0.236 g.	236.15	4.99E-4
Lead nitrate	Pb(NO3)2	0.085	0.086 g.	331.2	1.28E-4
Magnesium Nitrate	Mg(NO3)2.6H2O	0.057	0.057 g.	256.41	1.11E-4
Potassium Nitrate	KNO3	19.221	19.222 g.	101.1	9.51E-2
Silver Nitrate	AgNO3	0.026	0.026 g.	169.87	7.56E-5
Zinc Nitrate	Zn(NO3)2.6H2O	0.046	0.047 g.	297.47	7.72E-5
Glycolic Acid	HOCH2COOH, 70 wt%	1.665	1.666 g.	76.05	1.09E-2
Sodium Chloride	NaCl	14.984	14.985 g.	58.44	1.28E-1
Sodium Fluoride	NaF	0.420	0.421 g.	41.99	5.00E-3
Sodium Chromate	$Na2CrO4 \cdot 4 H_2O$	6.086 ~~4.205~~	6.088 g.	234.0~~161.970~~	1.30E-2
Sodium Sulfate	Na2SO4	1.140	1.140 g.	142.04	4.01E-3
Potassium Molybdate	K2MoO4	0.204	0.206 g.	238.140	4.27E-4
Ammonium Acetate	CH3COONH4	0.513	0.514 g.	77.080	3.33E-3

In a separate container mix the following

Compounds	Formula	Mass Needed	Mass Measured	FW	M
Sodium Aluminate	NaAlO2	120.61	120.60 g.	81.97	7.36E-1
Sodium Hydroxide	NaOH	137.83	137.84 g	40.00	1.72E+0
Selenium dioxide	SeO2	0.001	0.001 g.	110.960	6.27E-6
Sodium meta-silicate	Na2SiO3.9H2O	2.135	2.135 g	284.200	3.76E-3
Sodium Acetate	NaCH3COO.~~3H2O~~	2.330 ~~3.865~~	2.330 g.	82.0~~136.080~~	1.42E-2
Sodium Formate	HCOONa	4.351	4.354 g	68.010	3.20E-2
Sodium Oxalate	Na2C2O4	0.929	0.929 g.	134.000	3.47E-3
Sodium Phosphate	~~Na3PO4.12H2O~~ $Na_2HPO_4 \cdot 7H_2O$	~~1.608~~ ~~2.281~~ 1.608	1.608 g	268.0~~380.120~~	3.00E-3

Add	grams	Actual Wt, grams
Water	300	301.5 g.

Mix thoroughly. Then add this solution to the volumetric flask.

Add	Formula	Mass Needed	Mass Measured	FW	M
Sodium Carbonate	Na2CO3	22.149	22.15 g	105.990	1.04E-1

Mix thoroughly.

Add	Formula	Mass Needed	Mass Measured	FW	M
Sodium Nitrate	NaNO3	209.70	209.71 g	84.99	1.23E+0
Sodium Nitrite	NaNO2	166.48	168.49 g	69.00	1.21E+0

Mix thoroughly.

Mix thoroughly and dilute to the mark.

Record Final Weight	2489.8	grams
Record Final Density	1.24	gram/mL
Preparation Date	10/31/02	

weight flask, stopper + solution = 2928.2

Signed [signature]

11/1/00 Black ppt. formed in Batch #6 on sitting overnight.

Unique Numerical Designation: TI-PNNL-WTP-040 Author Approval: Brian Ryder 4-4-01
Revision number: 1 Technical Reviewer: David Z. Nash 4/4/01
Effective Date: 04/11/01
Controlling Procedure No: CHG-TP-41500-014

TITLE **Instructions for SuperLig 639 and SuperLig 644 Batch Contacts.**

Work Instructions

General Background: Pre-equilibration of SuperLig 639 and 644 resins

Materials: DI water (ca. 2000 mL)
5 M in $NaNO_3$/0.1 M NaOH solution (ca. 1000 mL)
0.2 micron Nylon filter and filter assembly
0.5 M HNO_3 (ca. 1000 mL)

balance #: _____
last calibration date: _____
calibration exp. Date: _____

stock solution preparation in BNW-57652 pg 12.

Performance test:

test weight	_____	found weight	_____
test weight	_____	found weight	_____
test weight	_____	found weight	_____
test weight	_____	found weight	_____
test weight	_____	found weight	_____

pg 1 & 2 of TI-PNNL-WTP-040 information in Attached pg 1

GB.1 Pre-equilibration of SuperLig 639. Place 15 grams of SuperLig 639 in a 250 mL polypropylene or polyethylene plastic bottle together with 150 mL of a solution 5 M in $NaNO_3$ and 0.1 M in NaOH. Place the bottle on a shaker and agitate for at least 1 hour. Decant off the supernatant and discard the liquid as waste. Repeat three more times for a total of 4 contacts with the alkaline solution. Contact the resin with 150 mL of deionized (DI) water as with the alkaline solution above. Measure the pH of the decanted liquid either with a pH meter or pH paper. Repeat the DI water treatment until the measured pH reads 8 or lower. Transfer the resin with a DI water slurry to a tared 0.2 micron Nylon filter system and pass air through the system until no weight change is observed over the time of at least 1 hour. Transfer the resin to a tared plastic bottle for storage.

SuperLig 639 production lot number: _____
Tare wt. 250 mL bottle: _____
Wt. 250 mL bottle + resin: _____

Initial resin wt: _____
pH of 1st DI water treatment: _____
pH of 2nd DI water treatment: _____
pH of 3rd DI water treatment: _____
pH of final DI water treatment: _____

Prep new batch (3-24) of Superliq 644 & 639

Batch # 010227 CTC-9-23 (Superliq 639).
Batch # 010319 SMC-IV-73 (Superliq 644).

Superliq 639.
START with 21.02 g in a 250mL polypropylene bottle.
Add 200mL 5M $NaNO_3$ / 0.1 M NaOH
START STIRRING 09:50 03-24-01
STOP STIRRING 12:40 03-24-01
Filter through 0.45 μ Nylon filter, transfer back to bottle with 150 mL 0.1 M NaOH / 5 M $NaNO_3$.
START STIRRING 12:50 03-24-01
STOP STIRRING 18:00 03-24-01.
Filter, transfer back with ~150mL 5M $NaNO_3$ / 0.1 M NaOH
START STIRRING 18:40 03-24-01
STOP STIRRING 09:00 03-25-01
Filter, Transfer back to bottle with ~150mL 5M $NaNO_3$ / 0.1 M NaOH
START STIRRING 09:30 03-25-01
STOP STIRRING 13:30 03-25-01
Filter, Transfer back to bottle with ~150 mL H_2O.
START STIRRING 13:45 03-25-01
STOP STIRRING 15:40 03-25-01
Filter, pH of filtrate same as H_2O by pH paper. air dry.

-B. Rapko
03-25-01

GB2b. Determining the F factor for the as-received SuperLig ~~639~~ 644 through drying at ~~85~~ 50°C under vacuum.

Transfer a ca. 0.25 gram sample of the as-received SuperLig 639 to a tared 20 mL glass LSC vial. Weigh the material and record the weight. Cover the open vial with a piece of paper and dry in a vacuum over at ca. 50°C for ca. 24 hours. Remove the sample from the oven, remove the paper and cap, allow the sample and vial to cool to room temperature, and weigh. Repeat until the resin mass changes by less than 5% after 24 hours of drying.

	SL644 F1	SL 644 F2
Tare wt. vial:	17.3187 g	17.2793
Wt. vial + resin:	~~17.4244~~ g	17.3882
Initial resin wt.:	_____ g	_____
Temperature of oven (Heating 1):	~~48~~ °C	48°C
Start Heating 1	16:00 4-19-01	16:00 4-19-01
Stop Heating 1	10:00 4-25-01	12:00 4-25-01
Wt. vial + resin (Heating 1):	17.4125 g	17.3762.
Temperature of oven (Heating 2):	_____ °C	
Start Heating 2	_____	
Stop Heating 2	_____	
Wt. vial + resin (Heating 2):	_____ g	
Temperature of oven (Heating 3):	_____ °C	
Start Heating 3	_____	
Stop Heating 3	_____	
Wt. vial + resin (Heating 3):	_____ g	
Final resin wt.:	_____ g	

not needed for this report BMR 03-07-02

GB2c. Determining the F factor for the pre-equilibrated SuperLig 639 through drying at ~~85~~ 50°C under vacuum

In duplicate, transfer a ca. 0.25 gram sample of the pre-equilibrated SuperLig 639 to a tared 20 mL glass LSC vial. Weigh the material and record the weight. Cover the open vial with a piece of paper and dry in a vacuum over at ca. 85°C for ca. 24 hours. Remove the sample from the oven, remove the paper, cap, allow to vial and sample to cool to room temperature, and weigh. Repeat until the resin mass changes by less than 5% after 24 hours of drying. Dispose of this resin after completion of the experiment.

	F1		F2	
Tare wt. vial:	17.1494 ~~17.3478~~ g		~~17.3463~~ g	17.1947
Wt. vial + resin:	17.3478 ~~17.1494~~ g		~~17.3~~ g	17.3963
Initial resin wt.:	0.1984 g		0.2016 g	

Temperature of oven (Heating 1):	49 °C	49 °C
Start Heating 1	11AM 4-17	11AM 4-17
Stop Heating 1	12:30 4-18	12:30 4-18
Wt. vial + resin (Heating 1):	~~17.2994~~ 17.3017 g	17.3405 g
Temperature of oven (Heating 2):	°C	°C
Start Heating 2	12:40 4-18	12:40 4-18
Stop Heating 2	10:30 4-19	10:30 4-19
Wt. vial + resin (Heating 2):	17.3016 g	17.3452 g
Temperature of oven (Heating 3):	°C	°C
Start Heating 3		
Stop Heating 3		
Wt. vial + resin (Heating 3):	g	g
Final resin wt.:	g	g

note: sample seems to be picking up moisture.

NOT needed as system drying appears to be complete BMR 03-07-02

GB3 Pre-equilibration of SuperLig 644. Sieve SuperLig 644. Collect fraction < 0.425 mm (40 mesh) and greater than 0.212 mm (70 mesh) for pre-equilibration. Place 25 or more grams of SuperLig 644 in a tared 250 mL polypropylene or polyethylene plastic bottle together with ca. 150 mL DI water. Place the bottle on a shaker and agitate for at least 1 hour. Decant off the supernatant and discard the liquid as waste. Carefully add 0.5 M nitric acid to a total volume of 250 mL. Place the bottle on a shaker and agitate for at least 1 hour. Decant off the supernatant and discard the liquid as waste. Repeat the 0.5 M nitric acid contact three more times for a total of 4 contacts with the acid solution. Next contact the resin with 150 mL of deionized (DI) water as with the acid solution. Measure the pH of the decanted liquid either with a pH meter (preferred) or pH paper. Repeat the DI water treatment until the measured pH reads 4 or greater. Transfer the resin with a DI water slurry to a tared 0.2 micron Nylon filter and pass air through the system until less than a 5% weight change is observed over the time of at least 1 hour. Transfer the resin to a tared plastic bottle for storage.

SuperLig 644 production lot number: 90319 SMC-IV-73

Tare wt. 250 mL bottle:	g
Wt. 250 mL bottle + resin:	g
Initial resin wt.:	g
pH of 1st DI water treatment:	
pH of 2nd DI water treatment:	
pH of 3rd DI water treatment:	
pH of final DI water treatment:	

NOT needed for this report B. Lapka 03-07-02

Wt. filter:	g
Wt. filter + resin:	g
Wt. filter + resin:	g
Wt. filter + resin:	g

Tc Distribution Measurements

A) Kinetics of SuperLig 639 with pertechnetate at ambient temperature.

A.1. Prepare sixteen, 20 mL sample vials. Label and tare the sample vials. Record the weights in Table A1 below.

A.2. Determine the density of the test solution (1.8 M sodium nitrate, 0.1 M sodium hydroxide and 3. 1 M in sodium chloride) by weighing three, 1 mL aliquots.

4/20/01 ng/cm pipet check # pipet 114172 set at 1 ml 22.3°C

Temperature: 22.3 °C pipet 114172

Size of aliquot: 1 mL Weight 1 mL aliquot: 1.1810 g

Size of aliquot: ↓ mL Weight 1 mL aliquot: 1.2060 g

Size of aliquot: ↓ mL Weight 1 mL aliquot: 1.1863 g

Ave. density: _____ g/mL (A3a)

0.9701 g
0.9833
0.9648
0.9590
0.9824

A.3. Place ca. 0.100 grams of SuperLig 639 in each vial listed in part A.1. Record the new weight in Table A1 below.

A.4. Place 10.1 mL of the test solution (1.8 M sodium nitrate, 0.1 M sodium hydroxide and 3. 1 M in sodium chloride) into a different set of 20 mL LSC vials using a pipette. Record the weight change in Table A1.

A.5. Place a 35 microliter aliquot from a ca. 0.3 M Tc stock solution into each solution-containing vial.

A.6. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information (t = 0) in table A2.

A.7. Transfer each solution to the corresponding vial described in A1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Seal the cap to the vial with a piece of tape and place the vials in an orbital shaker set to 25°C ± 5°C. Record the vial's temperature. Stir the solutions at ca. 225 rpm for the times indicated in Table A1. Shaker speed: 225

A.8. At the indicated time remove the sample, filter the liquid through a 0.2 micron syringe filter, and place an 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the sample in record the weight in Table A2.

A.9. Count the LSC samples and record this information in Table A.2.

A.10. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table A1. Tc Kinetics Collection Data

Vial Label	Tare wt. (g)	Tare Wt. + SuperLig (g)	Wt. Initial soln. (g)	Stir Time	Start Time	Stop Time	Temp. (°C)
A-1a	17.4360	17.5474	12.2803	2 hr	4-23-01 12:00	4-23-01 14:00	25.2 °C
A-1b	17.3179	17.4112	12.2089	2 hr	↓	↓	↓
A-2a	17.4404	17.5496	12.1978	4 hr	↓	4-23-01 16:00	25.2°C
A-2b	17.2272	17.3293	12.1826	4 hr	↓	↓	↓
A-3a	17.3165	17.4203	12.2003	8 hr	↓	4-24-01 0900	25.1 C
A-3b	17.2158	17.3338	12.2064	8 hr	↓	↓	↓
A-4a	17.2570	17.3635	12.2065	24 hr	↓	4-24-01 16:00	25.1°C
A-4b	17.1788	17.2953	12.1864	24 hr	↓	↓	↓
A-5a	17.1507	17.2571	12.1991	48 hr	↓	4-25-01 11:00	25.1°C
A-5b	17.3024	17.4056	12.2029	48 hr	↓	↓	↓
A-6a	16.9912	17.0815	12.1975	72 hr	↓	4-26-01 12:00	25.1°C
A-6b	17.3851	17.4975	12.2297	72 hr	↓	↓	↓
A-7a	17.3325	17.4328	12.1373	96 hr	↓	4-27-01 10:00	25.1°C
A-7b	16.9815	17.0863	12.0850	96 hr	↓	↓	↓
A-8a	17.1013	17.1934	12.2403	168 hr	↓	4-30-01 10:10	24.7
A-8b	17.2991	17.4162	12.2357	168 hr	↓	↓	↓

(2-300)
cpm

Table A2. Tc kinetics: LSC data

Vial Label	Sample reaction time	Initial wt. (g)*	Initial wt. + aliquot (g)	Activity (cpm)
A-1Ia	0 hr	27.1634	~~27.8~~ 27.2844	499490
A-1Fa	2 hr	27.2108	27.3318	147381
A-1Ib	0 hr	27.2752	27.3958	503536
A-1Fb	2 hr	27.2392	27.3589	159942
A-2Ia	0 hr	27.3033	27.4240	500722
A-2Fa	4 hr	~~27.3042~~	26.8860	115865
A-2Ib	0 hr	27.2205	27.3416	493866
A-2Fb	4 hr	26.7650 ~~27.1680~~ OK 27.1680	27.2889	114190
A-3Ia	0 hr	27.2520	27.3735	502507
A-3Fa	8 hr	27.2188	27.3402	89629.6
A-3Ib	0 hr	27.1993	27.3202	503097
A-3Fb	8 hr	27.1130	27.2331	74549.8
A-4Ia	0 hr	27.2530	27.3737	500484
A-4Fa	24 hr	27.2780	~~27.4002~~	~~497371~~ 86389.5
A-4Ib	0 hr	27.1217	27.2430	~~501083~~ 484104
A-4Fb	24 hr	27.3096	27.4318	71100.4
A-5Ia	0 hr	27.2570	27.3776	499389
A-5Fa	48 hr	27.1385	27.2622	80878.1
A-5Ib	0 hr	27.3134	27.4342	500087
A-5Fb	48 hr	27.2464	27.3698	84159.6
Vial Label	Sample	Initial wt.	Initial wt. +	Activity (cpm)

	reaction time		aliquot	
A-6Ia	0 hr	27.2644	27.3850	497371
A-6Fa	72 hr	27.3118	27.4346	948408
A-6Ib	0 hr	27.2161	27.3368	501083
A-6Fb	72 hr	27.2295	27.3528	958021
A-7Ia	0 hr	27.2340	27.3548	502081
A-7Fa	96 hr	27.2431	27.3656	856910
A-7Ib	0 hr	27.0964	27.2175	501122
A-7Fb	96 hr	27.3147	27.4330	807827
A-8Ia	9 hr	27.1661	27.2865	497580
A-8Fa 55	168 hr	27.3435	~~27.4643~~ ~~27.6040~~	953421
A-8Ib	0 hr	27.1923	27.3115	492360
A-8Fb	168 hr	27.2327	27.3542	956247
Blank a	0 hr			29
Blank b	0 hr			28

*Note: In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

B) Effect of "Spectator Ions" on SuperLig 639 distribution measurements.

B.1. Prepare ten, 20 mL sample vials. Label and tare the sample vials. Record the weights in Table B1 below.

B.2. Place ca. 0.100 grams of SuperLig 639 in each vial listed in B.1. Record the new weight in Table B1 below.

B.3. Place 10.1 mL of the test solution as indicated in Table B1 into a different set of 20 mL LSC vials using a pipette. Record the weight change in Table B1.

B.4. Place a 10 microliter aliquot from a ca. 0.3 M Tc stock solution into each solution-containing vial.

B.5. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information in Table B2.

B.6. Transfer each solution to the corresponding vial described in B1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Seal the cap to the vial with a piece of tape and place the vials in an orbital shaker set to 25°± 5°C. Stir the solutions at ca. 225 rpm. Record the initial and final vial temperatures.

Initial temperature: 26.1 °C 5/14/01 1:58 pm
Final temperature: _____ °C
Shaker speed 225

B.7. After 72 hours, stop shaking and remove the sample vial. Filter the liquid through a 0.2 micron syringe filter, and place a 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record the weight in Table B2.

B.8. Count the LSC samples and record this information in Table B2.

B.9. If needed, perform pipette performance checks on the pipettes used to transfer the 10 microliter stock solutions, the 0.1 mL LSC aliquots and the 10.1 mL test solution transfers. Record the data in attachment 1.

B.10. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table B1. Effect of "Spectator Ions" on SuperLig 639 - Collection Data

Vial Label	Tare wt. (g)	Wt soln. (g)	Tare Wt. + SuperLig (g)	Solution Type
B-1a	17.2660	11.9953	17.3846	A "m"
B-1b	17.2350	11.9364	17.3338	A
B-2a	17.2385	12.0354	17.3379	B
B-2b	17.1979	11.9518	17.3121	B
B-3a	17.2042	12.0177	17.3035	C "p"
B-3b	17.4101	11.8572	17.5141	C
B-4a	17.3472	11.9924	17.4493	D "q"
B-4b	17.3209	11.8812	17.4336	D
B-5a	17.2737	11.7977	17.3895	E "r"
B-5b	17.3219	11.7655	17.4272	E

Table B1 code:

30ml A = 0.018 M $NaNO_3$, 0.1 M NaOH, 4.9 M in NaCl

B = 0.018 M $NaNO_3$, 1 M NaOH, 4 M in NaCl

30ml C = 0.018 M $NaNO_3$, 2.5 M NaOH, 2.5 M in NaCl

30ml D = 0.018 M $NaNO_3$, 4 M NaOH, 1 M in NaCl

30ml E = 0.018 M $NaNO_3$, 4.9 M NaOH, 0.1 M in NaCl

Start Time: 5/14/01 1:58 p.m.

Stop Time: 5/17/01 1:56 p.m.

Initial Temperature: 26.1 °C Final Temperature: 25.6 °C

Table B2. Effect of "Spectator Ions" on SuperLig 639 - LSC data

Vial Label	Initial wt. (g)	Initial wt. + aliquot (g)	Activity (cpm)
B-1Ia	27.4229	27.5402	149947
B-1Fa	27.5199	27.6293	5322.00
B-1Ib	27.4770	27.5939	150203
B-1Fb	27.5105	27.6179	6099.35
B-2Ia	27.6458	27.7576	145432
B-2Fa	27.4108	27.5207	6201.10
B-2Ib	27.4296	27.5436	149738
B-2Fb	27.3347	27.4490	5575.66
B-3Ia	27.6280	27.7417	147377
B-3Fa	27.4099	27.5138	6531.32
B-3Ib	27.4013	27.5140	151208
B-3Fb	27.5352	27.6489	6859.85
B-4Ia	27.3228	27.4342	147334
B-4Fa	27.4997	27.6121	7163.32
B-4Ib	27.5263	27.6425	149980
B-4Fb	27.4064	27.5098	6547.82
B-5Ia	27.3285	27.4430	149153
B-5Fa	27.5124	27.6241	8738.97
B-5Ib	27.4508	27.5663	154107
B-5Fb	27.5072	27.6133	10779.9
Blank a			28.05

Blank b			28.95

*Note: In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

C) Kinetics of SuperLig 639 with Pertechnetate at 65°C Under Stripping Conditions.

C.1. Prepare sixteen, 20 mL sample vials. Label and tare the sample vials. Record the weights in Table C1 below.

C.2. Place ca. 0.100 grams of SuperLig 639 in each vial listed in C.1. Record the new weight in Table C1 below.

C.3. Place 10.1 mL of the test solution (0.005 M sodium nitrate, 0.001 M sodium hydroxide) into a different set of 20 mL LSC vials using a pipette. Record the weight change in Table C1.

5/4 C.4. Place a 10 microliter aliquot from a ca. 0.3 M Tc stock solution into each solution-containing vial.

5/4 C.5. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information in Table C2.

5/7 C.6. Transfer each solution to the corresponding vial described in C1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Bring the shaker table temperature to ca. 65°± 5°C. Place the sample vials on the shaker table and stir the solutions at ca. 225 rpm for the times indicated in Table C1.

Initial temperature: 63.9 °C
Final temperature: 64.8 °C
Shaker speed 225

C.7. At the indicated time remove the sample vial, filter the liquid through a 0.2 micron syringe filter, and place an 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the sample and record the weight in Table C2.

C.8. Count the LSC samples and record this information in Table C.2.

C.9. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table C1. Tc Kinetics Under Stripping Conditions at 65°C Collection Data

Vial Label	Tare wt. (g)	Tare Wt. + SuperLig (g)	Wt. soln. (g)	Stir Time	Start Time	Stop Time	Temp. (°C)
C-1a	17.2292	17.3333	10.1509	2 hr	5/7/01 10:02	5/7/01 12:02	65.5°C
C-1b	17.1905	17.3042	10.0962	2 hr	↓	↓	↓
C-2a	17.2499	17.3620	10.1377	4 hr		5/7/01 14:02	65.5°C
C-2b	17.3067	17.4162	10.1264	4 hr		↓	↓
C-3a	17.1635	17.2591	10.1262	8 hr		5/7/01 16:52	65.6°C
C-3b	17.2525	17.3597	10.1084	8 hr		↓	↓
C-4a	17.1107	17.2117	10.0891	24 hr		5/8/01 10:02	65.5°C
C-4b	17.1346	17.2410	10.0926	24 hr		↓	↓
C-5a	17.3370	17.4317	10.1118	48 hr		5/9/01 10:00	65.6°C
C-5b	17.3086	17.4117	10.0831	48 hr		↓	↓
C-6a	17.2213	17.3133	10.1166	72 hr		5/10/01 10:02	65.3°C
C-6b	17.2302	17.3454	10.1242	72 hr		↓	↓
C-7a	17.1789	17.2862	10.1184	96 hr		5/11/01 10:02	65.3°C
C-7b	17.3604	17.4121	10.1048	96 hr		↓	↓
C-8a	17.2750	17.3776	10.0878	168 hr		5/14/01 10:00	64.8°C
C-8b	17.4438	17.5515	10.1376	168 hr	↓	↓	↓

5/3/2001 [signature]
Balance 362-86-01-043
calibration expires 2/2002
10 g = 10.0000 g.
100 g = 100.0001 g.

Table C2. Tc Kinetics Under Stripping Conditions at 65°C LSC data

Vial Label	Sample reaction time	Initial wt. (g)	Initial wt. + aliquot (g)	Activity (cpm)
C-1Ia	0 hr	27.3224	27.4183	145141
C-1Fa	2 hr	27.2929	27.3872	144426
C-1Ib	0 hr	27.2675	27.3627	145473
C-1Fb	2 hr	27.3951	27.4906	143305
C-2Ia	0 hr	27.3179	27.4136	150520
C-2Fa	4 hr	27.3553	27.4485	149718
C-2Ib	0 hr	27.3755	27.4705	142720
C-2Fb	4 hr	27.1842	27.2757	139194
C-3Ia	0 hr	27.3983	27.4947	151327
C-3Fa	8 hr	27.4085	27.4999	154072
C-3Ib	0 hr	27.4700	27.5659	146469
C-3Fb	8 hr	27.3807	27.4765	149631
C-4Ia	0 hr	27.1794	27.2751	146358
C-4Fa	24 hr	27.3378	27.4317	151182
C-4Ib	0 hr	27.2650	27.3614	145153
C-4Fb	24 hr	27.4152	27.5051	147455
C-5Ia	0 hr	27.2003	27.2961	137996
C-5Fa	48 hr	27.3253	27.4179	149638
C-5Ib	0 hr	27.4145	27.5112	146515
C-5Fb	48 hr	27.4751	27.5653	159808
Vial Label	Sample	Initial wt.	Initial wt. +	Activity (cpm)

		Initial wt.	+ aliquot	Activity (cpm)
C-6Ia	0 hr	27.2259	27.3219	146154
C-6Fa	72 hr	27.3211	27.4094	158802
C-6Ib	0 hr	27.2316	27.3273	141817
C-6Fb	72 hr	27.3167	27.4046	153902
C-7Ia	0 hr	27.3117	27.4074	144244
C-7Fa	96 hr	27.3981	27.4881	166090
C-7Ib	0 hr	27.2185	27.3145	145905
C-7Fb	96 hr	27.2744	27.3621	165623
C-8Ia	9 hr	27.2366	27.3313	144939
C-8Fa	168 hr	27.3837	27.4726	179320
C-8Ib	0 hr	27.3548	27.4513	140558
C-8Fb	168 hr	27.4856	27.5682	165253
Blank a	NA			41.00
Blank b	NA			34.60

*Note: In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

C*) Kinetics of SuperLig 639 with Pertechnetate at 65°C Under Loading Conditions.

C*.1. Prepare sixteen, 20 mL sample vials. Label and tare the sample vials. Record the weights in Table C*1 below.

C*.2. Place ca. 0.100 grams of SuperLig 639 in each vial listed in C.1. Record the new weight in Table C*1 below.

C*.3. Place 10.1 mL of the loading test solution used in part A (1.8 M sodium nitrate, 0.1 M sodium hydroxide and 3. 1 M in sodium chloride) into a different set of 20 mLs LSC vials using a pipette. Record the weight change in table C*1.

5/4 C*.4. Place a 35 microliter aliquot from a ca. 0.3 M Tc stock solution into each solution-containing vial.

5/4 C*.5. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information in Table C*2.

5/7 C*.6. Transfer each solution to the corresponding vial described in C*1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Bring the shaker table temperature to ca. 65°± 5°C. Place the sample vials on the shaker table and stir the solutions at ca. 225 rpm for the times indicated in Table C*1.

Initial temperature:	63.9	°C
Final temperature:	64.8	°C
Shaker speed	225	

C*.7. At the indicated time remove the sample vial, filter the liquid through a 0.2 micron syringe filter, and place an 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the sample and record the weight in Table C*2.

C*.8. Count the LSC samples and record this information in Table C*.2.

C*.9. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table C*1. Tc Kinetics Under Loading Conditions at 65°C Collection Data

Vial Label	Tare wt. (g)	Tare Wt. + SuperLig (g)	Wt soln. (g)	Stir Time	Start Time	Stop Time	Temp. (°C)
C*-1a	17.1229	17.2393	12.2325	2 hr	5/7/01 10:02	5/7/01 12:02	65.5°C
C*-1b	17.2194	17.3240	12.2453	2 hr	↓	↓	↓
C*-2a	17.2403	17.3330	12.1925	4 hr		5/7/01 14:02	65.5°C
C*-2b	17.2134	17.3241	12.2086	4 hr		↓	↓
C*-3a	17.3321	17.4424	12.2500	8 hr		5/7/01 16:52	65.6°C
C*-3b	17.3100	17.4140	12.2042	8 hr		↓	↓
C*-4a	17.3398	17.4455	12.2416	24 hr		5/8/01 10:02	65.5°C
C*-4b	17.2628	17.3626	12.2541	24 hr		↓	↓
C*-5a	17.2298	17.3288	12.2269	48 hr		5/9/01 10:00	65.6°C
C*-5b	17.2064	17.3096	12.2328	48 hr		↓	↓
C*-6a	17.2450	17.3475	12.2300	72 hr		5/10/01 10:02	65.3°C
C*-6b	17.3092	17.4273	12.2095	72 hr		↓	↓
C*-7a	17.2038	17.3041	12.1822	96 hr		5/11/01 10:02	65.3°C
C*-7b	17.0892	17.2038	12.2247	96 hr		↓	↓
C*-8a	17.4544	17.5523	12.2035	168 hr		5/14/01 10:00	64.8°C
C*-8b	17.2220	17.3144	12.2190	168 hr	↓	↓	↓

Table C*2. Tc Kinetics Under Loading Conditions at 65°C - LSC data

Vial Label	Sample reaction time	Initial wt. (g)	Initial wt. + aliquot (g)	Activity (cpm)
C*-1Ia	0 hr	27.3393	27.4581	510125
C*-1Fa	2 hr	27.3875	27.4990	202816
C*-1Ib	0 hr	27.4075	27.5245	502809
C*-1Fb	2 hr	27.2348	27.3460	228812
C*-2Ia	0 hr	27.4153	27.5319	507093
C*-2Fa	4 hr	27.3082	27.4255	243027
C*-2Ib	0 hr	27.4680	27.5849	503582
C*-2Fb	4 hr	27.3327	27.4460	214622
C*-3Ia	0 hr	27.2052	27.3216	501281
C*-3Fa	8 hr	27.4577	27.5721	228370
C*-3Ib	0 hr	27.3993	27.5159	503779
C-3Fb	8 hr	27.4905	27.6033	233795
C*-4Ia	0 hr	27.3899	27.5065	504713
C*-4Fa	24 hr	27.4129	27.5260	227719
C*-4Ib	0 hr	27.3270	27.4423	495733
C*-4Fb	24 hr	27.3729	27.4857	235540
C*-5Ia	0 hr	27.4341	27.5519	506021
C*-5Fa	48 hr	27.2415	27.3543	240810
C*-5Ib	0 hr	27.3652	27.4820	502868
C*-5Fb	48 hr	27.3631	27.4769	232235
Vial Label	Sample	Initial wt.	Initial wt. +	Activity (cpm)

		Initial wt.	+ aliquot	Activity (cpm)
C*-6Ia	0 hr	27.8246	27.9402	499674
C*-6Fa	72 hr	27.3505	27.4698	242092
C*-6Ib	0 hr	27.2915	27.4083	508631
C*-6Fb	72 hr	27.2835	27.3928	213162
C*-7Ia	0 hr	27.3093	27.4259	500407
C*-7Fa	96 hr	27.1855	27.2930	223675
C*-7Ib	0 hr	27.3092	27.4259	503992
C*-7Fb	96 hr	27.2522	27.3488	196174
C*-8Ia	9 hr	27.4607	27.5777	504768
C*-8Fa	168 hr	27.2225	27.3329	236747
C*-8Ib	0 hr	27.4035	27.5207	499669
C*-8Fb	168 hr	27.4353	27.5499	232388
Blank a	NA			41.00
Blank b	NA			34.60

*Note: In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

D) The Effects of Varying Nitrate/Pertechnetate Ratios on SuperLig 639 Distribution Measurements at Room Temperature.

D.1. Prepare twenty, 20 mL sample vials. Label and tare the sample vials. Record the weights in Table D1 below.

D.2. Place ca. 0.100 grams of SuperLig 639 in each vial listed in D.1. Record the new weight in Table D1 below.

D.3. Place 10.1 mL of the test solution as indicated in Table D1 into a different set of 20 mL LSC vials using a pipette. Record the weight change in Table D1.

D.4. Place an aliquot from the appropriate stock solution as indicated in Table D1 into each solution-containing vial.

D.5. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information in Table D2.

D.6. Transfer each solution to the corresponding vial described in D1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Seal the cap to the vial with a piece of tape and place the vials in an orbital shaker set to 25°± 5°C. Stir the solutions at ca. 225 rpm. Record the initial and final vial temperatures.

Initial temperature: 26.1 °C 5/14/01 11:58 pm.
Final temperature: 25.6 °C
Shaker speed 225

D.7. After 72 hours, stop shaking and remove the sample vials, filter the liquid through a 0.2 micron syringe filter, and place an 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record the weight in Table D2.

D.8. Count the LSC samples and record this information in Table D.2.

D.9. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table D1. Nitrate/Pertechnetate Ratio Room Temperature Collection Data

Vial Label	Tare wt. (g)	Tare Wt. + SuperLig (g)	Wt. Soln. (g)	Solution Type	Size of Stock Solution (λ)	Stock Soln. Type
D-1a	17.2380	17.3505	12.0172	A	35	Tc1
D-1b	17.3253	17.4319	11.9808	A	35	Tc1
D-2a	17.2116	17.3174	11.8491	B	35	Tc1
D-2b	17.2220	17.3282	11.8544	B	35	Tc1
D-3a	17.1252	17.2321	12.4006	C	35	Tc1
D-3b	17.2259	17.3233	12.2717	C	35	Tc1
D-4a	17.4083	17.5176	12.2752	C	3.5	Tc1
D-4b	17.1982	17.2997	12.2636	C	3.5	Tc1
D-5a	17.3057	17.4240	12.7628	D	35	Tc1
D-5b	17.3183	17.4136	12.7417	D	35	Tc1
D-6a	17.2636	17.3695	12.7157	D	6	Re1
D-6b	17.1418	17.2484	12.7192	D	6	Re1

Tare wt. + Syparlig wt. Soln

D-7a	17.2798	17.3898	12.7163	D	3.5	Tc1
D-7b	17.1280	17.2295	12.7111	D	3.5	Tc1
D-8a	17.2727	17.3906	12.6988	D	60	Re2
D-8b	17.1354	17.2408	12.7371	D	60	Re2
D-9a	17.2877	17.3928	12.7103	D	2	Re2
D-9b	17.2342	17.3316	12.7274	D	2	Re2
D-10a	17.4166	17.5107	12.2643	C	6	Re1
D-10b	17.1113	17.2134	12.1636	C	6	Re1

Table D1 code:
Tc1 = 0.3 M pertechnetate in DI water
Re1 = 1.8 M perrhenate in DI water
Tc2 = 0.3 mM pertechnetate in DI water
Re2 = 1.8 mM perrhenate in DI water
A = 0.0018 M $NaNO_3$, 0.1 M NaOH, 4.9 M in NaCl ~60 ml
B = 0.18 M $NaNO_3$, 0.1 M NaOH, 4.7 M in NaCl ~75 ml
C = 1.8 M $NaNO_3$, 0.1 M NaOH, 3.1 M in NaCl ~150 ml
D = Envelope A (AN-105) simulant – ~75 ml

Start Time: 5/14/01 1:58 p.m.
Stop Time: 5/17/01 1:56 p.m.
Initial Temperature: 26.1 °C Final Temperature: 25.6 °C

Table D2. Tc Nitrate/Pertechnetate Room Temperature LSC data

Vial Label	Initial wt. (g)	Initial wt. + aliquot (g)	Activity (cpm)
D-1Ia	27.3895	27.5046	503027
D-1Fa	27.5024	27.6158	25021.3
D-1Ib	27.5108	27.6254	502759
D-1Fb	27.3577	27.4671	26061.0
D-2Ia	27.3974	27.5052	491589
D-2Fa	27.4184	27.5327	28998.0
D-2Ib	27.5541	27.6708	512631
D-2Fb	27.4181	27.5280	28389.9
D-3Ia	27.4495	27.5692	501780
D-3Fa	27.4227	27.5398	79242.4
D-3Ib	27.4921	27.6109	501561
D-3Fb	27.4956	27.6064	84599.3
D-4Ia	27.4999	27.6183	52805.3
D-4Fa	27.5698	27.6841	5996.98
D-4Ib	27.4377	27.5552	51615.5
D-4Fb	27.4564	27.5727	6463.51
D-5Ia	27.4655	27.5858	497710
D-5Fa	27.5104	27.6038	43111.1
D-5Ib	27.5574	27.6778	494556
D-5Fb	27.4271	27.5462	64894.7
Vial Label	Initial wt.	Initial wt. +	Activity (cpm)

	Initial wt	Initial wt + aliquot	Activity (cpm)
D-6Ia	6.1383	6.2630	
D-6Fa	6.1367	6.2611	
D-6Ib	6.1207	6.2201	
D-6Fb	6.0523	6.1647	
D-7Ia	27.4579	27.5805	50219.7
D-7Fa	27.4429	27.5628	4151.66
D-7Ib	27.3826	27.5052	52818.0
D-7Fb	27.4920	27.6083	4546.67
D-8Ia	6.1445	6.2690	
D-8Fa	6.1157	6.2326	
D-8Ib	6.1550	6.2795	
D-8Fb	6.1006	6.2198	
D-9Ia	6.0823	6.2053	
D-9Fa	6.0271	6.1468	
D-9Ib	6.1368	6.2595	
D-9Fb	6.1229	6.2454	
D-10Ia	6.1201	6.2404	
D-10Fa	6.0957	6.2162	
D-10Ib	6.0936	6.2099	
D-10Fb	6.1311	6.2498	
Blank a			28.05
Blank b			28.95

1 chem vials

1 chem vials

*Note: In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. In addition, #8, 10, 12 and 13 are to be sampled into an

empty 1 dram vial. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

E) The Effects of Varying Nitrate/Pertechnetate Ratios on SuperLig 639 Distribution Measurements at 65°C.

E.1. Prepare twenty, 20 mL sample vials. Label and tare the sample vials. Record the weights in Table E1 below.

E.2. Place ca. 0.100 grams of SuperLig 639 in each vial listed in E.1. Record the new weight in Table E1 below.

E.3. Place 10.1 mL of the test solution as indicated in Table E1 into a different set of 20 mL LSC vials using a pipette. Record the weight change in Table E1.

E.4. Place an aliquot from the appropriate stock solution as indicated in Table E1 into each solution-containing vial.

E.5. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information in Table E2.

E.6. Transfer each solution to the corresponding vial described in E1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Bring the shaker table temperature to ca. 65°± 5°C. Place the sample vials on the shaker table and stir the solutions at ca. 225 rpm for the times indicated in Table E1.

Initial temperature: 65.2 °C
Final temperature: 65.2 °C
Shaker speed 225

E.7. After 72 hours, stop shaking and remove the sample vials, filter the liquids through a 0.2 micron syringe filter, and place an 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record the weight in Table E2.

E.8. Count the LSC samples and record this information in table E.2.

E.9. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table E1. Nitrate/Pertechnetate Ratio Collection Data at 65°C

Vial Label	Tare wt. (g)	Tare Wt. + SuperLig (g)	Wt Soln. (g)	Solution Type	Size of Stock Solution (λ)	Stock Soln. Type
E-1a	17.2184	17.3365	11.8951	A	35	Tc1
E-1b	17.1355	17.2533	11.9294	A	35	Tc1
E-2a	17.1856	17.2947	11.9713	B	35	Tc1
E-2b	17.2362	17.3322	12.0093	B	35	Tc1
E-3a	17.3050	17.4036	12.2615	C	35	Tc1
E-3b	17.2751	17.3792	12.2242	C	35	Tc1
E-4a	17.4345	17.5250	12.1578	C	3.5	Tc1
E-4b	17.3153	17.4223	12.1841	C	3.5	Tc1
E-5a	17.2659	17.3738	12.5985	D	35	Tc1
E-5b	17.4409	17.5401	12.5526	D	35	Tc1
E-6a	17.1817	17.2849	12.5476	D	6	Re1
E-6b	17.2099	17.3026	12.5323	D	6	Re1

	Tare wt.	+ Syp. Liq	wt. Sdn			
E-7a	17.1497	17.2531	12.3379	D	3.5	Tc1
E-7b	17.4009	17.5049	12.5434	D	3.5	Tc1
E-8a	17.2780	17.3811	12.5746	D	60	Re2
E-8b	17.2511	17.3568	12.5430	D	60	Re2
E-9a	17.1248	17.2315	12.5030	D	2	Re2
E-9b	17.2644	17.3670	12.4787	D	2	Re2
E-10a	17.0773	17.1862	12.1331	C	6	Re1
E-10b	17.2186	17.3375	12.1645	C	6	Re1

Table E1 code:
Tc1 = 0.3 M pertechnetate in DI water
Re1 = 1.8 M perrhenate in DI water
Tc2 = 0.3 mM pertechnetate in DI water
Re2 = 1.8 mM perrhenate in DI water
A = 0.0018 M $NaNO_3$, 0.1 M NaOH, 4.9 M in NaCl
B = 0.18 M $NaNO_3$, 0.1 M NaOH, 4.7 M in NaCl
C = 1.8 M $NaNO_3$, 0.1 M NaOH, 3.1 M in NaCl
D = Envelope A (AN-105) simulant

Start Time: 14:57 6/4/01
Stop Time: 13:00 6/7/01
Initial Temperature: 65.2 °C Final Temperature: 65.2 °C

Table E2. Tc Nitrate/Pertechnetate 65°C LSC data

Vial Label	Initial wt. (g)	Initial wt. + aliquot (g)	Activity (cpm)
E-1Ia	27.4539	27.5710	507273
E-1Fa	27.4629	27.5809	158180
E-1Ib	27.4490	27.5656	507615
E-1Fb	27.4417	27.5597	172124
E-2Ia	27.3763	27.4943	509043
E-2Fa	27.6057	27.7174	165919
E-2Ib	27.4715	27.5892	509424
E-2Fb	27.3994	27.5182	201151
E-3Ia	27.3127	27.4330	510725
E-3Fa	27.4016	27.5206	236794
E-3Ib	27.5277	27.6477	513367
E-3Fb	27.4568	27.5736	240590
E-4Ia	27.4345	27.5542	52936.3
E-4Fa	27.3741	27.4948	24433.1
E-4Ib	27.4641	27.5846	5149.7
E-4Fb	27.2222	27.3402	21330.2
E-5Ia	27.4149	27.5367	496527
E-5Fa	27.3665	27.4863	198892
E-5Ib	27.5403	27.6641	509574
E-5Fb	27.3976	27.5147	211359
Vial Label	Initial wt.	Initial wt. +	Activity (cpm)

Initial wt. +

		aliquot	
E-6Ia	6.3960	6.5196	
E-6Fa	6.5828	6.6706	
E-6Ib	6.4611	6.5748	
E-6Fb	6.4893	6.5944	
E-7Ia	27.3999	27.5209	51657.7
E-7Fa	27.4629	27.5869	19811.3
E-7Ib	27.3317	27.4558	54583.7
E-7Fb	27.4981	27.6236	21107.3
E-8Ia	6.4247	6.5461	
E-8Fa	6.4561	6.5802	
E-8Ib	6.4624	6.5808	
E-8Fb	6.4577	6.5773	
E-9Ia	6.4574	6.5814	
E-9Fa	6.5284	6.6469	
E-9Ib	6.4400	6.5623	
E-9Fb	6.5057	6.6307	
E-10Ia	6.4694	6.5880	
E-10Fa	6.4367	6.5581	
E-10Ib	6.5195	6.6395	
E-10Fb	6.5031	6.6256	
Blank a			29.45, 30.90
Blank b			28.75, 28.95

*Note: In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. In addition, #8, 10, 12 and 13 are to be sampled into an

empty 1 dram vial. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

F) Ionic Strength Effects on SuperLig 639 Distribution measurements.

F.1. Prepare twelve 20 mL sample vials. Label and tare the sample vials. Record the weights in Table F1 below.

F.2. Place ca. 0.100 grams of SuperLig 639 in each vial listed in F.1. Record the new weight in Table F1 below.

F.3. Place 10.1 mL of the test solution as indicated in Table F1 into a different set of 20 mL LSC vials using a pipette. Record the weight change in Table F1.

F.4. Place a 35 microliter aliquot from the ca. 0.3 M Tc stock solution and 6 microliters of the ca. 1.8 M Re stock solution as indicated in Table F1 into each solution-containing vial.

F.5. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information in Table F2.

F.6. Transfer each solution to the corresponding vial described in F1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Seal the cap to the vial with a piece of tape and place the vials in an orbital shaker set to 25°± 5°C. Stir the solutions at ca. 225 rpm. Record the initial and final vial temperatures.

Initial temperature: 25.6 °C
Final temperature: _____ °C
Shaker speed 225

F.7. After 72 hours, stop shaking and remove the sample vials, filter the liquids through a 0.2 micron syringe filter, and place an 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record the weight in Table F2.

F.8. Count the LSC samples and record this information in Table F.2.

F.9. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table F1. Tc Ionic Strength Collection Data

Vial Label	Tare wt. (g)	Tare Wt. SuperLig (g)	Wt. soln. (g)	Solution Type	Stock Soln. Type
F-1a	17.2125	17.3280	12.1328	A	Tc
F-1b	17.2279	17.3262	12.0758	A	Tc
F-2a	17.3403	17.4498	10.6029	B	Tc
F-2b	17.1732	17.2828	10.5955	B	Tc
F-3a	17.3427	17.4408 ~~17.4408~~	10.4129	C	Tc
F-3b	17.3135	17.4109	10.4046	C	Tc
F-4a	17.2435	17.3442	10.2090	D	Tc
F-4b	17.3388	17.4335	10.2002	D	Tc
F-5a	17.2832	17.3936	10.2048	E	Tc
F-5b	17.4365	17.5429	10.1720	E	Tc
F-6a	17.1664	17.2609	10.5297	B	Re
F-6b	17.3093	17.4190	10.5759	B	Re

Table F1 code:
Tc = 0.3 mM pertechnetate in DI water
Re = 1.8 mM perrhenate in DI water
A = 0.018 M $NaNO_3$, 0.01 M NaOH, 5 M in NaCl
need 40 ml → B = 0.018 M $NaNO_3$, 0.01 M NaOH, 1 M in NaCl
C = 0.018 M $NaNO_3$, 0.01 M NaOH, 0.5 M in NaCl
D = 0.018 M $NaNO_3$, 0.01 M NaOH, 0.1 M in NaCl
E = 0.018 M $NaNO_3$, 0.01 M NaOH

Start Time: 13:50 5/21/01
Stop Time: 11:20 5/24/01

Initial Temperature: 25.6 °C Final Temperature: 25.3 °C

Table F2. Tc Ionic Strength LSC data

Vial Label	Initial wt. (g)	Initial wt. + aliquot (g)	Activity (cpm)
F-1Ia	26.9085	27.0184	492748
F-1Fa	27.4058	27.5191	24365.6
F-1Ib	27.4352	27.5497	497275
F-1Fb	27.5308	27.6403	30766.5
F-2Ia	27.4548	27.5479	496736
F-2Fa	27.3052	27.3877	109756
F-2Ib	27.5079	27.6047	493530
F-2Fb	27.5447	27.6347	118832
F-3Ia	27.4788	27.5707	495577
F-3Fa	27.6427	27.7283	175805
F-3Ib	27.4278	27.5225	495442
F-3Fb	27.4562	27.5393	181017
F-4Ia	27.4756	27.5677	494411
F-4Fa	27.5519	27.6367	297945
F-4Ib	27.5221	27.6126	497786
F-4Fb	27.3729	27.4593	298352
F-5Ia	27.5478	27.6422	495787
F-5Fa	27.4994	27.5857	402759
F-5Ib	27.5429	27.6345	497260
F-5Fb	27.3224	27.4024	401537
Vial Label	Initial wt.	Initial wt. +	Activity (cpm)

Initial wt. +

		aliquot	
F-6Ia	6.1303	6.2333	
F-6Fa	6.1235	6.2240	
F-6Ib	6.1095	6.2094	
F-6Fb	6.1989	6.2998	
Blank a			31.15
Blank b			30.25

1 dram vial

*Note: In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. #6 (Re sample) is to be sampled into an empty 1 dram vial. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

G) Tc Loading Isotherm for SuperLig 639.

G.1. Prepare eight, 20 mL sample vials. Label and tare the sample vials. Record the weights in Table G1 below.

G.2. Place ca. 0.100 grams of SuperLig 639 in each vial listed in G.1. Record the new weight in Table G1 below.

G.3. Place 10.1 mL of the test solution as indicated in Table G1 into a different set of vials using a pipette. Record the weight change in Table G1.

G.4. Place an aliquot from the ca. 0.3 M Tc stock solution as indicated in Table G1 into each solution-containing vial.

G.5. Agitate the solutions for 1 minute. Remove a 0.1 mL aliquot and place in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record this information in Table G2.

G.6. Transfer each solution to the corresponding vial described in G1 containing the SuperLig 639. Take care not to disturb the resin so as to splash it on the vial's sides. Seal the cap to the vial with a piece of tape and place the vials in an orbital shaker set to 25 ± 5°C. Stir the solutions at ca. 225 rpm. Record the initial and final vial temperatures.

Initial temperature: 25.6 °C
Final temperature: _____ °C
Shaker speed 225

G.7. After 72 hours, stop shaking and remove the sample vials, filter the liquids through a 0.2 micron syringe filter, and place a 0.1 mL aliquot in a labeled 20 mL liquid scintillation vial containing 10 mL of Ultima Gold XR. Weigh the samples and record the weight in Table G2.

G.8. Count the LSC samples and record this information in Table G2.

G.9. Decant and discard the remaining liquids into a radioactive waste container and the resin into a solid "SuperLig 639" waste container containing DI water.

Table G1. Tc Loading Isotherm for SuperLig 639 Collection Data

Vial Label	Tare wt. (g)	Wt Soln. (g)	Aliquot size	~~Wt. + soln. + aliquot (g)~~ Tare wt. + Syringe	Solution Type
G-1a	17.2538	12.3615	1000 µL	17.3498	A
G-1b	17.2928	12.4055	1000 µL	17.3977	A
G-2a	17.2827	12.3418	500 µL	17.3905	B
G-2b	17.3606	12.2108	500 µL	17.4687	B
G-3a	17.1552	12.2160	100 µL	17.2506	C
G-3b	17.1193	12.1177	100 µL	17.2268	C
G-4a	17.2954	12.1879	10 µL	17.3983	D
G-4b	17.2539	12.1215	10 µL	17.3529	D

Table D1 code:

✓A = 1.8 M $NaNO_3$, 0.1 M NaOH, 3.1 M in NaCl
✓B = 0.9 M $NaNO_3$, 0.1 M NaOH, 4 M in NaCl
✓C = 0.18 M $NaNO_3$, 0.1 M NaOH, 4.7 M in NaCl
✓D = 0.018 M $NaNO_3$, 0.1 M NaOH, 4.9 M in NaCl

~~bottle #'s 6-0007 #~~

Start Time: 13:50 5/21/01
Stop Time: 11:20 5/24/01

Initial Temperature: 25.6°C °C Final Temperature: 25.3 °C

Table G2. Tc Loading Isotherm for SuperLig 639 LSC data

(Note: dilute all G1 samples by a factor of 40 by weight before counting; dilute all G2 samples by a factor of 20 before counting; and dilute all G3 samples by a factor of 4 before counting) all dilutions used 100 µl aliquot.

Vial Label	Initial wt. (g)	Initial wt. + aliquot (g)	Activity (cpm)	(DI water) wt. diluent (g.)
G-1Ia	27.5366	27.6302	325790	3.9899
G-1Fa	27.3873	27.4745	264642	3.9420
G-1Ib	27.4486	27.5359	322458	3.9810
G-1Fb	27.4605	27.5441	267283	3.8191
G-2Ia	27.4423	27.5335	331649	1.9712
G-2Fa	27.4234	27.5058	346647	1.9665
G-2Ib	27.3699	27.4614	334645	1.9776
G-2Fb	27.4017	27.4777	184899	1.9886
G-3Ia	27.5199	27.6140	406520	0.4010
G-3Fa	27.4669	27.5583	46633.7	0.3969
G-3Ib	27.5371	27.6350	279794	0.4048
G-3Fb	27.4373	27.5260	33007.2	0.4016
G-4Ia	27.4164	27.5281	144412	
G-4Fa	27.4135	27.5144	6367.40	
G-4Ib	27.3811	27.4937	143245	
G-4Fb	27.5714	27.6664	6361.09	
Blank a			35.95, 31.15	
Blank b			41.60, 30.25	

40X

20X

4X

In the table above the initial wt. refers to the weight of the 20 mL scintillation vial plus 10 mL Ultima Gold. Blanks refer to simple Ultima Gold in the absence of a radioactive aliquot.

Appendix C

Analytical Data

Battelle, Pacific Northwest National Laboratory
Richland, WA
Radiochemical Processing Group

filename 01-0544
3/12/2001

Client: Carson

Cognizant Scientist: C. Soderquist 3-13-01

Concur: LR Greenwood 3-13-01

Sample	Lab ID	Results of Tc-99 Assay dpm/mL	Ci/mL	g/mL	± 1s
Tc Stock 1	01-0544	1.55E+9	6.97E-4	4.11E-2	±1%
Tc Stock 2	01-0545	1.51E+6	6.80E-7	4.01E-5	±1%
	Spike	1.41E+5 dpm/mL observed			
		1.39E+5 dpm/mL expected			
		101% spike yield			

Battelle – PNNL / AIAL
Inorganic Analysis / 320-ICP/MS Data Report

Project / WP#: 42365 / W58010
ASR#: 6134
Client: K.J. Carson / B. Rapko
Total Samples: 42

RPL #	Client ID
01-1412 to 01-1447	See below

Procedure: PNL-SC-01 Rev. 1, *Inductively Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number: WB36913 ICP/MS, VG Elemental
512-06-01-014 Mettler AJ100 Balance

Analyst: Orville Thomas Farmer III

Report Written by: Orville Thomas Farmer III

Analysis Date(s): 8/19/01

Analysis Files: Experiments – 19Aug01S
Procedures – Manual data collection
Element Menus – 19Aug01S

Laboratory Record Book (LRB): LRB BNW 56465: Sample login: p.47-48
LRB BNW 56465: Rhenium assay: p.47-48

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Reviewed By 8-25-01

Concur 25Aug01

1. Analysis

Forty-two liquid samples for Rhenium analysis were received from the client and were analyzed by ICP/MS. All samples were first diluted by a factor of 100,000 and screened to determine an estimate of the Re concentration. After screening, the samples were dilution to yield a known Re solution concentration and bracketed by the calibration standards accordingly.

Data was collected using peak-hopping data acquisition parameters and manual solution sampling into the instrument. All solutions (Standards, Instrument QC's, Preparation QC's and Samples) were spiked with thallium that was used as the internal standard element.

ICP/MS results for all solutions are reported in units of **mg/L** and are instrument blank corrected. In Table 1, reported concentrations for instrument QC's are reported back to the instrument solutions only. In Table 2, final results have been corrected for all laboratory dilutions performed on the samples during analysis.

Table 1: Instrument Quality Control

File Name	Criteria	EQL	Expected (mg/L)	Found (mg/L)	% Rec
ICB	< EQL	1.1E-06		2.14E-08	
ICB [a]	< EQL	1.1E-06		1.41E-08	
CCB-1	< EQL	1.1E-06		4.53E-08	
CCB-2	< EQL	1.1E-06		2.83E-08	
CCB-1 [a]	< EQL	1.1E-06		3.10E-08	
CCB-2 [a]	< EQL	1.1E-06		6.78E-08	
CCB-3 [a]	< EQL	1.1E-06		1.21E-07	
ICV	(+ / -) 10 %		1.67E-04	1.68E-04	101
ICV [a]	(+ / -) 10 %		1.67E-04	1.68E-04	101
CCV-1	(+ / -) 10 %		1.67E-04	1.68E-04	101
CCV-2	(+ / -) 10 %		1.67E-04	1.70E-04	102
CCV-1 [a]	(+ / -) 10 %		1.67E-04	1.70E-04	102
CCV-2 [a]	(+ / -) 10 %		1.67E-04	1.69E-04	101
CCV-3 [a]	(+ / -) 10 %		1.67E-04	1.69E-04	
ICS				8.16E-04	
ICS Dup.	(+ / -) 10 %			8.14E-04	100
ICS Dup. Spike	(+ / -) 25 %		1.67E-04	1.71E-04	102
ICS [a]				8.12E-04	
ICS Dup. [a]	(+ / -) 10 %			8.12E-04	100
ICS Dup. Spike [a]	(+ / -) 25 %		1.67E-04	1.69E-04	101

[a] Second run was preformed on the samples due to nebulizer plugging. Samples between CCV-2 and CCV-3 in the first analytical run were re-analyzed in the second analytical run.

QC Summary:

Duplicate (DUP): No duplicate was submitted. However, a replicate instrument QC analysis (ICS and ICS Dup.) was performed on sample (D-6Fb) and met the QC criteria of ±20%.

Matrix Spike (MS): A post matrix spike (E-6Fa) was submitted, however no information as too the spiking level was provided, therefore no recovery was calculated.

Post Spike (PS): No post spike was submitted. However, an instrument post spike analysis (ICS Dup. Spike) was performed on sample (D-6Fb) and met the QC criteria of ±25%.

Preparation Blank (PB) and Laboratory Control Standard (LCS/BS): A preparation blank was submitted and analyzed and meet the criteria of being < 5% of the measured concentration in the samples but was greater than the calculated EQL. A LCS/BS was submitted and analyzed, however no information as too the spiking level in this QC was provided and no recover was calculated.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB): The ICB/CCB standards are 1-% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than the estimated quantitation limit (< EQL) were met for all ICBs and CCBs.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): The ICV/CCV standards met the QC criteria of ±10% were met for all ICV and CCV check solutions.

Internal Standard (ISTD): The ISTDs met the QC criteria of 30% to 120%.

Table 2: Sample results

ACL Sample ID	Client ID	Vial ID	Dilution Factor	Re (mg/L)	(+ / -) 1 sigma
01-1412	D-6Ia	A	2069	1.76E+00	1.16E-03
01-1413	D-6Fa	B	500	4.32E-01	2.81E-04
01-1414	D-6Ib	C	1714	1.48E+00	9.64E-04
01-1415	D-6Fb	D	484	3.93E-01	2.73E-04
DUP 01-1415	D-6Fb	D	484	3.93E-01	2.72E-04
DUP Spike 01-1415	D-6Fb	D	484	4.75E-01	2.72E-04
01-1416	D-8Ia	E	120	1.76E-02	6.74E-05
01-1417	D-8Fa	F	120	3.25E-03	6.74E-05
01-1418	D-8Ib	G	120	1.89E-02	6.74E-05
01-1419	D-8Fb	H	120	3.63E-03	3.58E-05
01-1420	D-9Ia	I	120	5.79E-04	4.01E-06
01-1421	D-9Fa	J	120	1.20E-04	2.62E-06
01-1422	D-9Ib	K	120	5.93E-04	7.98E-06
01-1423	D-9Fb	L	120	1.32E-04	1.45E-06
01-1424	D-10Ia	M	2069	1.78E+00	1.16E-03
01-1425	D-10Fa	N	750	6.29E-01	4.21E-04
01-1426	D-10Ib	O	2069	1.68E+00	1.16E-03
01-1427	D-10Fb	P	674	5.68E-01	3.80E-04
01-1428	E-6Ia	Q	2143	1.81E+00	1.21E-03
01-1429	E-6Fa	R	923	7.68E-01	5.19E-04
Reagent Blank Spike	DI-H2O spike	S	120	2.78E-03	7.24E-06
Process Water	DI-H2O	T	120	6.98E-06	1.39E-06
Post MS-1429	E-6Fa	U	414	3.89E-01	2.79E-04
01-1430	E-6Ib	A-1	2069	1.74E+00	1.17E-03
01-1431	E-6Fb	B-1	1176	9.55E-01	6.63E-04
01-1432	E-8Ia	C-1	120	1.86E-02	6.74E-05
01-1433	E-8Fa	D-1	120	1.14E-02	6.74E-05
01-1434	E-8Ib	E-1	120	1.97E-02	6.74E-05
01-1435	E-8Fb	F-1	120	1.11E-02	6.74E-05
01-1436	E-9Ia	G-1	120	6.77E-04	1.26E-05
01-1437	E-9Fa	H-1	120	3.69E-04	1.20E-06
01-1438	E-9Ib	I-1	120	6.63E-04	5.63E-06
01-1439	E-8Fb	J-1	120	4.04E-04	7.25E-06
01-1440	E-10Ia	K-1	2143	1.80E+00	1.21E-03
01-1441	E-10Fa	L-1	1304	1.09E+00	7.34E-04
01-1442	E-10Ib	M-1	2143	1.82E+00	1.21E-03
01-1443	E-10Fb	N-1	1429	1.19E+00	8.03E-04
01-1444	F-6Ia	O-1	120	7.54E-04	7.85E-06
01-1445	F-6Fa	P-1	779	6.44E-01	4.38E-04
01-1446	F-6Ib	Q-1	120	1.04E-02	6.74E-05
01-1447	F-6Fb	R-1	822	6.87E-01	4.62E-04
Reagent Blank Spike	DI-H2O spike	S-1	120	2.68E-03	1.80E-06
Process Water	DI-H2O	T-1	120	5.24E-06	1.04E-06
Post MS-1447	F-6Fb	U-1	353	3.06E-01	2.80E-04

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

"New" SuperLig 639
RT Kinetics w/ shaker/heater
B. Rapko 4-25-01
shaker speed ≈ 225 rpm

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	29.70	8.21	28.88	8.32	8.80	15.08	94.998	512.78	Blank a
2	20.00	500460	0.06	499490	0.06	1183.30	1.30	144.80	441.78	A 1aI
3	20.00	144619	0.12	144381	0.12	266.89	2.74	142.79	439.22	A 1a F
4	20.00	504539	0.06	503536	0.06	1134.38	1.33	144.62	445.53	A 1 B I
5	20.00	160203	0.11	159942	0.11	279.97	2.67	142.53	442.04	A 1 B F
6	20.00	501728	0.06	500722	0.06	1022.99	1.40	144.50	448.85	A2A I
7	20.00	116056	0.13	115865	0.13	210.71	3.08	140.62	432.18	A2A F
8	20.00	494845	0.06	493866	0.06	961.64	1.44	144.55	450.18	A2B I
9	20.00	114369	0.13	114190	0.13	226.07	2.97	142.22	436.71	A2B F
10	20.00	503501	0.06	502507	0.06	1010.00	1.41	144.43	448.70	A 3a I
11	20.00	89772.4	0.15	89629.6	0.15	172.05	3.41	141.27	435.32	A3a F
1 MISSING TUBE(S)										
13	20.00	504104	0.06	503097	0.06	1046.73	1.38	144.33	448.14	A3b I
14	20.00	74671.0	0.16	74549.8	0.16	149.03	3.66	141.18	434.23	A3b F
15	20.00	501473	0.06	500484	0.06	1015.09	1.40	144.12	448.10	A4a I
16	20.00	485071	0.06	484104	0.06	905.65	1.49	144.26	451.13	A4b I
17	20.00	500364	0.06	499389	0.06	1114.66	1.34	144.30	445.03	A5a I
18	20.00	501075	0.06	500087	0.06	1052.91	1.38	143.99	445.97	A5b I
19	20.00	498352	0.06	497371	0.06	1017.16	1.40	144.14	447.35	A6a I
20	20.00	502061	0.06	501083	0.06	1015.06	1.40	144.56	448.57	A6b I
21	20.00	503061	0.06	502081	0.06	1136.31	1.33	144.25	444.31	A7a I
22	20.00	502123	0.06	501122	0.06	1008.55	1.41	144.28	449.41	A7b I
23	20.00	498552	0.06	497580	0.06	1018.96	1.40	144.56	448.66	A8a I
24	20.00	493326	0.06	492360	0.06	1021.21	1.40	144.71	448.77	A8b I
25	20.00	79823.0	0.16	79693.2	0.16	156.45	3.58	147.46	451.28	A7Fa SS
26	20.00	82228.9	0.16	82093.2	0.16	166.99	3.46	148.08	451.58	A7Fb SS
27	20.00	28.85	8.33	27.83	8.48	9.40	14.59	94.494	520.09	Blank b

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	41.00	6.98	39.86	7.08	8.45	15.38	75.655	444.16	Blank
2	20.00	86622.8	0.15	86489.5	0.15	196.68	3.19	142.81	433.60	A4AF
3	20.00	71209.8	0.17	71100.4	0.17	166.89	3.46	142.55	431.39	A4BF
4	20.00	80998.6	0.16	80878.1	0.16	181.60	3.32	142.05	432.22	A5AF
5	20.00	84285.9	0.15	84159.6	0.15	201.44	3.15	141.99	431.93	A5BF
6	20.00	94985.3	0.15	94840.8	0.15	211.36	3.08	144.58	439.58	A6AF
7	20.00	75616.8	0.16	75502.1	0.16	174.61	3.38	145.53	439.15	A6BF
8	20.00	85820.2	0.15	85691.0	0.15	192.01	3.23	145.22	439.44	A7AF
9	20.00	80902.9	0.16	80782.7	0.16	164.19	3.49	146.03	443.02	A7BF
10	20.00	92527.0	0.15	92391.1	0.15	225.85	2.98	142.81	433.51	A8AFSS
11	20.00	70426.5	0.17	70320.3	0.17	164.74	3.48	143.07	434.97	A8BFSS
12	20.00	95491.4	0.14	95342.1	0.14	214.67	3.05	149.13	450.36	A8AF
13	20.00	95773.9	0.14	95624.7	0.14	250.75	2.82	149.97	450.54	A8BF
14	20.00	145141	0.12	144921	0.12	383.64	2.28	147.52	444.68	C1IA
15	20.00	145473	0.12	145243	0.12	371.62	2.32	147.67	448.23	C1IB
16	20.00	150520	0.12	150291	0.12	409.43	2.21	148.28	447.67	C2IA
17	20.00	142720	0.12	142499	0.12	362.81	2.35	148.42	449.10	C2IB
18	20.00	151327	0.11	151092	0.12	389.07	2.27	148.01	449.20	C3IA
19	20.00	146469	0.12	146248	0.12	392.07	2.26	148.55	448.57	C3IB
20	20.00	146358	0.12	146129	0.12	386.46	2.27	148.36	447.43	C4IA
21	20.00	145153	0.12	144936	0.12	380.30	2.29	148.51	448.34	C4IB
22	20.00	137996	0.12	137789	0.12	385.81	2.28	148.49	446.61	C5IA
23	20.00	146515	0.12	146291	0.12	407.06	2.22	148.88	447.23	C5IB
24	20.00	146154	0.12	145930	0.12	365.37	2.34	148.03	450.20	C6IA
25	20.00	141817	0.12	141606	0.12	381.03	2.29	148.10	448.32	C6IB
26	20.00	144244	0.12	144021	0.12	395.57	2.25	148.64	448.84	C7IA
27	20.00	145905	0.12	145677	0.12	381.39	2.29	148.65	449.83	C7IB
28	20.00	144939	0.12	144727	0.12	416.23	2.19	148.53	445.98	C8IA
29	20.00	140558	0.12	140347	0.12	381.65	2.29	149.36	450.30	C8IB
30	20.00	510125	0.06	509130	0.06	1435.84	1.18	149.80	454.80	C°1IA
31	20.00	502809	0.06	501812	0.06	1299.63	1.24	150.04	458.02	C°1IB
32	20.00	507093	0.06	506097	0.06	1360.09	1.21	150.02	457.52	C°2IA
33	20.00	503582	0.06	502586	0.06	1331.48	1.23	149.70	458.42	C°2IB
34	20.00	501281	0.06	500302	0.06	1319.06	1.23	149.57	457.16	C°3IA
35	20.00	503779	0.06	502794	0.06	1323.91	1.23	149.58	457.02	C°3IB
36	20.00	504713	0.06	503713	0.06	1310.68	1.24	149.13	456.52	C°4IA
37	20.00	495733	0.06	494771	0.06	1380.93	1.20	148.98	453.39	C°4IB
38	20.00	506021	0.06	505031	0.06	1295.82	1.24	148.39	454.19	C°5IA

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
39	20.00	502868	0.06	501865	0.06	1327.62	1.23	149.39	456.10	C° 5IB
40	20.00	499674	0.06	498717	0.06	1408.04	1.19	148.86	451.70	C° 6IA
41	20.00	508631	0.06	507645	0.06	1331.40	1.23	148.83	453.94	C° 6IB
42	20.00	500407	0.06	499434	0.06	1316.98	1.23	148.43	453.37	C° 7IA
43	20.00	503992	0.06	502983	0.06	1267.81	1.26	148.54	455.35	C° 7IB
44	20.00	504768	0.06	503783	0.06	1315.83	1.23	148.73	454.23	C° 8IA
45	20.00	499669	0.06	498695	0.06	1276.63	1.25	148.62	454.70	C° 8IB
46	20.00	34.60	7.60	33.90	7.68	8.85	15.03	81.979	449.27	Blank b

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	48.90	6.40	48.26	6.44	10.80	13.61	97.622	444.38	-BlKa
2	20.00	144426	0.12	144199	0.12	375.54	2.31	146.49	442.95	-C1Fa
3	20.00	143305	0.12	143083	0.12	360.47	2.36	146.37	443.87	-C1Fb
4	20.00	149718	0.12	149481	0.12	393.61	2.25	146.55	443.16	-C2Fa
5	20.00	139194	0.12	138979	0.12	373.29	2.31	146.59	442.15	-C2Fb
6	20.00	154072	0.11	153831	0.11	418.62	2.19	146.39	441.12	-C3Fa
7	20.00	149631	0.12	149400	0.12	357.00	2.37	146.16	445.23	-C3Fb
8	20.00	151182	0.12	150954	0.12	422.00	2.18	146.19	439.07	-C4Fa
9	20.00	147455	0.12	147228	0.12	378.87	2.30	146.31	440.98	-C4Fb
10	20.00	202816	0.10	202499	0.10	511.72	1.98	146.20	443.26	-C*1Fa
11	20.00	228812	0.09	228445	0.09	589.27	1.84	146.33	443.63	-C*1Fb
12	20.00	243027	0.09	242635	0.09	588.22	1.84	146.35	446.51	-C*2Fa
13	20.00	214622	0.10	214286	0.10	561.58	1.89	146.43	443.02	-C*2Fb
14	20.00	228370	0.09	228018	0.09	597.81	1.83	146.18	442.91	-C*3Fa
15	20.00	233795	0.09	233417	0.09	582.07	1.85	146.42	444.96	-C*3Fb
16	20.00	227719	0.09	227362	0.09	572.42	1.87	145.51	442.27	-C*4Fa
17	20.00	235540	0.09	235172	0.09	610.47	1.81	146.14	442.80	-C*4Fb
18	20.00	49.25	6.37	48.60	6.42	10.60	13.74	106.14	448.61	-BlKb

K. Thomas 5/9/01

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	47.80	6.47	46.66	6.55	9.65	14.40	86.347	444.44	BIK 9
2	20.00	149638	0.12	149401	0.12	370.26	2.32	144.72	436.48	C 5 Fa
3	20.00	159808	0.11	159561	0.11	386.90	2.27	144.44	437.24	C 5 Fb
4	20.00	240810	0.09	240408	0.09	577.28	1.86	143.40	436.09	C * 5 Fa
5	20.00	232235	0.09	231863	0.09	610.78	1.81	143.62	433.09	C * 5 Fb
6	20.00	49.70	6.34	48.76	6.40	8.95	14.95	93.537	444.47	BIK 5

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	62.67	5.65	60.87	5.73	11.58	13.14	121.78	515.75	Bkg
2	20.00	158802	0.11	158558	0.11	393.81	2.25	145.28	438.46	C 6Fg
3	20.00	153902	0.11	153662	0.11	405.27	2.22	144.46	435.22	C 6Fg
4	20.00	242092	0.09	241705	0.09	595.52	1.83	142.72	432.68	C* 6Fg
5	20.00	213162	0.10	212823	0.10	522.64	1.96	143.26	434.25	C* 6Fg
6	20.00	65.00	5.55	63.21	5.63	13.15	12.33	116.39	518.11	Bkg

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	33.75	7.70	32.11	7.89	6.80	17.15	68.990	446.24	BK9
2	20.00	166090	0.11	165827	0.11	445.13	2.12	145.86	438.92	C7F9
3	20.00	165623	0.11	165364	0.11	440.80	2.13	146.19	441.67	C7F6
4	20.00	223675	0.09	223324	0.09	571.05	1.87	145.36	439.82	C*7F9
5	20.00	196174	0.10	195869	0.10	518.67	1.96	145.70	439.37	C*7F5
6	20.00	29.60	8.22	28.86	8.33	8.25	15.57	72.664	445.72	BK 5

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	28.05	8.44	27.61	8.51	7.75	16.06	70.064	444.41	B1K4
2	20.00	179320	0.11	179031	0.11	476.37	2.05	147.02	443.94	C8F4
3	20.00	165253	0.11	164993	0.11	443.62	2.12	147.06	442.73	C8F6
4	20.00	236747	0.09	236369	0.09	583.71	1.85	147.31	445.07	C8F4
5	20.00	232388	0.09	232018	0.09	568.81	1.88	147.33	445.15	C8F6
6	20.00	149947	0.12	149725	0.12	334.03	2.45	146.90	443.54	B1I4
7	20.00	150203	0.12	149974	0.12	334.37	2.45	147.04	444.46	B1I6
8	20.00	145432	0.12	145210	0.12	313.57	2.53	146.46	444.75	B2I4
9	20.00	149738	0.12	149511	0.12	326.15	2.48	146.96	445.32	B2I6
10	20.00	147377	0.12	147152	0.12	322.08	2.49	147.65	446.40	B3I4
11	20.00	151208	0.12	150976	0.12	360.91	2.35	147.11	443.05	B3I6
12	20.00	147334	0.12	147106	0.12	349.38	2.39	147.88	446.14	B4I4
13	20.00	149980	0.12	149755	0.12	356.05	2.37	147.63	443.78	B4I6
14	20.00	149153	0.12	148926	0.12	346.32	2.40	147.88	446.27	B5I4
15	20.00	154107	0.11	153869	0.11	358.67	2.36	147.69	444.77	B5I6
16	20.00	503027	0.06	502057	0.06	1235.84	1.27	148.35	451.04	D1I4
17	20.00	502759	0.06	501780	0.06	1265.53	1.26	149.29	452.94	D1I6
18	20.00	491589	0.06	490643	0.06	1184.34	1.30	149.20	454.62	D2I4
19	20.00	512631	0.06	511623	0.06	1271.67	1.25	149.64	453.87	D2I6
20	20.00	501780	0.06	500817	0.06	1161.55	1.31	147.84	450.61	D3I4
21	20.00	501561	0.06	500611	0.06	1234.58	1.27	148.10	450.19	D3I6
22	20.00	52805.3	0.19	52725.3	0.19	106.74	4.33	145.33	440.80	D4I4
23	20.00	51615.5	0.20	51537.4	0.20	108.41	4.30	145.31	440.66	D4I6
24	20.00	497710	0.06	496893	0.06	568.48	1.88	106.93	320.85	D5I4
25	20.00	494556	0.06	493732	0.06	560.31	1.89	106.73	319.96	D5I6
26	20.00	50219.7	0.20	50159.2	0.20	41.31	6.96	103.83	310.80	D7I4
27	20.00	52818.0	0.19	52757.9	0.19	45.89	6.60	104.48	312.20	D7I6
28	20.00	28.95	8.31	28.45	8.38	8.85	15.03	74.677	449.00	B1K6

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	46.90	6.53	45.91	6.60	10.05	14.11	97.606	446.63	Blk a
2	20.00	5322.00	0.61	5312.94	0.61	20.06	9.99	145.34	440.10	B 1 Fa
3	20.00	6099.35	0.57	6089.02	0.57	21.45	9.66	145.74	440.88	B 1 Fb
4	20.00	6201.10	0.57	6190.90	0.57	23.60	9.21	145.88	441.20	B 2 Fa
5	20.00	5575.66	0.60	5567.24	0.60	21.49	9.65	146.73	442.13	B 2 Fb
6	20.00	6531.52	0.55	6519.74	0.55	22.88	9.35	147.16	445.32	B 3 Fa
7	20.00	6859.85	0.54	6850.24	0.54	23.50	9.23	147.49	442.76	B 3 Fb
8	20.00	7163.32	0.53	7151.14	0.53	24.18	9.09	147.01	445.65	B 4 Fa
9	20.00	6547.82	0.55	6536.60	0.55	24.58	9.02	147.58	443.63	B 4 Fb
10	20.00	8738.97	0.48	8723.93	0.48	26.23	8.73	147.47	446.03	B 5 Fa
11	20.00	10779.9	0.43	10763.8	0.43	30.27	8.13	147.97	445.17	B 5 Fb
12	20.00	25021.3	0.28	24986.3	0.28	60.48	5.75	146.65	441.31	D 1 Fa
13	20.00	26061.0	0.28	26019.9	0.28	59.39	5.80	146.27	442.18	D 1 Fb
14	20.00	28998.0	0.26	28952.7	0.26	70.42	5.33	146.57	442.21	D 2 Fa
15	20.00	28389.9	0.27	28345.8	0.27	70.44	5.33	146.20	437.83	D 2 Fb
16	20.00	79242.4	0.16	79121.7	0.16	192.62	3.22	146.35	439.02	D 3 Fa
17	20.00	84599.3	0.15	84471.8	0.15	186.42	3.28	146.74	443.30	D 3 Fb
18	20.00	5996.98	0.58	5987.58	0.58	22.82	9.36	145.50	440.57	D 4 Fa
19	20.00	6463.51	0.56	6454.29	0.56	22.74	9.38	143.44	436.67	D 4 Fb
20	20.00	43111.1	0.22	43050.8	0.22	56.56	5.95	116.10	346.45	D 5 Fa
21	20.00	64894.7	0.18	64812.0	0.18	68.89	5.39	110.60	327.19	D 5 Fb
22	20.00	4151.66	0.69	4144.21	0.69	13.94	11.98	108.77	324.67	D 7 Fa
23	20.00	4546.67	0.66	4538.48	0.66	14.24	11.85	108.33	321.77	D 7 Fb
24	20.00	41.90	6.91	41.10	6.98	8.80	15.08	94.431	446.64	Blk b

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	35.95	7.46	35.50	7.51	8.50	15.34	95.092	443.82	BLK
2	20.00	492748	0.06	491813	0.06	1241.42	1.27	149.45	450.18	F1I
3	20.00	497275	0.06	496309	0.06	1268.00	1.26	149.06	452.01	F1I
4	20.00	496736	0.06	495799	0.06	1353.75	1.22	149.47	450.59	F2I
5	20.00	493530	0.06	492583	0.06	1251.13	1.26	149.40	453.43	F2I
6	20.00	495577	0.06	494648	0.06	1276.87	1.25	149.47	452.99	F3I
7	20.00	495442	0.06	494487	0.06	1292.90	1.24	149.24	451.89	F3I
8	20.00	494411	0.06	493455	0.06	1239.95	1.27	149.30	454.49	F4I
9	20.00	497786	0.06	496836	0.06	1395.88	1.20	149.19	451.35	F4I
10	20.00	495787	0.06	494820	0.06	1226.76	1.28	149.72	455.10	F5I
11	20.00	497260	0.06	496277	0.06	1146.23	1.32	150.20	459.40	F5I
12	20.00	325790	0.08	325244	0.08	766.44	1.62	149.23	452.62	GI
13	20.00	322450	0.08	321912	0.08	807.47	1.57	149.44	450.94	GI
14	20.00	331649	0.08	331101	0.08	829.07	1.55	149.81	452.07	G2I
15	20.00	334645	0.08	334078	0.08	791.81	1.59	149.33	452.28	G2I
16	20.00	406520	0.07	405800	0.07	1077.72	1.36	149.87	451.64	G3I
17	20.00	279794	0.08	279355	0.08	645.45	1.76	150.03	448.99	G3I
18	20.00	144412	0.12	144182	0.12	347.10	2.40	149.67	450.93	G4I
19	20.00	143245	0.12	143034	0.12	337.76	2.43	148.79	448.67	G4I
20	20.00	41.60	6.93	40.83	7.00	9.45	14.55	92.884	451.99	BLK

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	31.15	8.01	30.21	8.14	10.10	14.07	81.908	445.24	Blk 9
2	20.00	1977.00	1.01	1971.34	1.01	21.20	9.71	147.61	457.54	waste jug 511-0228
3	20.00	24365.6	0.29	24326.0	0.29	50.62	6.29	146.32	445.66	F 1 F9
4	20.00	30766.5	0.25	30713.1	0.26	65.10	5.54	147.38	446.38	F 1 F5
5	20.00	109756	0.13	109579	0.14	227.00	2.97	148.07	448.97	F 2 F9
6	20.00	118832	0.13	118647	0.13	247.38	2.84	147.89	448.64	F 2 F5
7	20.00	175805	0.11	175528	0.11	373.60	2.31	148.46	449.67	F 3 F9
8	20.00	181017	0.11	180722	0.11	381.28	2.29	148.43	449.75	F 3 F5
9	20.00	297945	0.08	297442	0.08	682.72	1.71	149.41	451.04	F 4 F9
10	20.00	298352	0.08	297829	0.08	663.02	1.74	149.58	454.46	F 4 F5
11	20.00	402759	0.07	401991	0.07	874.49	1.51	150.16	457.69	F 5 F9
12	20.00	401537	0.07	400776	0.07	924.80	1.47	150.59	457.85	F 5 F5
13	20.00	264642	0.09	264191	0.09	604.46	1.82	149.56	453.84	6 1 F9
14	20.00	267283	0.09	266825	0.09	587.72	1.84	149.84	455.75	6 1 F5
15	20.00	346647	0.08	346024	0.08	760.15	1.62	150.17	457.63	6 2 F9
16	20.00	184899	0.10	184601	0.10	406.63	2.22	149.48	453.45	6 2 F5
17	20.00	46633.7	0.21	46560.6	0.21	95.20	4.58	147.70	448.71	6 3 F9
18	20.00	33007.2	0.25	32955.7	0.25	74.26	5.19	148.11	449.33	6 3 F5
19	20.00	6367.40	0.56	6355.95	0.56	21.35	9.68	146.67	448.22	6 4 F9
20	20.00	6361.09	0.56	6350.97	0.56	19.11	10.23	147.64	451.06	6 4 F5
21	20.00	30.25	8.13	29.84	8.19	8.15	15.67	78.620	450.81	Blk 6

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	29.45	8.24	28.90	8.32	7.65	16.17	80.526	447.70	Blk a
2	20.00	507273	0.06	506284	0.06	1233.59	1.27	149.19	452.44	E 1Ia
3	20.00	507615	0.06	506601	0.06	1155.20	1.32	149.64	457.44	E 1Ib
4	20.00	509043	0.06	508025	0.06	1148.53	1.32	148.81	456.55	E 2Ia
5	20.00	509424	0.06	508431	0.06	1298.03	1.24	149.72	453.99	E 2Ib
6	20.00	510725	0.06	509722	0.06	1191.11	1.30	148.25	452.31	E 3Ia
7	20.00	513367	0.06	512342	0.06	1090.60	1.35	148.08	456.03	E 3Ib
8	20.00	52936.3	0.19	52854.4	0.19	113.70	4.19	145.30	442.16	E 4Ia
9	20.00	51149.7	0.20	51072.1	0.20	112.44	4.22	145.13	439.09	E 4Ib
10	20.00	496527	0.06	495662	0.06	661.81	1.74	114.42	343.27	E 5Ia
11	20.00	509574	0.06	508656	0.06	632.53	1.78	113.23	342.07	E 5Ib
12	20.00	51657.7	0.20	51591.7	0.20	58.61	5.84	110.50	329.42	E 7Ia
13	20.00	54583.7	0.19	54512.9	0.19	58.94	5.83	109.88	327.80	E 7Ib
14	20.00	28.75	8.34	28.04	8.45	9.40	14.59	75.018	451.00	Blk b

Time: 20.00
Data Mode: CPM Nuclide: MANUAL
Background Subtract: None

	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	300	0	0.0	0.00
Region B:	2.0 -	300	0	0.0	0.00
Region C:	300 -	2000	0	0.0	0.00

Quench Indicator: tSIE/AEC
Ext Std Terminator: Count
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: A:\AEN09\PROT.DAT
Count Data Filename: A:\AEN09\SDATA21.DAT
Spectrum Data Drive & Path: A:\AEN09

S#	TIME	CPMA	A:2S%	CPMB	B:2S%	CPMC	C:2S%	SIS	tSIE	FLAG
1	20.00	30.90	8.05	30.35	8.12	8.85	15.03	84.731	446.09	BLK9
2	20.00	158180	0.11	157929	0.11	328.43	2.47	148.56	450.67	E1F4
3	20.00	172124	0.11	171846	0.11	368.26	2.33	148.52	451.44	E1F6
4	20.00	165919	0.11	165654	0.11	352.42	2.38	148.74	450.43	E2F4
5	20.00	201151	0.10	200817	0.10	418.24	2.19	148.72	452.17	E2F5
6	20.00	236794	0.09	236413	0.09	510.21	1.98	148.31	449.91	E3F4
7	20.00	240590	0.09	240187	0.09	497.57	2.00	148.30	451.43	E3F5
8	20.00	24433.1	0.29	24398.0	0.29	53.16	6.13	146.22	443.62	E4F4
9	20.00	21330.2	0.31	21295.8	0.31	50.13	6.32	146.68	443.81	E4F5
10	20.00	198892	0.10	198608	0.10	235.85	2.91	118.68	352.49	E5F4
11	20.00	211359	0.10	211057	0.10	231.55	2.94	120.02	356.40	E5F5
12	20.00	19811.3	0.32	19781.1	0.32	26.54	8.68	116.07	347.79	E7F4
13	20.00	21107.3	0.31	21077.1	0.31	24.17	9.10	116.27	347.38	E7F5
14	20.00	28.95	8.31	28.42	8.39	8.30	15.52	79.970	456.13	BLK5

SYSTEM NORMALIZED

C14 IPA DATA PROCESSED - 08-Jun-2001 06:05
C14 Eff (0-156 keV) = 96.66 %
C14 CHI SQUARE IPA DATA PROCESSED - 08-Jun-2001 06:15
C14 Chi Square = 22.54
H3 IPA DATA PROCESSED - 08-Jun-2001 06:16
H3 Eff (0-18.6 keV) = 64.95 %
H3 CHI SQUARE IPA DATA PROCESSED - 08-Jun-2001 06:27
H3 Chi Square = 13.80

Appendix D

Sample Calculations

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ENGINEERING WORKSHEET

Prepared By:	Date:	Project:
Brian Rapko	12-18-01	42365

Title/Subject: Sample Calculations for SuperLig® 639 Report

Sample Calculations:

① Calculation of molarity, M. Ex: Figure 6 (see BNW 57652, pg. 12)

Data: Formula weight NaCl = 58.44 g/mol
Formula weight $NaNO_3$ = 84.99 g/mol

Note: 0.1 M NaOH solution prepared by dilution of stock solution (see BNW ~~57652~~ 57652, pg 3 (BR 12-18-01)

Ex: Target Solution 4 M NaCl, 0.9 M in $NaNO_3$, 0.1 M in NaOH

Data: 11.6974 g NaCl } in 50mL 0.1 M NaOH
& 3.8155g $NaNO_3$ }

$$M = \frac{\text{moles component}}{L} \quad \text{(definition)}$$

$$\text{moles} = \frac{\text{mass}}{\text{molecular wt = formula wt}} = \frac{11.6974\text{ g NaCl}}{0.05\text{ L} \;\; 58.44\text{ g NaCl/mole NaCl}} = 0.2002 \text{ moles NaCl}$$

(BR 12-18-01)

$$M_{NaCl} = \frac{0.2002 \text{ moles}}{0.05 \text{ L}} = 4.003 \text{ M NaCl}$$

Similarly $\frac{3.8155\text{ g } NaNO_3}{84.99\text{ g/mole}} = 0.04489 \text{ moles } NaNO_3$

$$\frac{0.04489 \text{ moles}}{0.05 \text{ L}} = 0.8979 \text{ M } NaNO_3$$

Total M (Na^+) = $M_{NaCl} + M_{NaNO_3} + M_{NaOH}$ = 4.003 + 0.8979 + 0.1 = 5.001 M ≈ 5 M

Note: Average Kd and standard deviations calculated by Excel® spreadsheet directly.

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Prepared By: Brian Rapko | Date: 12-18-01 | Project: 42365

Title/Subject: Sample Calculations for SuperLig® 639 Report

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② Calculation of molality, m. $m = \frac{\text{moles component}}{\text{kg solute}}$

Note: solute here is water

Ex: Calculation molality of solution 5 M NaCl, 0.01 M NaOH, 0.018 M $NaNO_3$

Data: solution density = $\frac{\text{g solution}}{\text{mL solution}}$ Data: Formula wt. NaOH = 40.00 g/mole

Aside density calculation data: 5 mL volumetric flask tare wt = 9.0029 g
flask + 5 mL solution = 14.9481 g

$$\text{density} = \frac{(\text{Final wt} - \text{Tare wt})\,g}{5\,mL} = \frac{(14.9481 - 9.0029)\,g}{5\,mL} = 1.189\,g/mL$$

So 1 L of solution = $1.188\,g/mL \times \frac{1000\,mL}{L} = 1189\,g/L$

Solution 5 M in NaCl has $\left(58.44\,g\ NaCl/mol \times 5\ mole\right)/L = 292.2\,g\ NaCl/L$

0.01 M in NaOH has $\left(40.00\,g\ NaOH/mol \times 0.01\ mol\right)/L = 0.4\,g\ NaOH/L$

0.018 M in $NaNO_3$ has $\left(84.99\,g\ NaNO_3/mol \times 0.018\ mol\right)/L = 1.5\,g\ NaNO_3/L$

So in 1 L total solute = 292.2 g + 0.4 g + 1.5 g = 294.1 g

Since total wt = solute + solvent ⇒ 1189 = 294.1 + g water

= 894.9 g water.

In 1000 g solvent the amount (g) of each solute component would need to be increased by $\frac{1000}{894.9} = 1.117$ factor. ⇒ molal = 1.117 × (Molarity)

So for NaCl molarity: 5 × 1.117 = 5.59
NaOH = 0.01 × 1.117 = 0.011
$NaNO_3$ = 0.018 × 1.117 = 0.0201

ENGINEERING WORKSHEET

Prepared By: Brian Rapko | Date: 12-18-01 | Project: 42365

Title/Subject: Sample Calculations for SuperLig® 639 Report

③ Calculation of solution ionic strength, I.

$I = \frac{1}{2} \sum m_i z_i^2$ where m_i = molality of ith component
z_i = charge of ith component

Note since $z = |1|$ for all ions in a solution

5 M NaCl, 0.01 M NaOH, 0.018 M $NaNO_3$ $I = \frac{1}{2} \sum m_i$

From ② we determined that for this solution $m = 1.117 * M$

$[Na]_T, M = ([NaCl]_M + [NaOH] + [NaNO_3]), M$

$= 5 + 0.01 + 0.018 = 5.028 = [Na]_T, M$

Similarly $[Cl] = 5 M$, $[NO_3] = 0.018 M$, $[OH] = 0.01 M$

So $[Na]_m = [Na]_M * 1.117$; $[Cl]_m = [Cl]_M * 1.117$; $[NO_3]_m = [NO_3]_M * 1.117$

& $[OH]_m = [OH]_M * 1.117$

So $[Na]_m = 5.028 * 1.117 = 5.62$; $[Cl]_m = 5 * 1.117 = 5.59$; $[NO_3]_m = 0.018 * 1.117 = 0.02$

$[OH] = 0.01 * 1.117 = 0.01$

total molality $= \frac{1}{2} \sum m_i = \frac{1}{2}(5.62 + 5.59 + 0.02 + 0.01)$

$= 5.62$

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ENGINEERING WORKSHEET

Prepared By: Brian Rapko	Date: 12-18-01	Project: 42365

Title/Subject: Sample Calculations for SuperLig® 639 Report

F-factor Calculation

Definition: $F = \frac{\text{dried resin wt, g}}{\text{stored resin wt, g}}$

Data: (from F_1 test data)

tare wt vial = 17.1494 g (a)
vial + stored resin = 17.3498 g (b)
vial + dried resin = 17.3016 g (c)

stored resin wt = b − a = 17.3498 − 17.1494 = 0.1984 g

dry resin wt = c − a = 17.3016 − 17.1494 = 0.1522 g

$F\text{-factor} = \frac{0.1522\ g}{0.1984\ g} = 0.7671$

K_d Calculation

$K_d = \frac{(C_o - C_i)}{C_i} * \frac{V}{(m * F)}$ definition.

C_o = initial mass conc. or activity conc.
C_i = equilibrium mass conc. or activity conc.
V = Test solution volume (10 mL for all tests)
m = mass of resin (g)
F = F-factor. (0.7671)

Ex: RT kinetics test - point 7A

Data: initial aliquot = 0.1003 g Activity = 502081 cpm
equilibrium aliquot = 0.1225 g Activity = 85691 cpm.

Volume = 10 mL, wt resin = 0.1003 g

$$K_d = \frac{\left[\left(\frac{502081\ cpm}{0.1003\ g}\right) - \left(\frac{85691\ cpm}{0.1225\ g}\right)\right]}{\left(\frac{85691\ cpm}{0.1225\ g}\right)} * \frac{10\ mL}{(0.7671 * 0.1003\ g)}$$

$$= 645\ \frac{mL}{g}$$

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ENGINEERING WORKSHEET

Prepared By: Brian Rapko	Date: 12-18-01	Project: 42365

Title/Subject: Sample Calculations for SpecLig 639 Report

Calculation of Equilibrium $[NO_3^-] / [TcO_4^-]$ Ex: Test D-1A

Data: Initial Activity concentration (see pg 4 of sample calculations)

$= 4370348 \frac{cpm}{g}$

Final activity concentration $= 220646 \frac{cpm}{g}$

35 microliters 0.415 M Tc stock solution into 10.1 mL test solution;

Dilution of ~~Test~~ Stock Solution to form Test Solution:

$= \frac{(\text{volume stock solution})}{(\text{Total volume Test solution})} * M \text{ stock solution}$

$= \left(\frac{0.035 mL}{10.135 mL}\right) * 0.415 M = 1.43 \times 10^{-3} M \ TcO_4^-$

Assuming $^{99}TcO_4^-$ activity $\propto$ $^{99}TcO_4$ concentration, then:

$$\frac{\text{Activity final Test solution}}{\text{Activity initial Test solution}} = \frac{M\ TcO_4^- \text{ in final test solution}}{M\ TcO_4^- \text{ in initial test solution}}$$

Rearranging & plugging in data yields.

$$(1.43 \times 10^{-3} M\ TcO_4^-) * \left(\frac{220646 cpm/g}{4370348 cpm/g}\right) = TcO_4, M \text{ equilibrium test solution},$$

$$= 7.22 \times 10^{-5} M.$$

Since the Test solution is ~~Assumed~~ known to 0.0018 M in NO_3 initial and (BA 12-18-01) is assumed to be unchanged:

$$\frac{[NO_3]}{[TcO_4]} \text{ initial} = \frac{[0.0018 M]}{[0.00143 M]} = 1.26$$

$$\frac{[NO_3]}{[TcO_4]} \text{ at equilibrium} \sim \frac{[0.0018 M]}{[7.22 \times 10^{-5} M]} = 2.49 \times 10^{1}$$

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