SRNL-STI-2012-00218
Revision 1

Determination of the Impact of Glycolate on ARP and MCU Operations

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December 2012

Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
Aiken, SC 29808

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

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REVIEWS AND APPROVALS

AUTHORS:

_____

K. M. L. Taylor-Pashow, Separations and Actinide Science Programs Date

_____

T. B. Peters, Separations and Actinide Science Programs Date

_____

F. F. Fondeur, Separations and Actinide Science Programs Date

_____

T. C. Shehee, Separations and Actinide Science Programs Date

_____

A. L. Washington, Advanced Characterization and Processing Date

TECHNICAL REVIEW:

_____

D. T. Hobbs, Separations and Actinide Science Programs Date

APPROVAL:

_____

S. D. Fink, Manager Date
Separations and Actinide Science Programs

_____

S. L. Marra, Manager Date
Environmental & Chemical Process Technology Research Programs

_____

E. J. Freed, Manager Date
SRR, Waste Solidification Engineering

EXECUTIVE SUMMARY

Savannah River Remediation (SRR) is evaluating an alternate flowsheet for the Defense Waste Processing Facility (DWPF) using glycolic acid as a reductant. An important aspect of the development of the glycolic acid flowsheet is determining if glycolate has any detrimental downstream impacts. Testing was performed to determine if there is any impact to the strontium and actinide sorption by monosodium titanate (MST) and modified monosodium titanate (mMST) or if there is an impact to the cesium removal, phase separation, or coalescer performance at the Modular Caustic-Side Solvent Extraction Processing Unit (MCU).

Sorption testing was performed using both MST and modified MST (mMST) in the presence of 5000 and 10,000 ppm (mass basis) glycolate. 10,000 ppm is the estimated bounding concentration expected in the DWPF recycle stream based on DWPF melter flammable gas model results. The presence of glycolate was found to slow the removal of Sr and Pu by MST, while increasing the removal rate of Np. Results indicate that the impact is a kinetic effect, and the overall capacity of the material is not affected. There was no measurable effect on U removal at either glycolate concentration. The slower removal rates for Sr and Pu at 5000 and 10,000 ppm glycolate could result in lower DF values for these sorbates in ARP based on the current (12 hours) and proposed (8 hours) contact times. For the highest glycolate concentration used in this study, the percentage of Sr removed at 6 hours of contact decreased by 1% and the percentage of Pu removed decreased by nearly 7%. The impact may prove insignificant if the concentration of glycolate that is returned to the tank farm is well below the concentrations tested in this study.

The presence of glycolate also decreased the removal rates for all three sorbates (Sr, Pu, and Np) by mMST. Similar to MST, the results for mMST indicate that the impact is a kinetic effect, and the overall capacity of the material is not affected. The presence of glycolate did not change the lack of affinity of mMST for U.

Pre-contacting the MST or mMST with glycolate did not have a significant effect on the performance of the materials when compared to tests having the same concentration of glycolate present in the simulant. These findings suggest that the glycolate is likely influencing removal by sorbate complexation and not by depositing onto or forming a film on the surface of the MST solids.

Since the DF values are salt batch dependent, it is not possible to a priori quantify the impacts of glycolate on future processing campaigns. However, we recommend that the impacts of glycolate be evaluated during each salt batch qualification when a final processing concentration is defined, and recommendations can then be made on how to mitigate negative impacts, if needed. Impacts to the performance of the MST or mMST could be mitigated by increasing contact time or increasing sorbent concentrations.

Contacting mMST with glycolate did not reduce the concentration of peroxide groups on the solids, suggesting no reaction between the peroxide groups and added glycolate. Analysis of the slurries after 5 months showed minimal amounts of dissolved Ti in solution, suggesting little, if any, impact of glycolate on the dissolution rate for the MST and mMST. Addition of glycolate had a minor impact on the measured particle size distribution for MST, shifting the mean particle size slightly lower. No significant shift in particle size was observed for mMST.

Testing was performed to determine if there is an impact to the cesium removal at Modular Caustic-Side Solvent Extraction Processing Unit (MCU). An Extraction-Scrub-Strip (ESS) test

routine was used to simulate cesium removal at the MCU. For this, SRNL performed three ESS tests, using the same basic aqueous waste simulant and solvent. For one test, SRNL added 5000 ppm (mass basis) of glycolate and added 10,000 ppm of glycolate to a second test. A control test contained no glycolate. The results of all three tests were virtually identical for all the extraction, scrub and strip tests. A single data point in the 5000 ppm test is physically impossible and SRNL does not feel that it affects the conclusion of these tests. At this time, SRNL concludes that the presence of up to 10,000 ppm of glycolate does not affect cesium removal by the current solvent system used in the MCU.

SRNL also performed a series of three dispersion tests with the BOBCalixC6 solvent against the caustic salt simulant. For one test, SRNL added 5000 ppm (mass basis) of glycolate and added 10,000 ppm of glycolate to a second test. A control test contained no glycolate. The results of all three tests were virtually identical, indicating to detrimental effect of glycolate on the phase disengagement behavior.

Further ESS testing was performed with glycolate to determine if glycolate has a detrimental effect on the Next Generation Solvent (NGS)[a] proposed for use in MCU and in the Salt Waste Processing Facility (SWPF). For this, SRNL performed three ESS tests, using the same basic aqueous waste simulant and solvent. For one test, SRNL added 5000 ppm (mass basis) of glycolate and added 10,000 ppm of glycolate to a second test. A control test contained no glycolate. The results of all three tests were virtually identical for the extraction, scrub and strip tests. At this time, SRNL concludes that the presence of up to 10,000 ppm of glycolate does not affect cesium removal by the new solvent system proposed for use in the MCU and SWPF.

Microscopic and coalescing tests demonstrated that salt solution containing 10,000 ppm sodium glycolate had no effect on the coalescing function of the MCU coalescer media. Glycolate had no effect on the coalescing ability of a gamma irradiated coalescer (8 E6 rad). Observed losses in glycolate concentration are due to solution dilution and sorption onto CSSX solvent droplets.

SRNL recommends determining the amount of glycolate that partitions to the solvent during ESS testing. If the amount that partitions to the solvent is significant testing should also be performed to examine the glycolate – coalescer interactions during stripping (acidic conditions). We also recommend performing material compatibility evaluations with the various polymers used in MCU to ensure that glycolate does not negatively affect the physical properties. Finally, SRNL recommends that additional testing be performed if the glycolate concentration exceeds 10,000 ppm in the DWPF recycle stream.

[a] For the purposes of this report, NGS solvent refers to the NGS formulation using the LIX-79 ® guanidine derivative.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
BOBCalixC6	cesium extractant used in baseline MCU process; calix[4]arene-bis-(tert-octylbenzo-crown-6)
CSSX	Caustic-Side Solvent Extraction; the baseline solvent extraction process for MCU and SWPF which uses BOBCalixC6 as the extractant and trioctylamine as the suppressor
DF	decontamination factor
ESS	Extraction-Scrub-Strip
FTIR	Fourier transform infrared spectroscopy
IC	ion chromatography
ICP-ES	inductively couples plasma – emission spectroscopy
ICP-MS	inductively coupled plasma – mass spectroscopy
MaxCalix	cesium extractant for NGS process; 1,3-alt-25,27-bis(3,7-dimethyloctyl-1-oxy)calix[4]arenebenzocrown-6
MCU	Modular Caustic-Side Solvent Extraction Processing Unit
MST	monosodium titanate
mMST	modified monosodium titanate
NGS	Next Generation Solvent
NTU	Nephelometric turbidity units
PuTTA	plutonium thenoyltrifluoroacetone scintillation
PVDF	polyvinylidene fluoride
SEE	Systems Engineering Evaluation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SWPF	Salt Waste Processing Facility
TTQAP	Task Technical and Quality Assurance Plan

1.0 Introduction

Savannah River Remediation (SRR) recently conducted a Systems Engineering Evaluation (SEE) to determine the optimum alternate reductant flowsheet for the Defense Waste Processing Facility (DWPF). Specifically, two proposed flowsheets (Nitric/Formic/Glycolic and Nitric/Formic/ Sugar) were evaluated based upon results from preliminary testing. Comparison of the two flowsheets against several weighted technical and business evaluation criteria indicated a preference towards the Nitric/Formic/Glycolic flowsheet. As a result, the Nitric/Formic/Glycolic flowsheet was recommended for further testing.[1] Subsequently, SRNL demonstrated the viability of a Glycolic/Nitric Acid flowsheet, and SRR is currently proceeding with the development and demonstration of that flowsheet.[2]

An important aspect of the development of the glycolic acid flowsheet is determining if glycolate has any detrimental downstream impacts. Therefore, testing was performed to determine if there is any impact to the strontium and actinide sorption by monosodium titanate (MST) in the Actinide Removal Process (ARP). Testing was also performed to determine the impact to cesium mass transfer in the solvent extraction process in the Modular Caustic Side Solvent Extraction Unit (MCU). A third set of tests examined the interaction of the glycolate with the coalescer material used in MCU.

This work was performed at the request of SRR Engineering[3] and was controlled by a Task Technical and Quality Assurance Plan (TTQAP).[4]

2.0 Experimental Procedure

2.1 Sources of MST and mMST

The baseline MST used in these studies was prepared using a sol-gel process developed at the Savannah River National Laboratory (SRNL) and supplied by Optima Chemical Group LLC (Douglas, GA, Lot #00-QAB-417) as a 15 wt % suspension in water containing 0.10-0.15 M NaOH and 100-150 mg L^{-1} $NaNO_2$.[5] The modified MST (mMST) used in these studies was prepared by the post-synthesis treatment of MST with hydrogen peroxide. The details of this procedure have been previously published.[6] A 25 g supply of the mMST (LS-11) was prepared using the Optima-supplied MST.

2.2 Simulant Preparation for MST and mMST Testing

The simulant used in this testing was prepared by the addition of glycolate to an already prepared simulant (SWS-5-2009) with the composition shown in Table 2-1. This simulant is considered conservative for measuring the effect of glycolate on MST sorption, due to the lower hydroxide and sodium concentrations. At high hydroxide concentrations, the hydrolysis products of the actinides are expected to be dominant (see Appendix A). Therefore, increasing the sodium and hydroxide concentrations of the simulant would further reduce any interaction of glycolate with the species of interest (i.e., strontium and actinides).

Two glycolate containing simulants were prepared with targeted glycolate concentrations of 10,000 and 5000 ppm (on a mass basis). 10,000 ppm is the estimated bounding concentration expected in the DWPF recycle stream.[7] Tests were also performed using simulant that had not been spiked with glycolate for comparison. For the 5000 ppm glycolate simulant, 2.2870 g (0.0233 mol) of sodium glycolate was dissolved in 15 mL of salt solution (SWS-1-2010, see Table 2-1 for composition). This solution was then added to 350 mL of SWS-5-2009. The simulant was equilibrated at room temperature for 4 days, after which a sample was removed,

filtered, and analyzed for glycolate concentration using ion chromatography (IC). A similar procedure was followed to prepare the 10,000 ppm glycolate simulant, using 4.5743 g (0.0467 mol) of sodium glycolate dissolved in 25 mL of SWS-1-2010. In this case the sodium glycolate did not completely dissolve in the 25 mL of salt solution, but the suspension was added to the simulant, where the remaining sodium glycolate dissolved. The equilibrated simulants were then used directly for the sorption testing, without filtering.

Table 2-1. Compositions of Simulated Waste Solution (SWS-5-2009) and Salt Solution (SWS-1-2010).

Component	Simulant (SWS-5-2009)	Salt Solution (SWS-1-2010)
Free NaOH	1.37 M	1.33 M
Total $NaNO_3$	2.13 M	2.90 M
$NaAl(OH)_4$	0.404 M	-
$NaNO_2$	0.133 M	0.149 M
Na_2SO_4	0.483 M	0.581 M
Na_2CO_3	0.0298 M	0.029 M
Total Na	5.05 M	5.6 M
^{85}Sr	30,000 dpm/mL (target)	-
Total Sr	6.85×10^{-6} M	-
^{137}Cs	96,300 dpm/mL	-
Total Cs	1.26×10^{-4} M	-
Pu	220 µg/L	-
Np	460 µg/L	-
U	10,700 µg/L	-

2.3 Simulant preparation for the ESS test

Simulant for the ESS test was provided by a previously prepared general purpose simulant. To three bottles (205 mL) of this material, glycolic acid was added at 0, 5000, or 10,000 ppm (by mass). The simulants were stirred for three days with no observable precipitation. Each solution was then spiked with enough ^{137}Cs to achieve a final activity of 1.50E+05 dpm/mL. See Table 2-2 for a summary of the composition.

Table 2-2. Compositions of Simulated Waste Solutions for the ESS Tests

Component	Simulant (M)
Free NaOH	2.02
Total $NaNO_3$	1.99
$NaAl(OH)_4$	0.274
NO_2^-	0.490
SO_4^{2-}	0.137
CO_3^{2-}	0.147
Total Na	5.47
^{137}Cs	1.50E+05 dpm/mL

2.4 Sorption Tests

A total of 20 individual sorption tests were performed. Tests 1-5 were performed using simulant SWS-5-2009 with no glycolate present, tests 6-10 were performed using SWS-5-2009 spiked with 5000 ppm glycolate, and tests 11-15 were performed with SWS-5-2009 spiked with 10,000 ppm glycolate. 60 mL of the appropriate simulant were used for each of the tests 1-15. Tests 16-20 represent a more conservative test which was modeled based on previous testing to evaluate possible scale inhibitors for the high level waste evaporators.[8] In this set of tests, samples of MST and mMST were contacted with sodium glycolate overnight (without agitation)

prior to adding to the simulated waste solution. The amount of glycolate contacted with the MST and mMST was the amount required to provide a 10,000 ppm solution once the mixture was added to the test bottles containing the simulant. A stock solution of sodium glycolate was prepared by dissolving 4.31073 g of sodium glycolate in 8.25 mL of distilled water. Aliquots (0.95 mL) of this solution were then added to the samples of MST and mMST to be used in tests 16-20. After contacting overnight, the MST/mMST and glycolate mixtures were added to the test bottles containing 38 mL of SWS-5-2009 each. Table 2-3 provides the molar concentrations of the sorbates compared to the molar concentrations of glycolate and the peroxo species on mMST (mMST tests only).

Table 2-3. Molar Concentrations of Sorbates, Glycolate, and Peroxo Groups on mMST.

	5000 ppm Glycolate Tests	10,000 ppm Glycolate Tests
Sr	6.85×10^{-6} M	6.85×10^{-6} M
Pu	9.20×10^{-7} M	9.20×10^{-7} M
Np	1.94×10^{-6} M	1.94×10^{-6} M
U	4.50×10^{-5} M	4.50×10^{-5} M
Glycolate	6.66×10^{-2} M	1.33×10^{-1} M
Peroxo Groups (mMST tests only)	6.42×10^{-4} M	6.42×10^{-4} M

Each set of 5 tests consisted of a control bottle (no sorbent), two bottles containing MST (duplicate tests) and two bottles containing mMST (duplicate tests). The control bottle was sampled at each sampling event to monitor for any changes in sorbate concentration due to precipitation or sorption by the polyethylene bottle. MST and mMST were added to the appropriate bottles at concentrations of 0.4 g/L and 0.2 g/L, respectively. After adding the sorbents, the bottles were placed in a shaker-oven, maintained at an average temperature of 27.0 ± 1.1 °C for tests 1-15, and 25.8 ± 1.2 °C for tests 16-20. The target temperature for both sets of tests was 25 °C; however, heat from the shaker motor make maintaining this temperature difficult. The bottles were continually shaken at 175 rpm for the duration of the test. Samples were removed at times of 6, 12, 24, and 168 hours. At each sampling event, the bottle was removed from the oven and manually shaken for 30 seconds to ensure the solids were homogeneously suspended. A sample was then removed and filtered through a 0.1-µm polyvinylidene fluoride (PVDF) syringe filter to remove the solids. An aliquot of the filtrate was acidified with an equal volume of 5 M nitric acid and submitted for inductively coupled plasma – mass spectroscopy (ICP-MS), gamma scan, and plutonium thenoyltrifluoroacetone scintillation (PuTTA) analyses.

2.5 Post-Sorption Testing Measurements

Samples of the supernate from the test bottles were removed by filtering samples through a 0.1-µm PVDF syringe filter to remove the solids. These samples were then submitted for inductively coupled plasma – emission spectroscopy (ICP-ES) and IC anion analyses to determine the concentrations of Ti and glycolate in the supernate. These samples were removed from the test bottles approximately 5 months after the start of the sorption testing.

In addition to the supernate samples, samples of the solids were also removed at this time and were submitted for particle size analysis.

2.6 Measurement of Peroxide Content

To determine if glycolate reacts with the peroxide groups present in mMST, iodometric titrations were performed on samples of mMST before and after exposure to sodium glycolate. The glycolate contact was modeled after the pre-contact performed for sorption Tests 19 and 20 (see Section 2.4). 13.064 g of sodium glycolate was dissolved in 25 mL of distilled water. 1.292 g of

a 15.53 wt % slurry of mMST (LS-11) was then added to the glycolate solution, and the mixture was stirred at room temperature, overnight. For the control experiment, 1.290 g of the 15.53 wt % slurry was added to 25 mL of distilled water, and was stirred overnight. The mixtures were then filtered to isolate the mMST solids. The solids were washed three times with distilled H_2O, and were then slurried from the filter into 10-mL volumetric flasks. Concentrated sulfuric acid (0.42 mL) was then added to each flask, and the mixtures were diluted to total volumes of 10 mL with additional distilled H_2O. The mixtures were then transferred to glass vessels, and 10 mL of 0.27 M NaI solution was added to each vessel. The reactions were then stirred at room temperature, overnight. Aliquots (6-mL) of the reaction mixtures were then titrated to the end-point with 0.1 M sodium thiosulfate, using starch as an indicator. The titrations were performed in triplicate for each sample.

2.7 Examination of Glycolate on MST by FTIR

The MST and mMST solids from the sorption testing (Tests 1-20) were collected by filtration and allowed to air dry. The solids were not washed prior to FTIR measurements.

In addition, a series of experiments was performed exposing MST to a series of solutions of varying pH containing sodium glycolate. For these experiments solutions containing 10,000 ppm glycolate were prepared and the pH of the solution was adjusted with either nitric acid or sodium hydroxide to reach final pH values of 3, 5, 7, 9, and 11. Aliquots of MST were then contacted with the solutions for 24 hours. After contacting, the solids were isolated and washed with the same pH solution without glycolate present. The FTIR spectra were then collected on the dried solids.

2.8 ESS Tests

For each ESS test, the researchers used a nominal starting volume of 120 mL of aqueous salt simulant and 40 mL of fresh, unused solvent (S2-D1-YESBOB-T-WI).[Υ] For the MCU solvent testing, the aqueous to organic phase ratio volume was 3:1. The general test protocol is the same one used in all MCU feed qualification work.[9]

The ESS test sequence involves vigorously contacting the cesium loaded aqueous phase with fresh, unused CSSX solvent, in a 3:1 aqueous:organic volume phase ratio. The aqueous phase is then removed, and the remaining organic phase is contacted in turn, with scrub acid (0.05 M HNO_3) twice and strip acid (0.001 M HNO_3) three times. In each case, the time of contact is 24 hours and, except for the initial contact, the aqueous:organic volume phase ratio is 1:5. After the 24 hour contact period, the aqueous phase is removed. During each step, samples of each phase are removed and analyzed for ^{137}Cs content. The resulting D-value is defined as the activity of the ^{137}Cs in the organic phase divided by the ^{137}Cs activity in the aqueous phase. This value is then temperature corrected.

ESS tests using NGS solvent were also performed. NGS solvent uses the same modifier and diluent as the current solvent, but with a different extractant and suppressor. See Table 2-4 for a comparison of the current and NGS compositions.

[Υ] This batch of solvent was originally prepared with no extractant as S2-NOBOB-T-WI (see WSRC-NB-2005-00060). The extractant was added later (see WSRC-NB-2007-00054).

Table 2-4. Compositions of CSSX Solvents (Current and NGS)

Component	Concentration
Current Solvent Composition	
BOBCalixC6[⊃]	0.007 M
Cs-7B Modifier [Σ]	0.75 M
Trioctylamine	0.003 M
Isopar-L ™	balance
NGS Solvent Composition	
MAXCalix [♣]	0.05 M
Cs-7B Modifier	0.5 M
LIX-79 guanidine [ℜ]	0.003 M
Isopar-L ™	balance

For the NGS solvent the same general procedure was used, except for changes required by the different material chemistries. The extraction aqueous:organic volume phase ratio was 4:1. The scrub and strip aqueous:organic volume phase ratios were 1:3.75. The NGS scrub solution was 0.025 M NaOH and the NGS strip acid was 0.01 M boric acid. See Table 2-5 for a comparison of conditions for the current and NGS solvents.

Table 2-5. Comparison Conditions of Current and NGS Testing

Solvent/Step	A:O Ratio	Solution
Current/Extraction	3:1	n/a
Current/Scrub	1:5	0.05 M HNO_3
Current/Strip	1:5	0.001 M HNO_3
NGS/Extraction	4:1	n/a
NGS/Scrub	1:3.75	0.025 M NaOH
NGS/Strip	1:3.75	0.01 M boric acid

2.9 Dispersion Testing

Dispersion testing is a method to test phase disengagement between an organic and aqueous phase.[10] For these tests, the researchers used 75 mL of the same salt simulant outlined in Section 2.3, but without the radioisotopes present, and 25 mL of fresh, unused solvent (S2-D1-YESBOB-T-WI). Sodium glycolate was added to aliquots of the salt simulant to give three different solutions with glycolate concentrations of 0, 5000, and 10,000 ppm (mass basis). The two phases were carefully layered into a 100 mL graduated cylinder, with the aqueous phase being added first. The phase boundary line was then marked. After the stopper was added the flask was turned end-over-end 10 times and then set down. A stopwatch was triggered and the time for the two phases to completely disengage was noted. The test was repeated so each aqueous and organic phase combination had two trials, for a total of six tests. The dispersion value was then calculated, with higher values indicating a shortened time to fully phase disengage.

[⊃] BoBCalixC6 stands for calix[4] arene-bis-(tert-octylbenzo)-crown-6
[Σ] Modifier is 1-(2,2,3,3,-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol
[♣] MAXCalix stands for 1,3-*alt*-25,27-Bis(3,7-dimethyloctyloxy)calix[4]arenebenzocrown-6
[ℜ] The LIX-79 suppressor is a derivitized guanidine, N, N'-cyclohexyl, N''-tridecyl guanidine

2.10 Impacts of Glycolate on Coalescer

Coalescer Description

A 1.2 meter long coalescer consisting of nonwoven Ryton (or polyphenylene sulfide) fibers (10 microns in diameter and with ~89 vol % porosity) wrapped around a perforated stainless steel tube (containing 56 holes of 2.4 mm diameter per inch of tube) was cut to generate four cylindrical tubes, 2.7 inches tall each. Two of the coalescer pieces were placed in a Shepherd Gamma source where one piece received 8 E6 rad (i.e., ~109 times the annual dose at SWPF) and the other received 4 E5 rad (i.e., ~5.4 times the annual dose at SWPF). The calculated dose for the coalescer is based on an expected 73.4 krad of exposure annually at SWPF.[11] The lower dose was chosen to provide the equivalent of ~5 years of exposure, while the second, much higher dose, was chosen to increase the possibility of observing any effect of the irradiation.

Optical Picture of Solvent Droplets on Ryton Fibers

Estimates of the effect of glycolate on the interfacial tension of CSSX on Ryton fibers were obtained by examining the optical pictures of coalescer fibers exposed to a CSSX solvent emulsion in salt solution. The composition of the salt solution is provided in Table 2-6. A square portion of the coalescer (as received and gamma irradiated) was used to filter a recirculating salt solution containing dispersed CSSX solvent. After flowing for five minutes, the filter was removed and placed in a glass slide containing salt solution.

Table 2-6. Composition of Salt Solution Used in Coalescer Testing

Component	Concentration (M)
Free OH^-	1.33
$NaNO_3$	2.60
$NaAl(OH)_4$	0.429
$NaNO_2$	0.134
Na_2SO_4	0.521
Na_2CO_3	0.026
Total Na^+	5.59

CSSX Solvent Dispersion in Salt Solution

For this test approximately 12.55 grams of CSSX solvent were added to 1 L (1255.34 g) of salt solution. The aggregate was mixed with a Tissue-Tearor model 985370 homogenizer (Biospec Products) at 15,000 rpm for 5 minutes. The maximum temperature rise during the mixing was 2.1 °C. The outer rotor diameter is 12.7 mm or 0.5 inches and the inner rotor diameter is 8.89 mm or 0.35 inches. This mixer gave a shear rate of 6160 per minute at the rotor surface. A typical emulsion appearance from shearing salt solution containing CSSX with this homogenizer is shown in Figure 2-1. .

Figure 2-1. Physical appearance of the emulsion obtained using the homogenizer on salt solution containing CSSX solvent.

The turbidity of the emulsion was measured with a Micro 100 turbidity meter from Scientific Inc. This instrument measures the side scattered (90° from the incoming light source) and the transmitted radiation. The turbidity data are provided in Nephelometric turbidity units (NTU). The turbidity meter was calibrated before testing with three different concentrations of polystyrene bead standard suspensions that read 0.01, 10, and 10,000 nephelometric units. A 0.1% deviation was observed with the 10 NTU standard after conducting the dispersion and coalescing test.

Coalescing Test Protocol

To emulate the coalescer operation at MCU, a smaller scale test was conducted to provide insight to the impact of sodium glycolate, if any, on CSSX solvent coalescing on Ryton fibers. Emulsified salt solution containing approximately 10,000 ppm sodium glycolate was pumped at 50 mL/min through a piece of the coalescer (either irradiated or as received) as shown in Figure 2-2. The flow rate represents the minimum flow rate conducted in a recent centrifugal contactor and coalescer test at SRNL.[12] The coalescer was placed in vertical orientation (as opposed to the horizontal configuration used at MCU). In this configuration, the diameter of large droplets is physically comparable to the open spaces between the fibers retarding and stopping the larger droplets from exiting (forcing them to wonder longer paths in the MCU coalescer). Since the exterior of the coalescer was open to the atmosphere, the pressure differential between the inside of the coalescer and the outside was minimal, but was sufficient to promote CSSX solvent coalescing on the fibers.

Figure 2-2. A picture of the coalescer in vertical configuration.

Several pulses of salt solution and salt solution containing sodium glycolate were conducted on both the as-received and irradiated coalescer to evaluate the reversibility (or irreversibility) of the coalescer to sodium glycolate deposition as shown in Figure 2-3. Solution samples from every stage in the testing were submitted for particle size distribution to evaluate the coalescing performance of the coalescer. Samples were also submitted for IC - anions quantification and liquid Raman analysis to determine the glycolate level in solution and to determine any sorption onto the coalescer.

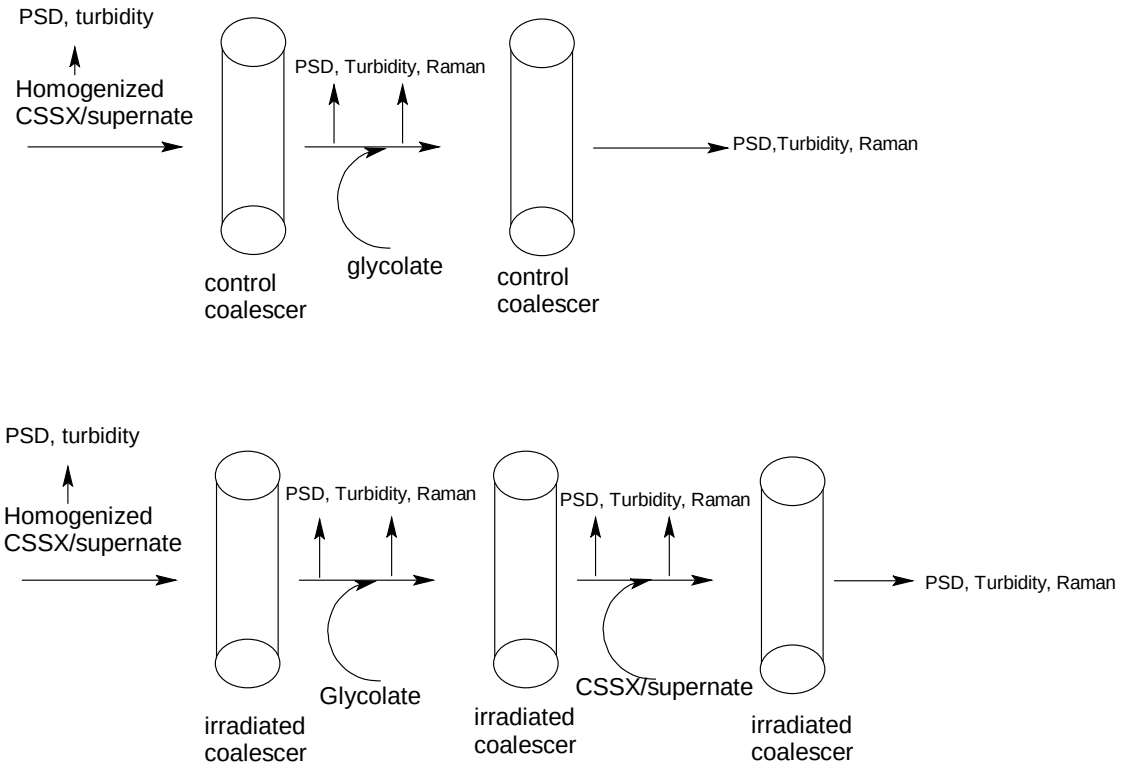

Figure 2-3. Schematic of the testing protocol conducted to evaluate the effect of glycolate on as-received and irradiated coalescer.

3.0 Results and Discussion

3.1 Simulants

The simulants used for the MST and mMST testing were prepared by spiking a previously prepared simulant with sodium glycolate, targeting final glycolate concentrations of 5000 and 10,000 ppm. After the addition of sodium glycolate, the simulants were equilibrated for 4 days and were then analyzed for soluble glycolate concentration using IC. The measured concentrations came within the 10% reported analytical uncertainty of the target concentrations, indicating no issues with glycolate solubility in the simulant. The results are provided in Table 3-1.

Table 3-1. Measured Glycolate Concentrations

Glycolate Concentration	SWS-5-2009 (no glycolate)	SWS-5-2009 w/5000 ppm glycolate	SWS-5-2009 w/10,000 ppm glycolate
Target	0 ppm	5000 ppm	10,000 ppm
Measured	< 100 ppm	4790 ppm	10,700 ppm

Given the lack of issues in the glycolate spiking in the MST and mMST testing, the glycolate content in the ESS simulants was not measured.

3.2 MST Performance

Figures 3-1 through 3-3 show the percent removal for ^{85}Sr, Pu, and Np as a function of contact time for sorption tests performed with MST in simulants with glycolate concentrations of 0 ppm, 5000 ppm, and 10,000 ppm (both in solution and pre-contacted with the MST). The data presented in these plots are the average of the duplicate trials, with the error bars representing 2 standard deviations. Plots of the concentrations versus time and tables summarizing the DFs are provided in Appendix B. The presence of glycolate has the most significant adverse effect on Sr removal by MST. The impact is a kinetic effect, where the removal of ^{85}Sr is inhibited in the presence of glycolate. After 1 week of contact the percent removed is the same within error; however, at the earlier time points, there is less removal in the presence of either 5000 or 10,000 ppm glycolate. The Pu removal kinetics also appear to be slowed in the presence of 10,000 ppm glycolate. The percent removal values at 6 and 12 hours are lower in the 10,000 ppm glycolate, compared to the 0 and 5000 ppm glycolate simulants. There is no measurable impact to the Pu removal in the presence of 5000 ppm glycolate. At the later time points, 24 and 168 h, the percent removal is the same across all glycolate concentrations. The pre-contacting of glycolate with the MST did not appear to have a noticeable effect on the Sr and Pu removal performance when compared to tests having the same concentration of glycolate in the simulant.

In contrast to the ^{85}Sr and Pu results, the presence of glycolate resulted in an increase in the removal of Np with MST when compared to the simulant without glycolate. Higher removal was seen in the 5000 ppm glycolate solution when compared to the 10,000 ppm glycolate solutions; however, the presence of 10,000 ppm glycolate still resulted in greater Np removal compared to the absence of glycolate. Again, the effect of glycolate appears to be a kinetic effect, in this case the presence of glycolate is increasing the Np removal rate. The pre-contacting of glycolate with the MST slowed the removal of Np compared when compared to tests having the same concentration of glycolate in the simulant. The presence of glycolate had no measurable effect on the removal of U by MST.

The fact that the Np removal was accelerated in the presence of glycolate while the Sr and Pu removal was inhibited suggests differing mechanisms for the different species. Based on these results it is not likely that surface fouling of the MST is the responsible mechanism for the decrease in Sr DF, as one would expect all sorbates to be impacted in that case. A decrease in Sr removal rate is not unexpected since calculations suggest that glycolate can complex Sr^{2+} to a limited degree under alkaline conditions (see Appendix A, Figure A-4). Glycolate-complexed strontium would be expected to be less likely to be adsorbed by MST than the free Sr^{2+}. Calculations (provided in Appendix A) suggest that minimal complexation of the actinides occurs in strongly alkaline solutions. However, some degree of complexation is suggested since we observe slower removal of plutonium in the presence of glycolate. We do not have an explanation for the enhanced rate of neptunium removal in the presence of glycolate.

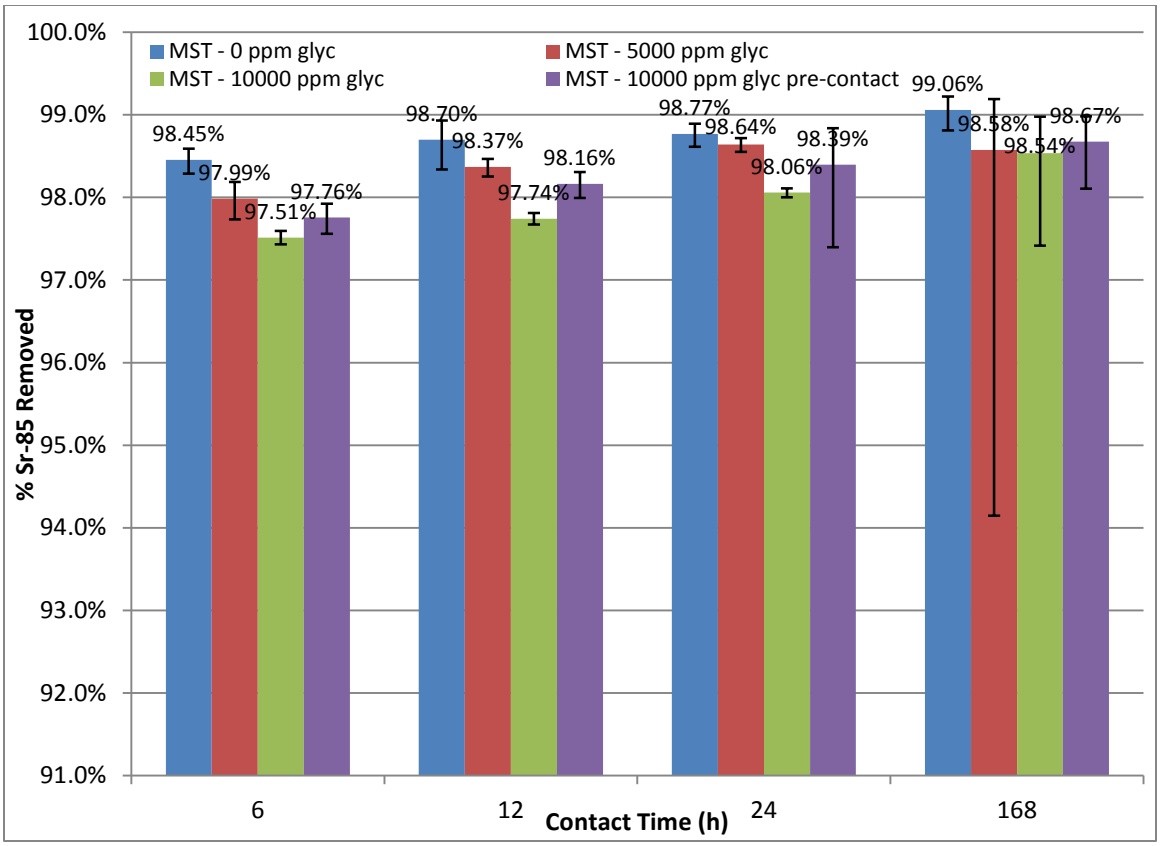

Figure 3-1. Percentage of ^{85}Sr removed versus contact time with MST in the presence of 0 ppm (blue), 5000 ppm (red), and 10,000 ppm glycolate in solution (green) or pre-contacted with MST (purple).

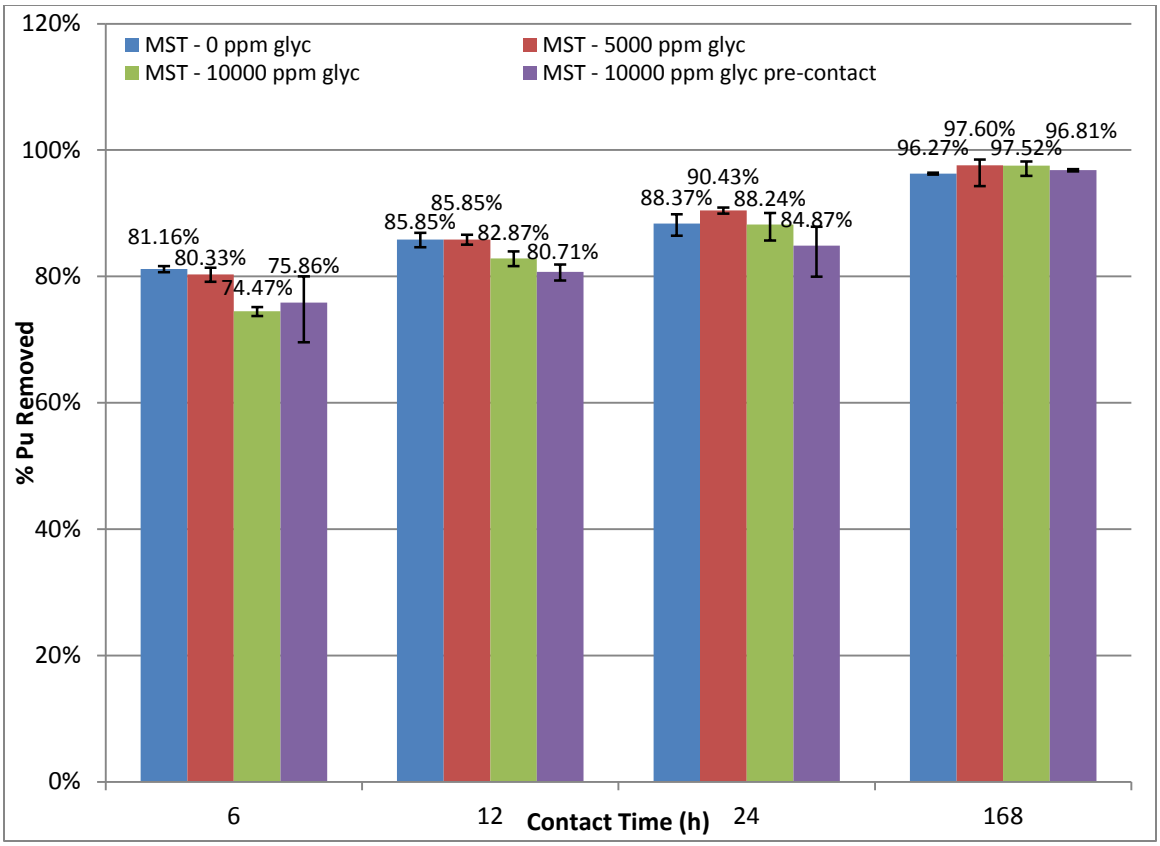

Figure 3-2. Percentage of Pu removed versus contact time with MST in the presence of 0 ppm (blue), 5000 ppm (red), and 10,000 ppm glycolate in solution (green) or pre-contacted with MST (purple).

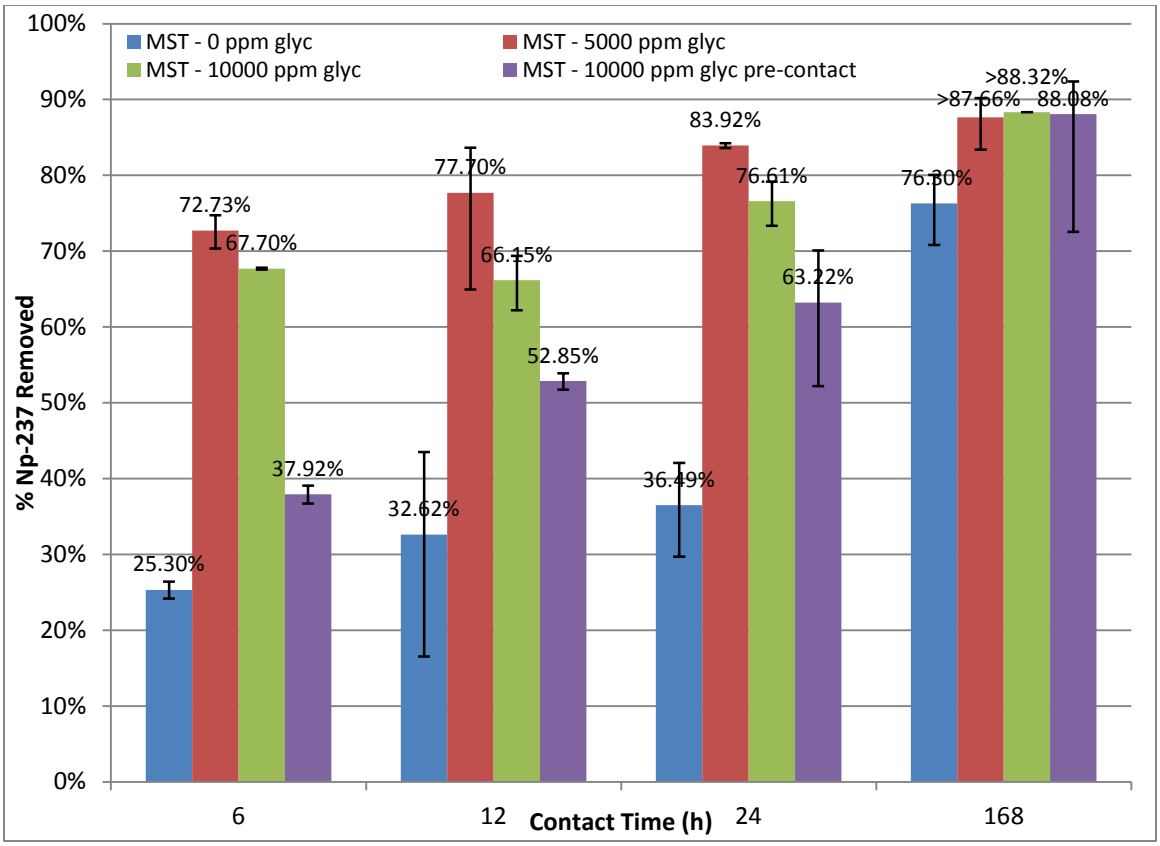

Figure 3-3. Percentage of Np removed versus contact time with MST in the presence of 0 ppm (blue), 5000 ppm (red), and 10,000 ppm glycolate in solution (green) or pre-contacted with MST (purple).

3.3 mMST Performance

Figures 3-6 through 3-8 show the percent removal of ^{85}Sr, Pu, and Np as a function of time for sorption tests performed with mMST in simulants with glycolate concentrations of 0 ppm, 5000 ppm, and 10,000 ppm (both in solution and pre-contacted with the mMST). Plots of the concentrations versus time and tables summarizing the DFs are provided in Appendix B. In contrast to what was observed with MST, the presence of glycolate appears to slow the removal of all three sorbates by mMST.

In the case of ^{85}Sr, at the 6-24 hour time points the percentage of ^{85}Sr removed is lower in the presence of either 5000 or 10,000 ppm glycolate compared to the glycolate free simulant; however, by the 168 hour time point there is overlap of the error bars for all three glycolate concentrations. There was a much larger spread in the Np data between the duplicate trials, resulting in larger error bars making comparison more difficult. However, the presence of glycolate appears to also slow the removal of Np. There is some overlap of the 0 ppm and 5000 ppm data at all the time points, except for 24 hours. The 10,000 ppm samples are consistently lower than the data in the absence of glycolate; however, by 168 hours the 10,000 ppm glycolate solution has reached the percent removal achieved in the 0 ppm glycolate solution at 6 hours, indicating a kinetic effect, rather than reduced capacity of the material.

The effects were less pronounced for Pu. There is no measurable decrease in Pu removal in the presence of 5000 ppm glycolate compared to the glycolate free simulant. There is a small

decrease in the percentage of Pu removed in the presence of 10,000 ppm glycolate through 24 hours; however, at the 168-h time point the Pu concentration is below the method detection limit for all three glycolate concentrations, resulting in greater than values for the percent removed. The presence of glycolate did not change the lack of affinity of mMST for U.

Even though the glycolate had a greater impact on the mMST performance, the material still outperforms the baseline MST for Sr and Pu removal. Even in the presence of 10,000 ppm glycolate the Pu DF for mMST is still much greater than that of MST, and for ^{85}Sr, the 168-h DF for mMST in the presence of 10,000 ppm glycolate was similar to the DF for MST in the absence of glycolate. For Np, the mMST 6 – 24 hour DFs in the presence of 10,000 ppm glycolate were similar to the MST DFs in the absence of glycolate. The 168-h Np DF for mMST was about 40% of the MST 168-h Np DF. As with MST, the pre-contacting of glycolate with the mMST did not appear to have a significant effect on the performance when compared to tests having the same concentration of glycolate in the simulant. Based on these findings, we conclude that the presence of 5000 and 10,000 ppm glycolate slows removal by complexing the sorbates to a limited degree and not by depositing or forming a film on the surface of the mMST.

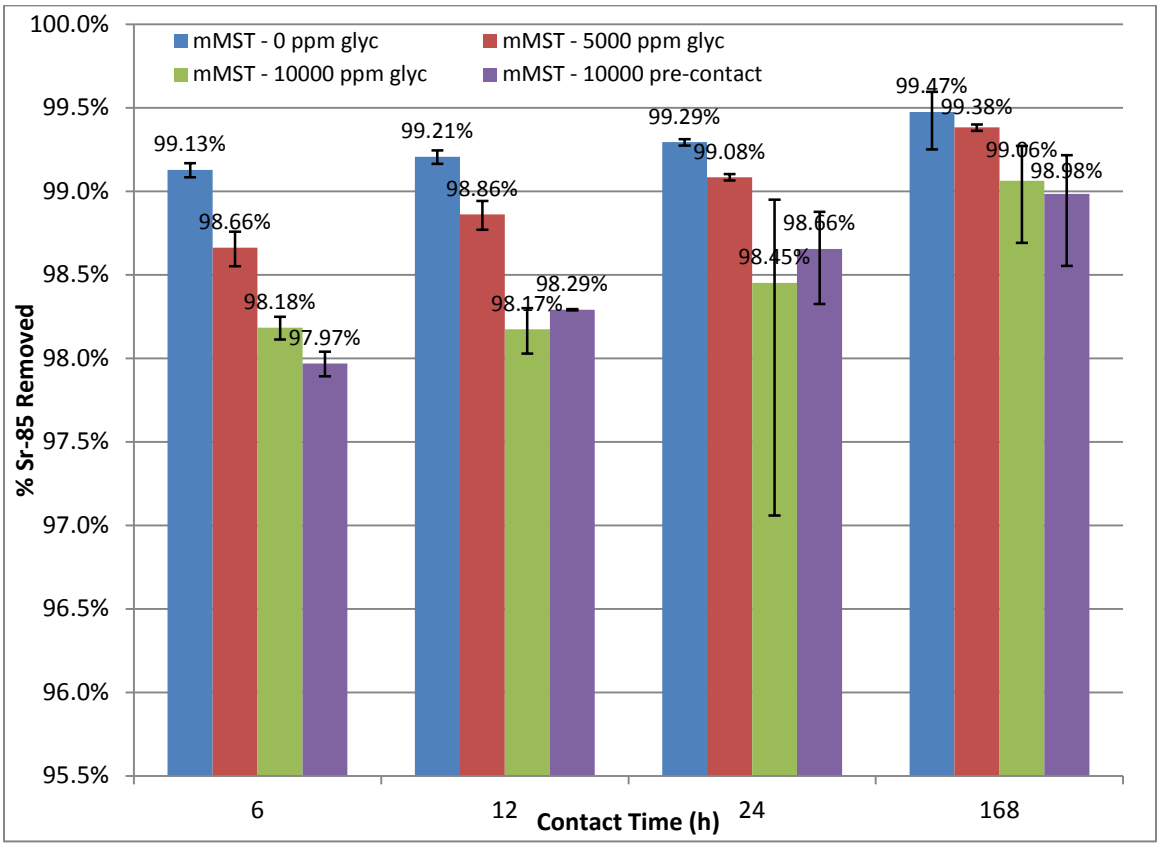

Figure 3-4. Percentage of ^{85}Sr removed versus contact time with mMST in the presence of 0 ppm (blue), 5000 ppm (red), and 10,000 ppm glycolate in solution (green) or pre-contacted with mMST (purple).

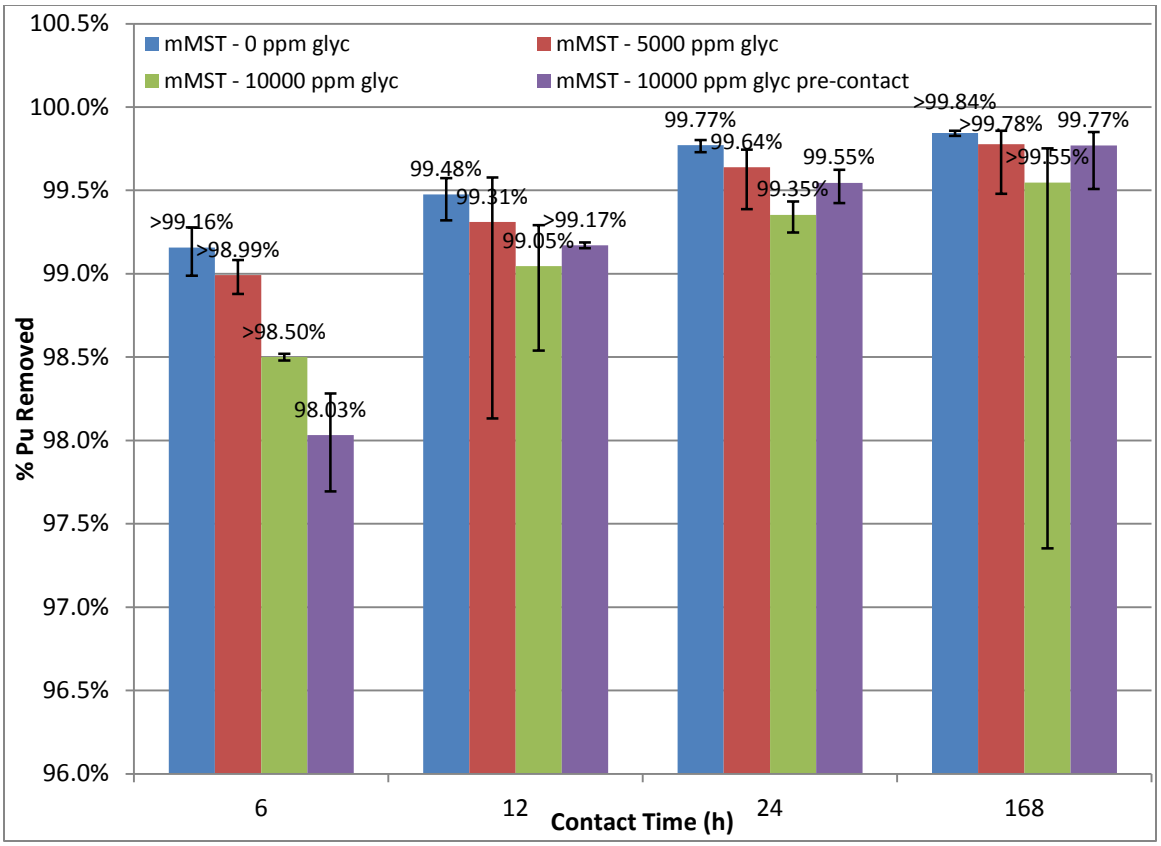

Figure 3-5. Percentage of Pu removed versus contact time with mMST in the presence of 0 ppm (blue), 5000 ppm (red), and 10,000 ppm glycolate in solution (green) or pre-contacted with mMST (purple).

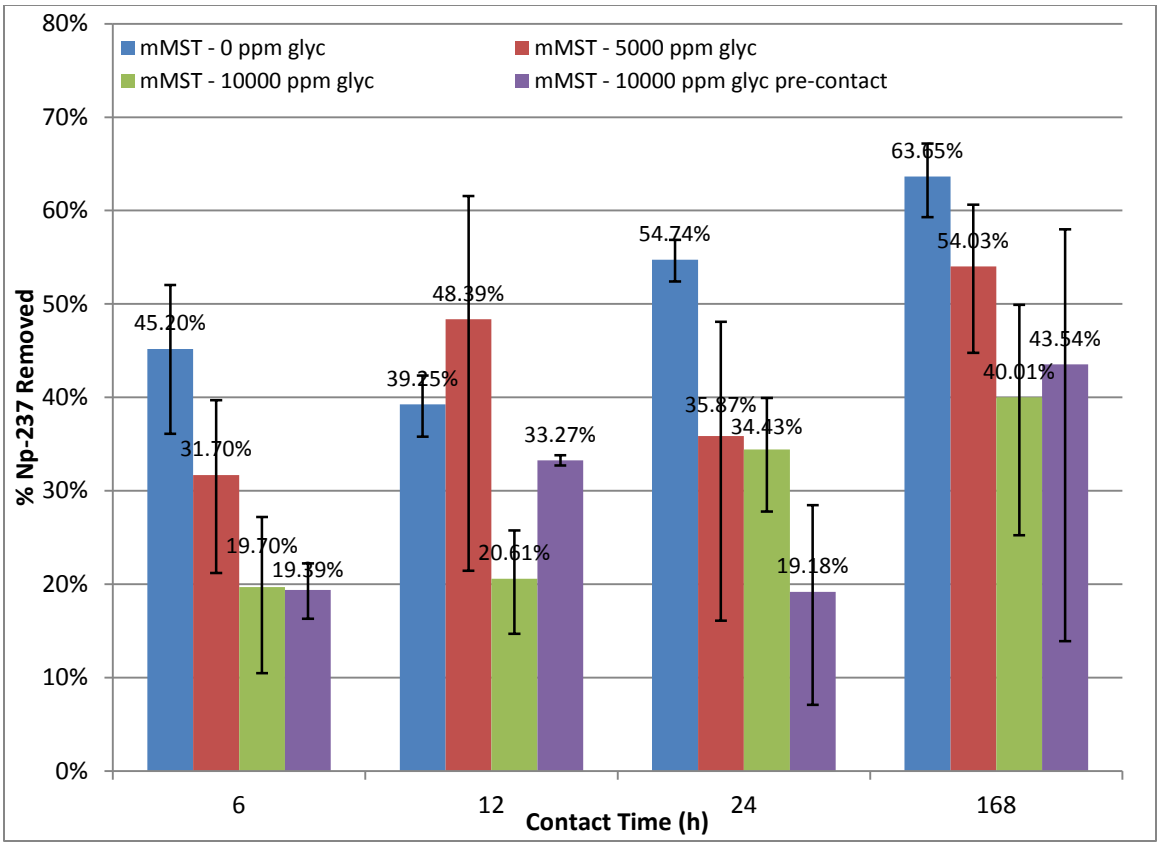

Figure 3-6. Percentage of Np removed versus contact time with mMST in the presence of 0 ppm (blue), 5000 ppm (red), and 10,000 ppm glycolate in solution (green) or pre-contacted with mMST (purple).

3.4 Glycolate Effects on MST and mMST

Measurement of the soluble Ti concentration in the test bottles from the MST and mMST sorption testing in the presence of glycolate showed a slight increase, which correlated with glycolate concentration in the simulant, with the exception of the pre-contact tests. However, the maximum soluble Ti concentration measured represents only approximately 3% dissolution of the MST (or mMST). The measured concentrations are provided in Figures 3-7 and 3-8. It should also be noted that previous studies have shown an increase in Ti leaching from MST and mMST with increased free hydroxide concentrations.[13] This sorption testing was performed with a simulant having a free hydroxide concentration of 1.4 M. Therefore, a combination of higher hydroxide concentrations in ARP and the presence of glycolate could lead to higher concentrations of leached Ti. The leaching of Ti could lead to later precipitation of Ti containing solids in MCU.

Measurements of the glycolate concentrations in the supernate after sorption testing was complete indicated loss of glycolate from the solution. However, the maximum loss was observed in the control samples, indicating precipitation of the glycolate or sorption onto the test bottles, rather than sorption onto the MST and mMST. The final concentrations are shown in Table 3-2.

Particle size analysis of the MST and mMST solids from the sorption testing was also completed, and the volume distributions are shown in Figures 3-9 and 3-10. For MST, the volumetric distribution was found to shift slightly to the left (smaller particle size) as the glycolate

concentration was increased. The volume average shifted from 8.3 µm in the absence of glycolate to 3.9 µm in the presence of 10,000 ppm glycolate. The fraction of particles below 0.8 micron increased slightly from 1.99 vol % in the absence of glycolate to 2.33 vol % in the presence of 10,000 ppm glycolate. However, these values are well within the normal particle sizes seen for the current supplies of MST for ARP.[14] In addition, the geometric standard deviations for all MST samples were also within the range seen for the current MST supplies.[11] In contrast very little change was observed in the mMST samples in the absence or presence of glycolate. The volume average particle size ranged from 4.1 µm in the absence of glycolate to 4.3 µm in the presence of 10,000 ppm glycolate.

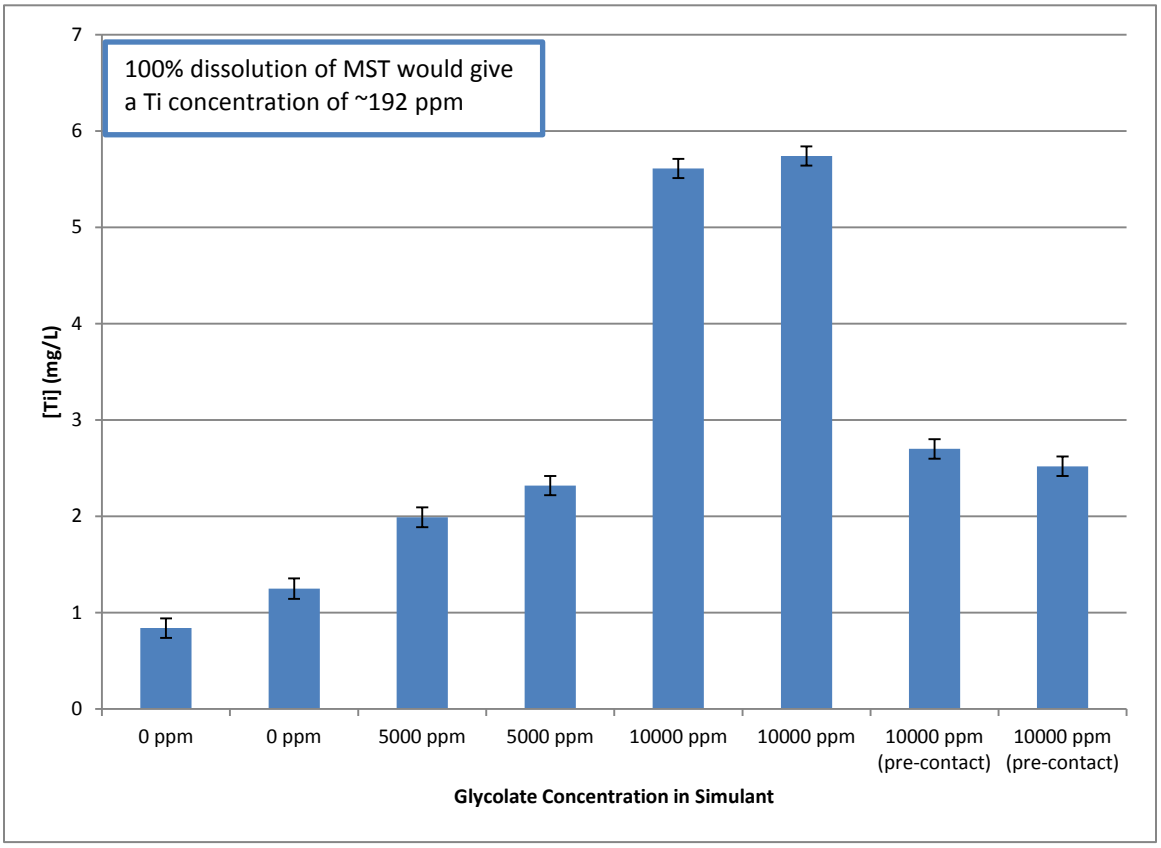

Figure 3-7. Measured soluble Ti concentration in supernate from MST sorption tests.

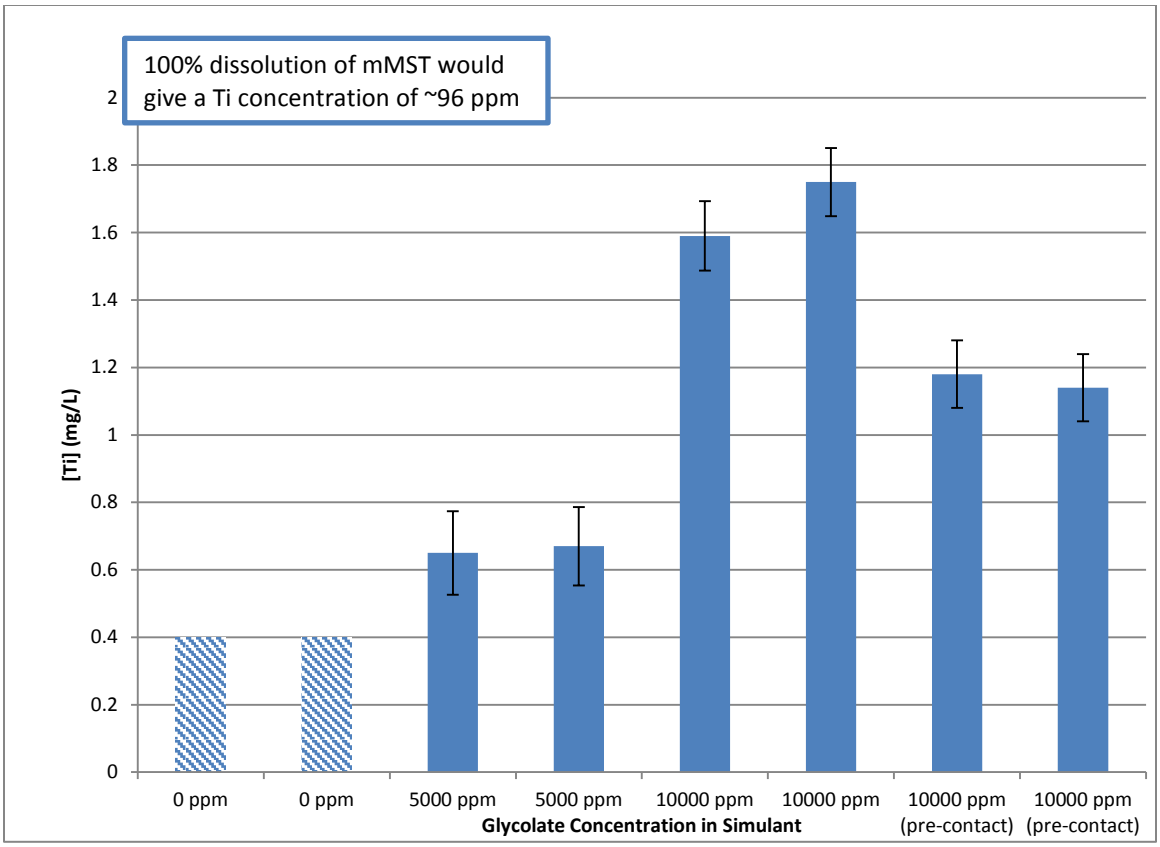

Figure 3-8. Measured soluble Ti concentration in supernate from mMST sorption tests. The Ti concentrations in the 0 ppm glycolate solutions are less than values.

Table 3-2. Final glycolate concentrations in supernate from sorption tests.

Test ID	Sorbent	Initial Glycolate Concentration (ppm)	Measured Final Glycolate Concentration (ppm)
GlycMST-6	none (control)	4790	3530
GlycMST-7	MST	4790	4710
GlycMST-8	MST	4790	5100
GlycMST-9	mMST	4790	4200
GlycMST-10	mMST	4790	4200
GlycMST-11	none (control)	10,700	6280
GlycMST-12	MST	10,700	9700
GlycMST-13	MST	10,700	7000
GlycMST-14	mMST	10,700	7190
GlycMST-15	mMST	10,700	7000
GlycMST-16	none (control)	10,000 (target)	4000
GlycMST-17	MST	10,000 (target)	6100
GlycMST-18	MST	10,000 (target)	5400
GlycMST-19	mMST	10,000 (target)	5640
GlycMST-20	mMST	10,000 (target)	5660

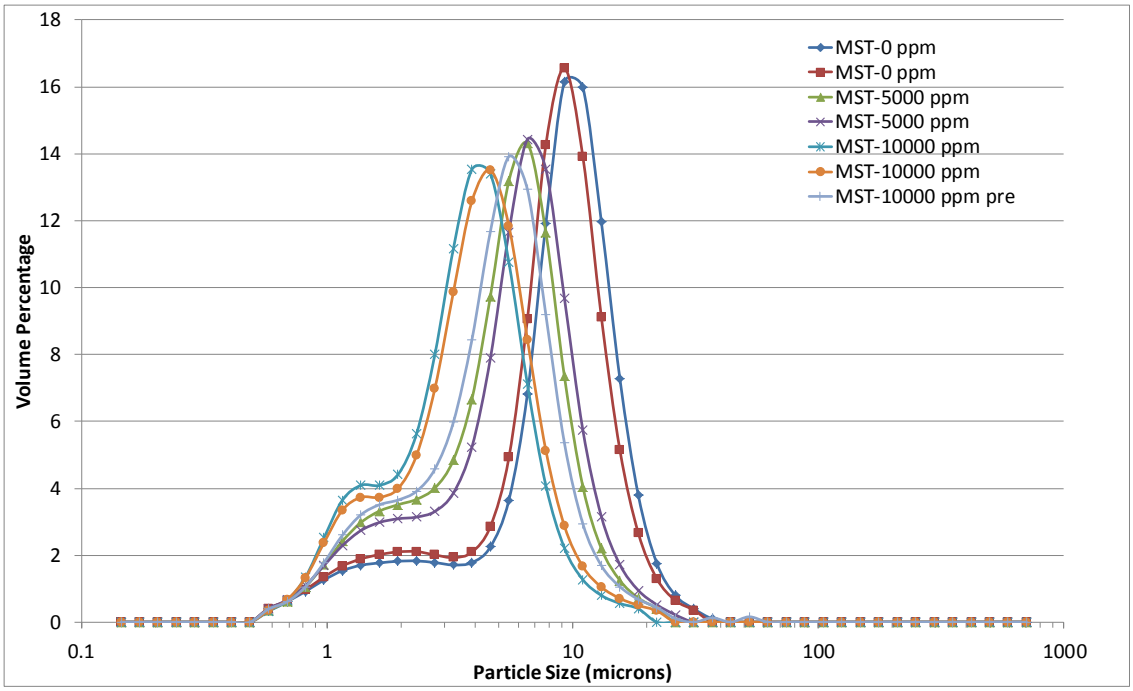

Figure 3-9. Volume based particle size distribution for MST from sorption tests containing various amounts of glyclate in the simulant.

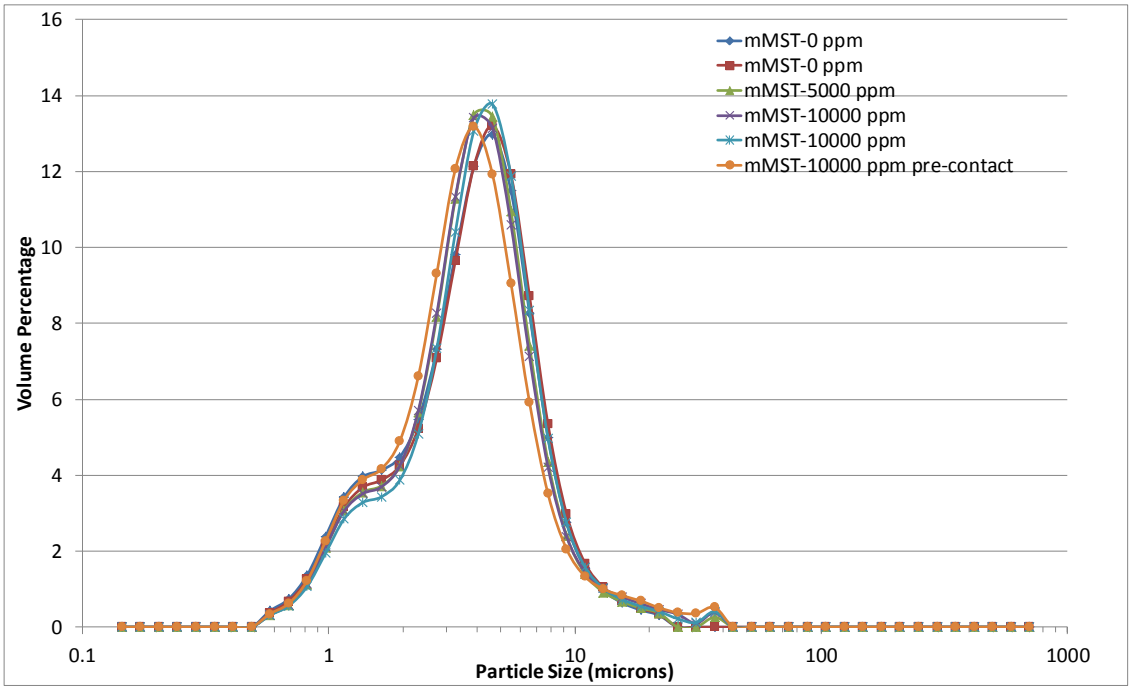

Figure 3-10. Volume based particle size distribution for mMST from sorption tests containing various amounts of glyclate in the simulant.

In addition to measurement of the soluble Ti and glycolate concentrations at the conclusion of the sorption testing, iodometric titrations were performed to examine the effect of glycolate on the peroxide content of mMST. The results are provided in Table 3-3. As can be seen from the

results, the peroxide to Ti molar ratios of mMST before and after exposure to glycolate are identical, indicating that the peroxide groups on mMST are not consumed by reaction with glycolate.

Table 3-3. Peroxide:Ti molar ratios in mMST before and after exposure to glycolate (65.1 g glycolate/g mMST).

	Ave. Peroxide:Ti molar ratio
mMST (Control)	0.272 ± 0.008
mMST w/glycolate	0.273 ± 0.002

3.5 Interaction of Glycolate with MST Examined by FTIR

Examination of glycolate on the MST and mMST solids isolated from the sorption tests proved difficult due to the presence of high concentrations of salt. The sample preparation method led to the drying of salt on the MST solids after removal of the supernatant salt solution. These salts prevented the FTIR from detecting any glycolate on MST. The spectra are shown in Figure 3-11, compared to a spectrum of MST exposed to sodium glycolate at pH 5, where the presence of glycolate can be identified. As can be seen in Figure 3-11, the peaks associated with sorbed glycolate at 1100 and 1590 cm^{-1} (bottom spectrum in this figure) are not clearly seen in the remaining spectra. The glycolate peaks are convoluted with other peaks associated with the remaining salts from the salt solution on MST.

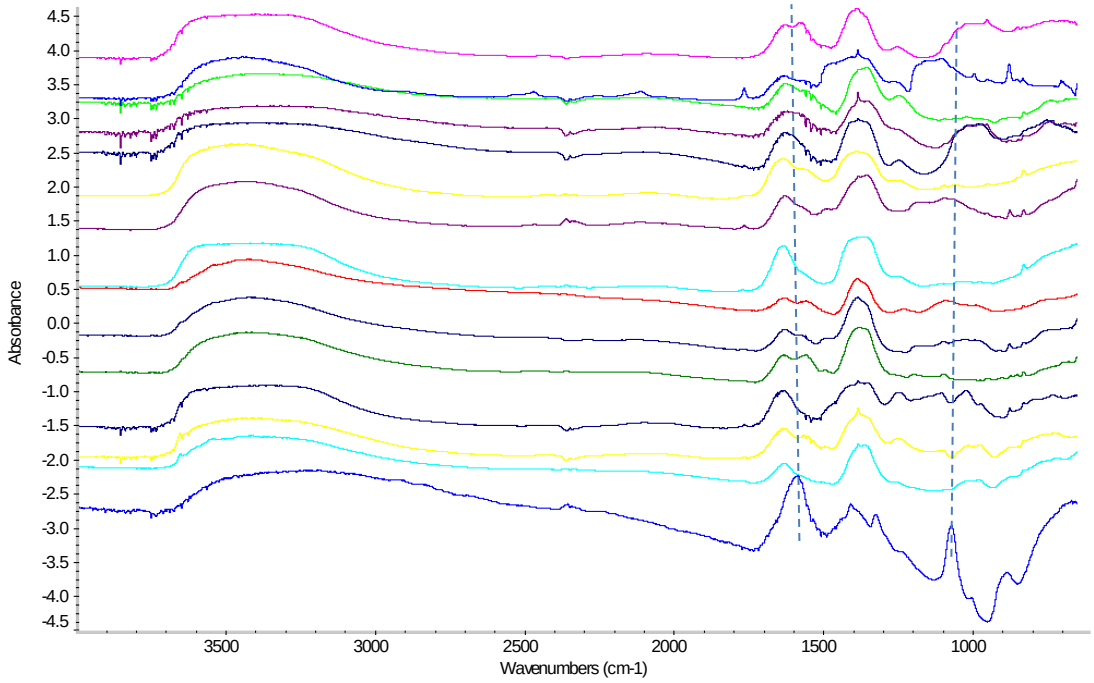

Figure 3-11. FTIR Spectra of MST exposed to various solutions. The bottom spectrum is from MST exposed to a solution containing 10,000 ppm sodium glycolate at pH 5. From bottom to top, the remaining spectra are from solids isolated from Tests 2-19 (increasing glycolate concentration).

In a separate set of tests, MST was exposed to solutions of varying pH containing 10,000 ppm sodium glycolate. As can be seen in Figure 3-12, the IR spectra of the MST exposed to sodium glycolate aqueous solutions at pH 3 and 5 does contain sodium glycolate. The bottom spectrum in Figure 3-12 is a solution of sodium glycolate for comparison. The sodium glycolate sorption onto MST at low pH was reversible and could be removed by washing with caustic solution. No evidence of glycolate adsorption on MST was observed when the aqueous solution had pH of 7 or higher. Since the pK_a of glycolic acid is 3.83, at pH 3 we expect some of the glycolate to be present as glycolic acid. Similarly, the isoelectric point of MST has been measured to be at a pH of 4.46.[15] At pH 3, both glycolic acid and MST are expected to have a significant fraction of the molecules in the protonated form (COOH and Ti-OH respectively). Under this condition, sorption could be enhanced by hydrogen bonding between the oxygen and hydrogen atoms of both MST and glycolic acid. At pH 7 or higher, both MST and glycolic acid would be expected to be present as the anionic form (i.e., non-protonated), and therefore, adsorption would be minimal due to electrostatic repulsion. It was expected that some of the titanium atoms located in a pentahedral configuration (valence state of +3 or +5) at the surface and could interact with the anionic glycolates. The data observed here appears to indicate this adsorption is not strong enough to keep glycolate atoms on the surface of MST after washing MST with caustic solution. Figure 3-12 shows that the peaks associated with glycolate (1100 and 1590 cm^{-1}) are not seen on the surface of MST when the solution pH is greater than 7.

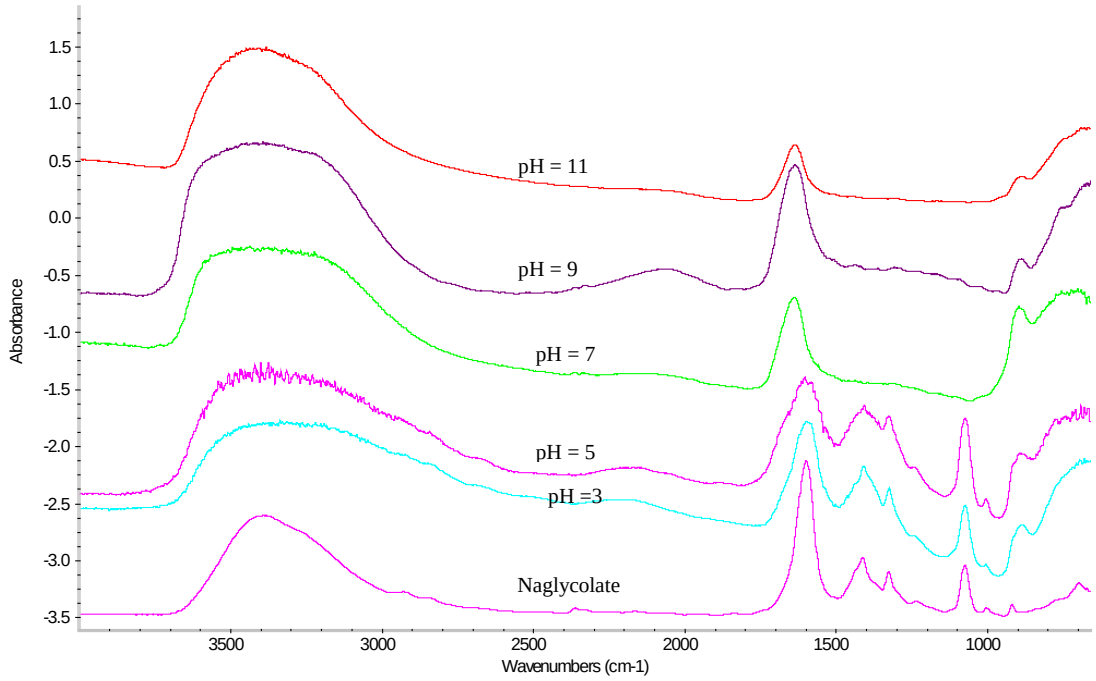

Figure 3-12. FTIR spectra of MST exposed to 10,000 ppm sodium glycolate solutions of varying pH. The bottom spectrum is of a solution of sodium glycolate for comparison.

3.6 Glycolate Effects on Cesium Removal (MCU Solvent)

Table 3-4 shows the results from the MCU solvent ESS Tests, corrected to the normal process operating temperatures (i.e., 23 ºC for extraction and 33 ºC for scrubbing and stripping).

Table 3-4. Cesium Distribution Values for the ESS Tests (MCU Solvent)

Material	Extraction	Scrub #1	Scrub #2	Strip #1	Strip #2	Strip #3
Reference Case (Expected Values)	>8	>0.6, <2	>0.6, <2	<0.2	<0.16	<0.16
0 ppm glycolate	19.3	2.23	1.47	0.0350	0.0270	0.0294
5000 ppm glycolate	148	1.93	1.79	0.0390	0.0240	<0.0194
10,000 ppm glycolate	18.8	1.80	1.65	0.0470	0.0216	<0.0223

All three tests gave acceptable values for all steps, with the exception of Scrub #1 for the 0 ppm test (blank). The slight deviation from the acceptable range is commonly seen and is not considered to be problematic.

From the bulk chemistry of the solutions, an extraction DF of ~17.1 is predicted.[16] In the 5000 ppm test, the Extraction #1 test point gave a result that is clearly impossible. Through the use of variable sensitivity analysis, SRNL believes that this value is due to an unanticipated dilution in the aqueous Extraction #1 analytical sample. Nevertheless, even with this unresolved data, there is no indication that the presence of 5000 ppm of glycolate affects the cesium removal behavior.

3.7 Dispersion Testing with MCU Solvent

For each of the organic and aqueous phases, two trials were performed. The resulting dispersion value is given as a unit-less value, with the value in parenthesis being the %RSD (see Table 3-5). It is generally considered that the typical analytical uncertainty associated with a dispersion test is 25%.[17] Given this, the differences between the three sets of tests are not different enough to declare that glycolate has a negative impact on phase disengagement. If anything, the glycolate appears to have a beneficial trend (i.e., more rapid phase disengagement) as evidenced by slightly higher dispersion values compared to that without glycolate present.

Table 3-5. Dispersion Results With MCU Solvent and Glycolate

Organic Phase	Aqueous Phase	Dispersion Value
MAX Solvent "D"	Salt Simulant + 0 ppm glycolate	9.27E-04 (14.3%)
MAX Solvent "D"	Salt Simulant + 5000 ppm glycolate	1.03E-03 (1.06%)
MAX Solvent "D"	Salt Simulant + 10,000 ppm glycolate	1.20E-03 (3.06%)

3.8 Glycolate Effects on Cesium Removal (NGS Solvent)

Table 3-6 shows the results from the MCU NGS solvent ESS tests, corrected to the normal process operating temperatures (i.e., 23 ºC for extraction and 33 ºC for scrubbing and stripping).

Table 3-6. Cesium Distribution Values for the ESS Tests (NGS Solvent)

Material	Extraction	Scrub #1	Scrub #2	Strip #1	Strip #2	Strip #3
0 ppm glycolate	176	24.8	12.3	0.00476	0.00220	0.0446
5000 ppm glycolate	185	24.7	23.3	0.0333	0.00334	0.0158
10,000 ppm glycolate	176	25.3	19.2	0.0352	0.00342	0.00720

All three tests gave similar results. Thus we conclude that there is no meaningful difference in the control and the two glycolate-spiked tests. The extraction and scrub values are far higher than typical (~70 and ~2.5, respectively) which SRNL attributes to the relatively dilute cesium spike in the aqueous phase (i.e., the extraction kinetics or affinity may be larger at the more dilute cesium concentration than used in prior tests).

3.9 Coalescer Adherence and Performance

Microscopic examination of CSSX solvent drops on both as-received coalescer media exposed to dispersed CSSX solvent with sodium glycolate and irradiated coalescer media exposed to dispersed CSSX solvent with sodium glycolate reveals no major differences in droplet size before detachment or wetting angle (at the early stages). As Figure 3-13 shows, global examination shows no obvious difference in the coalescing behavior that can be attributed to glycolate.

Evaluation of the fibers by infrared spectroscopy reveals the adsorption of glycolate on the irradiated coalescer and the adsorption of modifier on both the as-received and irradiated coalescer as shown in Figure 3-14.

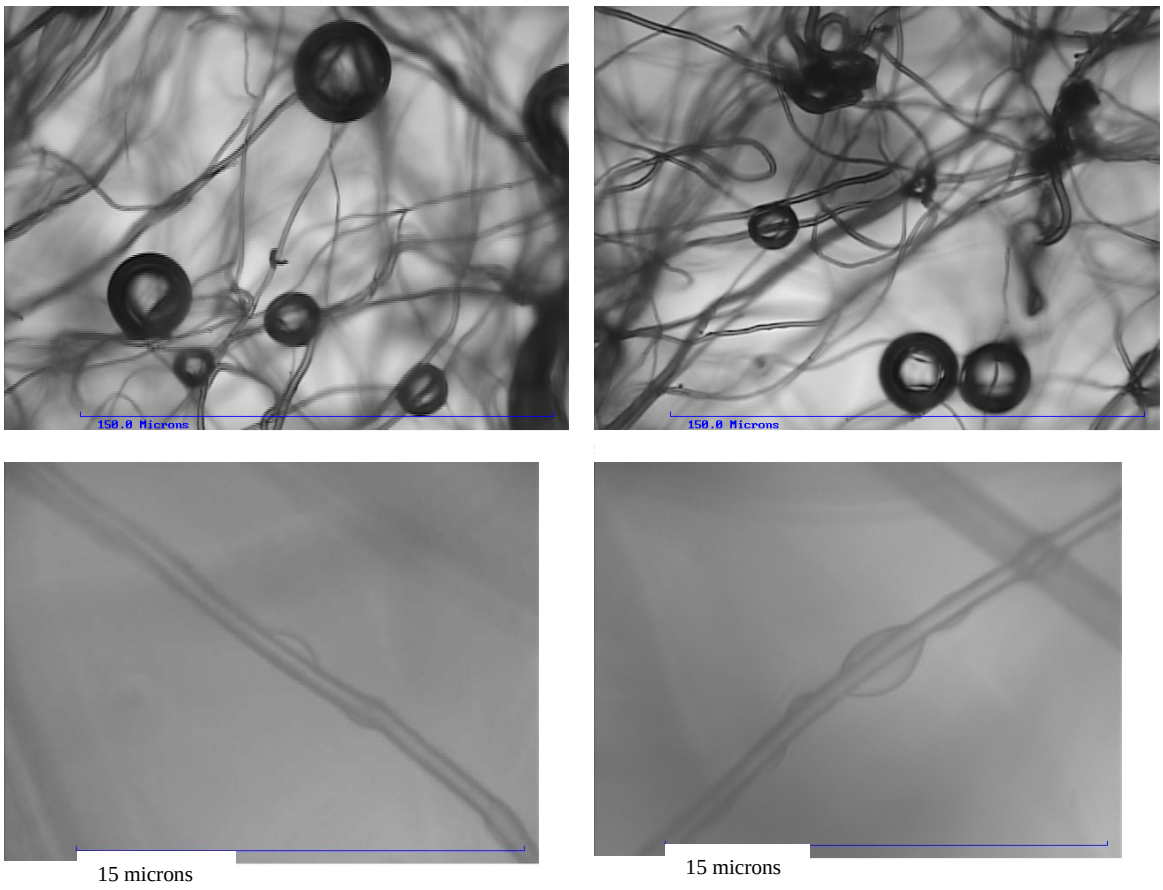

Figure 3-13. The top pictures were obtained at 20X while the bottom pictures were obtained at 100X. CSSX solvent appears to coalesce in similar way in both as-received (left photos) exposed to CSSX solvent dispersed in salt solution and on irradiated coalescer (right photo) exposed to CSSX solvent dispersed in salt solution containing 10,000 ppm sodium glycolate.

As can be seen in Fig. 3-14, glycolate is clearly observed on the fibers of the irradiated coalescer but it does not appear to affect the CSSX solvent coalescing mechanism on the fibers. Given that glycolate on fibers should make them more polar (or oleophobic) and, therefore, provide a lower surface energy for the aqueous solution, that effect is not observed. It appears that the modifier sorbed on the fibers promotes sorbed CSSX solvent droplet coalescing among them by providing a surface that supports cohesive energy (i.e., the energy to split a CSSX solvent droplet into two droplets or to join them). Since CSSX solvent contains modifier, a CSSX solvent droplet on a fiber will readily move and merge with other droplets if the surface is covered with modifier. Once a growing CSSX solvent droplet reaches a critical size (e.g., several times that of the fiber), then buoyancy and drag forces from the hydrodynamics detaches the CSSX solvent droplet (at this large size wetting plays a lesser role in the detachment). To verify this conclusion, a small scale coalescing test must be conducted with the coalescer receiving a CSSX dispersion in salt solution followed by evaluation of the particle size distribution of the dispersion before and after passing through the coalescer. The objective is to determine if glycolate impedes the coalescing function of Ryton.

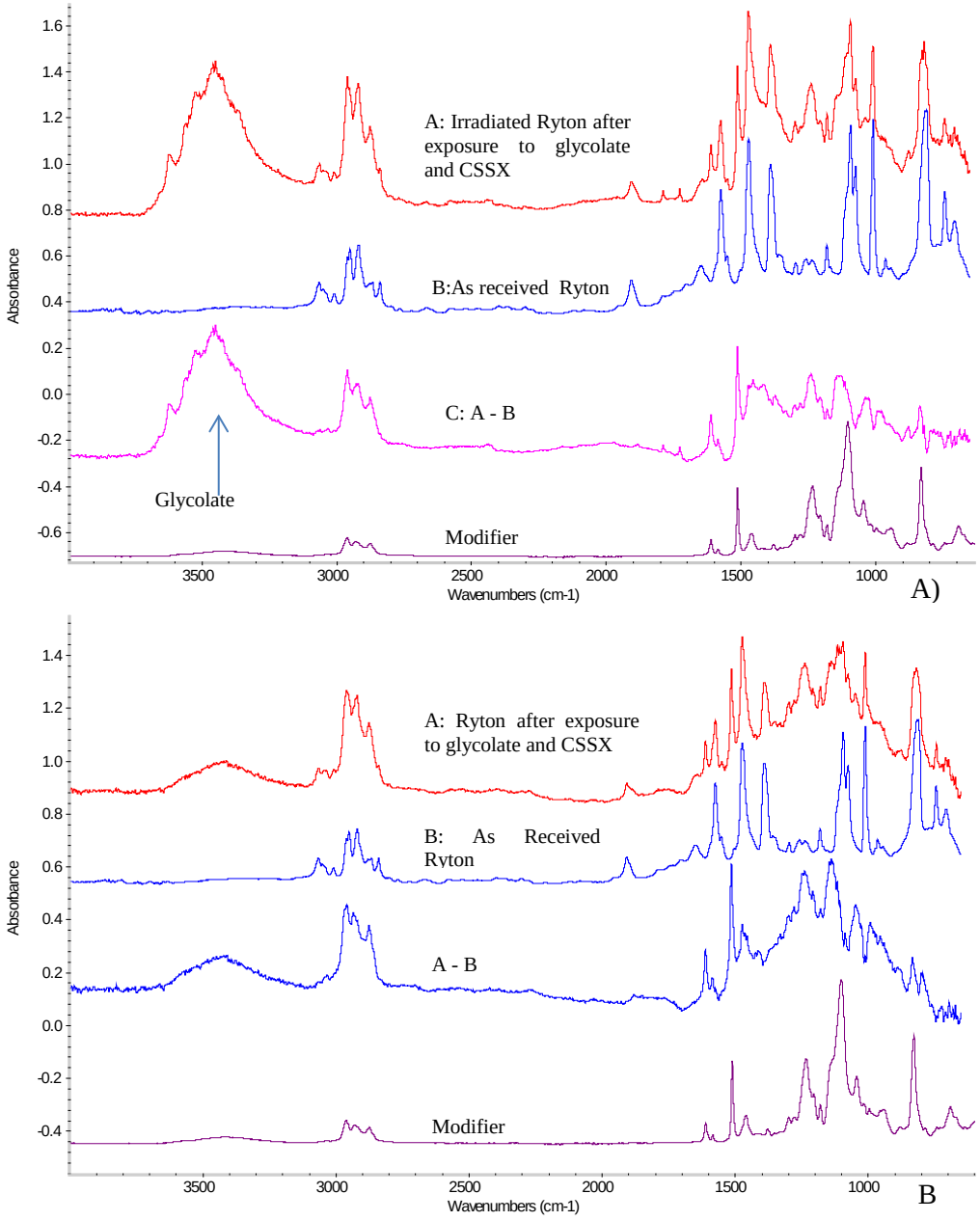

Figure 3-14. Sorption of glycolate and Modifier on as-received and irradiated Ryton fibers. Top figure shows the adsorption of both glycolate and Modifier on irradiated Ryton. The bottom figure shows a strong Modifier adsorption on as-received Ryton.

Since a finite solution volume was fed to the coalescer, the dispersion aggregation rate must be determined to evaluate the coalescing performance. Dispersed samples were evaluated with a turbidity instrument as a function of time. Figure 3-15 shows the turbidity of CSSX solvent dispersed in salt solution without glycolate and CSSX solvent dispersed in salt solution containing 10,000 ppm sodium glycolate. Inspection of the figure shows that the dispersion aggregation rate levels off after five minutes indicating the best time to pump this dispersion to the coalescer. In addition, the rate of CSSX solvent aggregation in salt solution with and without sodium glycolate appears similar. Figure 3-16 provides a log-log plot of the turbidity of the salt

solution with dispersed CSSX solvent versus time. The observed linear relationship indicates the aggregation of CSSX solvent has inverse power law relationship with observed time. This is a further confirmation that both flocculation (aggregation) and coalescing is occurring simultaneously.[18] Note, if a nonlinear correlation of the turbidity had been observed this would allow one to determine if the dominant mechanism was flocculation or coalescing.[19]

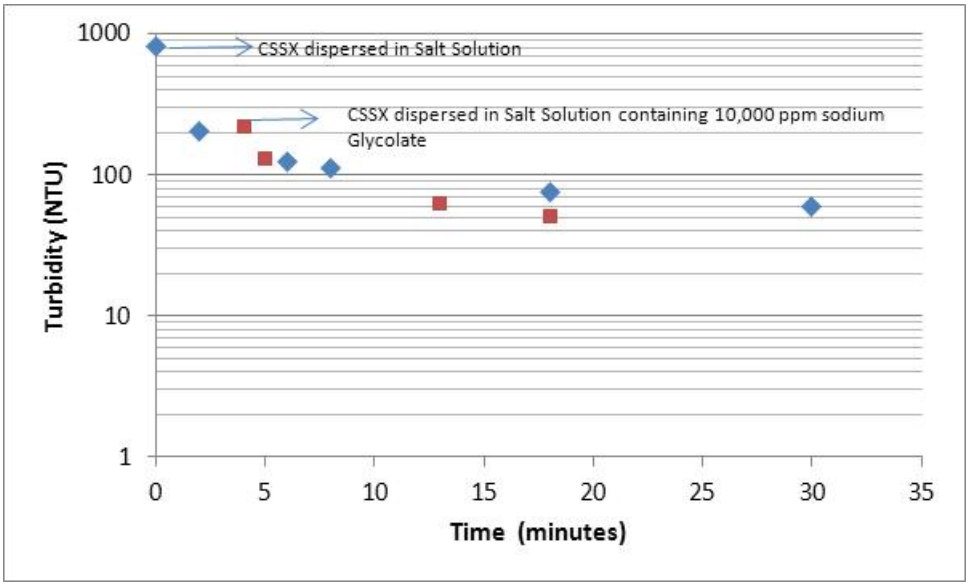

Figure 3-15. Rate of agglomeration of solvent dispersion. Five minutes after preparation the dispersion appears stable. The blue diamonds represent CSSX solvent in salt solution and the red squares represent the same dispersion in salt solution containing 10,000 ppm sodium glycolate.

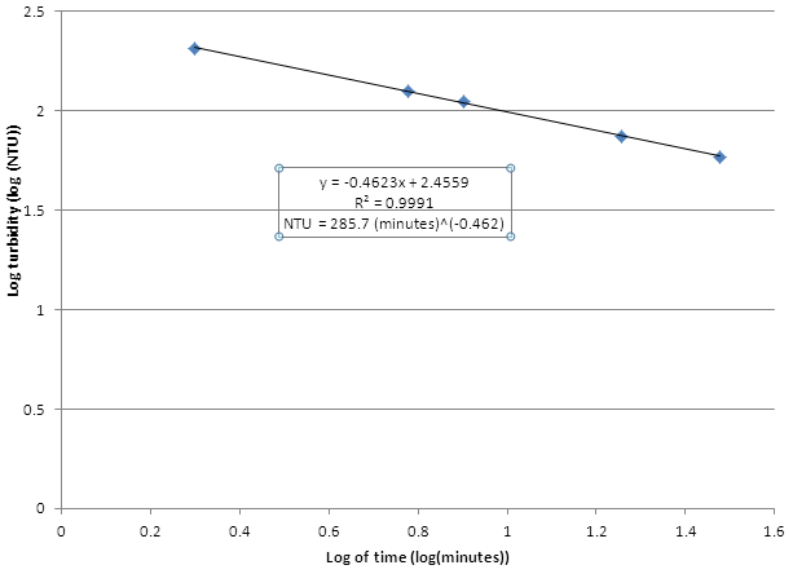

Figure 3-16. Turbidity versus time for solvent coalescence.

Coalescer Test: Particle Size Distribution

The presence of sodium glycolate at the 10,000 ppm level slightly affected the starting volume distribution population of the droplets obtained from homogenizing CSSX solvent in water (see Figure 3-17). In the presence of glycolate there is a small increase in the population of droplets centered at 5.5 and greater than 20 micron compared to the population distribution in the absence of glycolate. This difference is not significant and it is not expected to alter the particle size distribution expected from the mixing region of the centrifugal contactors at MCU.

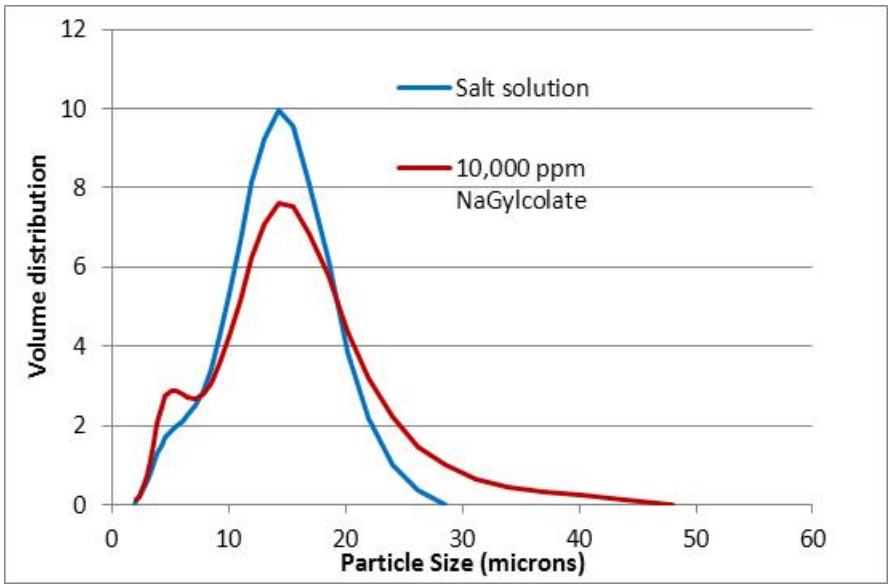

Figure 3-17. The effect of glycolate on the starting volume distribution population obtained from solvent dispersions with the homogenizer spun at 15,000 rpm. Both dispersions were analyzed 15 minutes after they were formed.

Two 1-L salt solutions containing dispersed solvent pulses were passed through the coalescer. As the particle size distribution (volume distribution) indicates any droplet larger than half the Ryton fiber diameter (as long the droplets are not larger than the free space between fibers or 30 microns) is coalesced into a larger droplet and trapped inside the coalescer as shown in Figure 3-18.

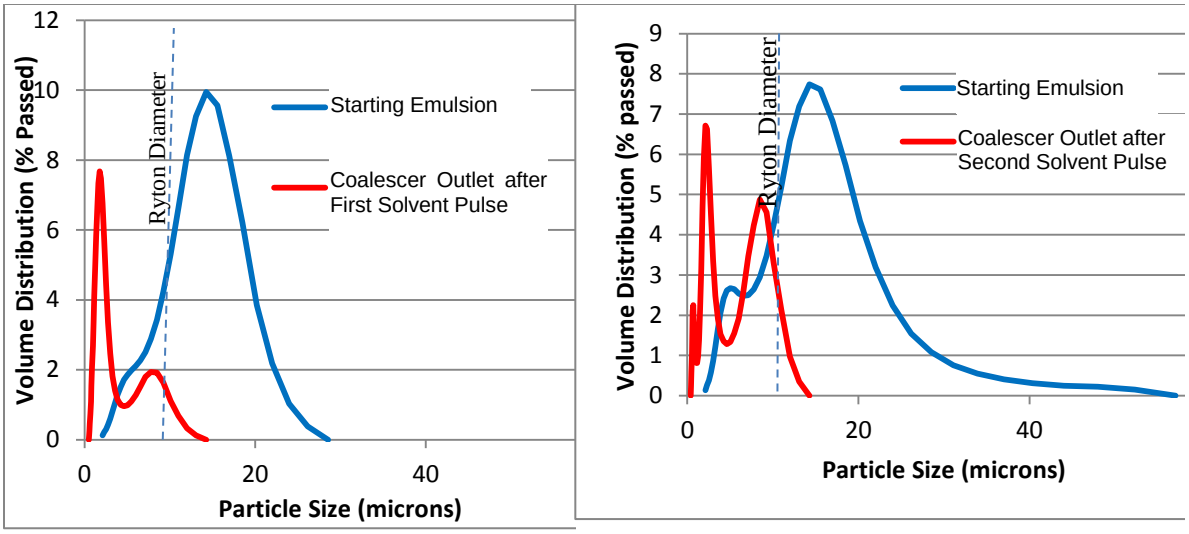

Figure 3-18. Volume distribution of the dispersed solvent phase through the coalescer after two pulses.

Evidence of large droplets that formed from coalescing smaller droplets is shown in Figure 3-19. Figure 3-19 shows the accumulation of large CSSX droplets that account for the missing large droplets in the droplet size distribution of Figure 3-18.

Figure 3-19. Picture of coalesced solvent forming at the bottom of the coalescer. Also shown is the typical turbidity observed 5 minutes and 20 minutes after sending the solution through the coalescer.

The presence of 10,000 ppm sodium glycolate in the salt solution had no effect on the droplet size distribution of dispersed phase that exited the coalescer. As shown in Figure 3-20, pumping three 1-L salt solution dispersions containing 10,000 ppm glycolate pulses through the coalescer appeared to narrow (i.e., sharper kurtosis and skewness) the droplet size distribution of the

dispersed phase. Thus, from a perspective that includes characteristics such as viscosity, surface tension, wettability rate, and coalescence rate, no evidence of glycolate effect on the overall coalescer performance was observed.

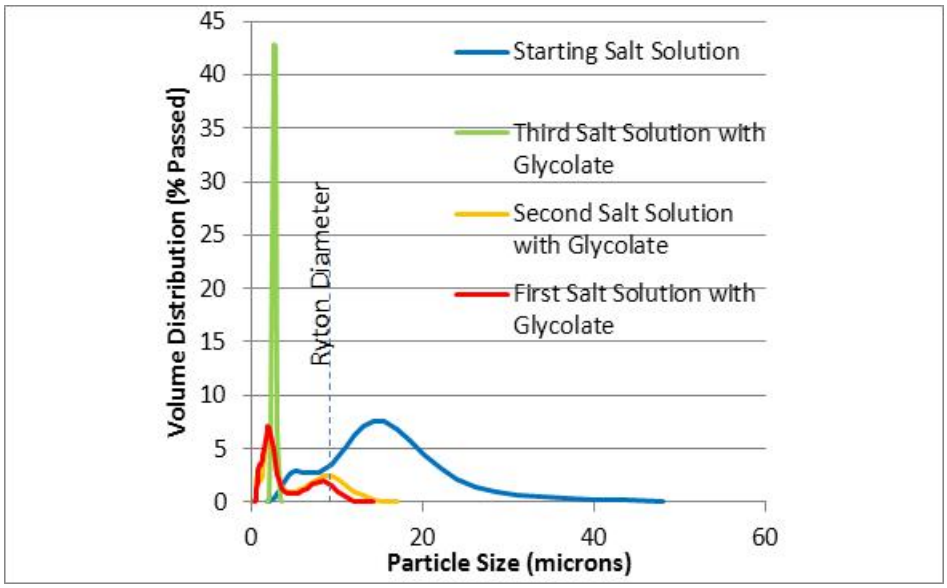

Figure 3-20. The effect of 10,000 ppm glycolate on the droplet size distribution (volume based) of the salt solution that flowed through the coalescer.

The next test used a coalescer that received 8 E6 rad of gamma irradiation. We circulated 1 L of salt solution containing dispersed CSSX solvent as before for approximately 20 minutes and finally, we circulated a salt solution containing dispersed CSSX solvent and 10,000 ppm of sodium glycolate. The starting solution and the salt solution exiting the coalescer 20 minutes after the initiation of the test were submitted for droplet size analysis. As shown in Figure 3-21, the coalescer is coalescing particles larger than half its diameter as previously seen indicating that an aged coalescer (irradiated) is not affected by the presence of a high concentration of sodium glycolate. Note, the concentration of solvent exiting the coalescer could be obtained by taking the ratio of the areas under these curves and multiplying this number by the starting solvent concentration (1 wt % dispersed solvent).

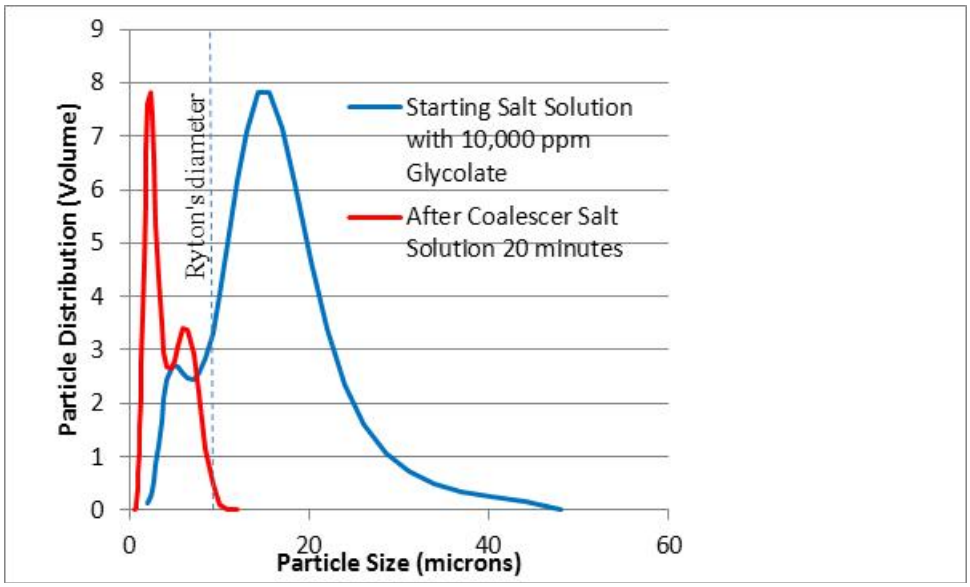

Figure 3-21. The effect of sodium glycolate on a coalescer that received 8 E6 rad.

Glycolate Adsorption on the Coalescer

The salt solutions used in the coalescing test were analyzed by Raman spectroscopy and IC. Figure 3-22 shows the glycolate concentration in the starting salt solution and the salt solution that exited the coalescer at 5 minutes and 20 minutes after the initiation of the test. As seen in Figure 3-22, there is an initial 60% decrease in the glycolate concentration that, despite additional glycolate containing pulses, is reduced to 36%. This is due to dilution with the salt solution that soaked the coalescer before glycolate was introduced. The same solutions were analyzed by Raman spectroscopy by using the C-C stretch band at 920 cm^{-1} normalized to the sulfate band at 1010 cm^{-1} (as shown in Figure 3-23 where a calibration line is shown). As shown in Figure 3-24 and Figure 3-25, the glycolate concentration decreased drastically initially but then it increased with more solution flowing through the coalescer. With the irradiated coalescer the glycolate concentration decrease was half of that of the as-received coalescer indicating that the initial glycolate concentration reduction is not due to sorption on the coalescer but rather to dilution effects with salt solution remaining at the coalescer.

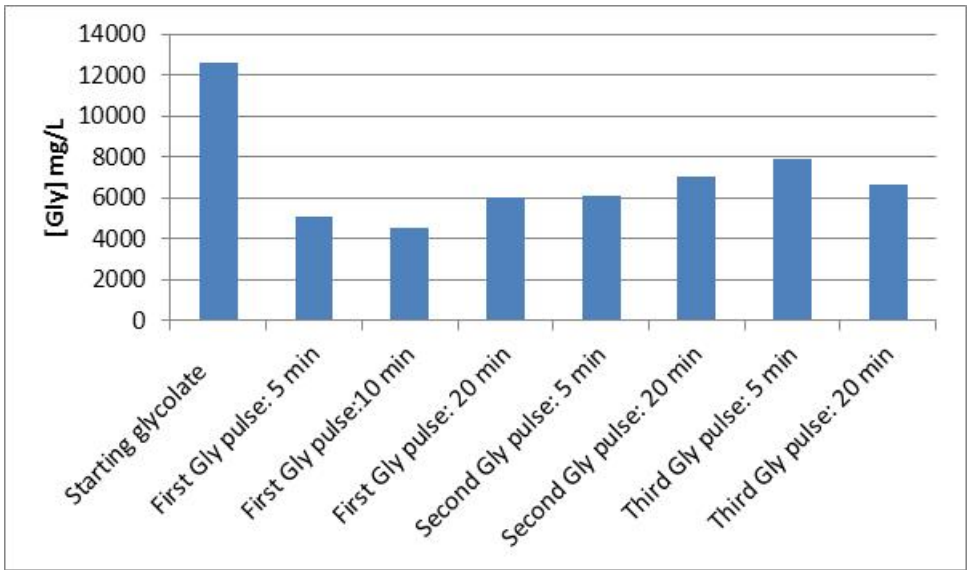

Figure 3-22. Glycolate concentration as measured by ion chromatography in solution after three dispersion pulses containing glycolate (each pulse containing about 12,600 mg/L). Before each glycolate pulse, the coalescer was pulsed with salt solution containing CSSX solvent.

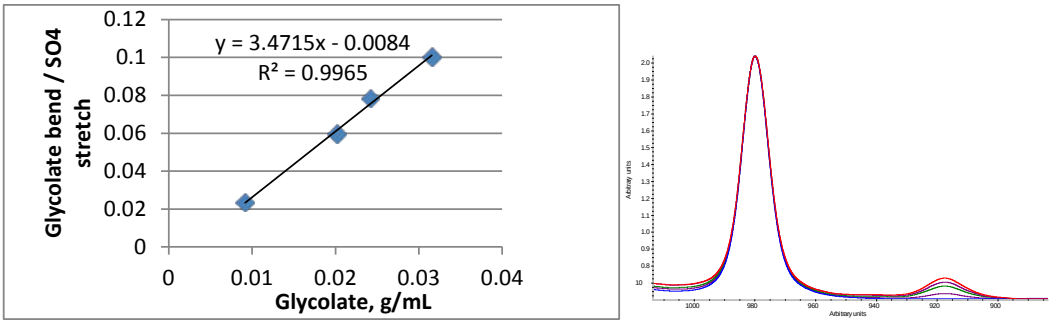

Figure 3-23. Glycolate calibration curved obtained from C-C stretch at 917 cm^{-1}.

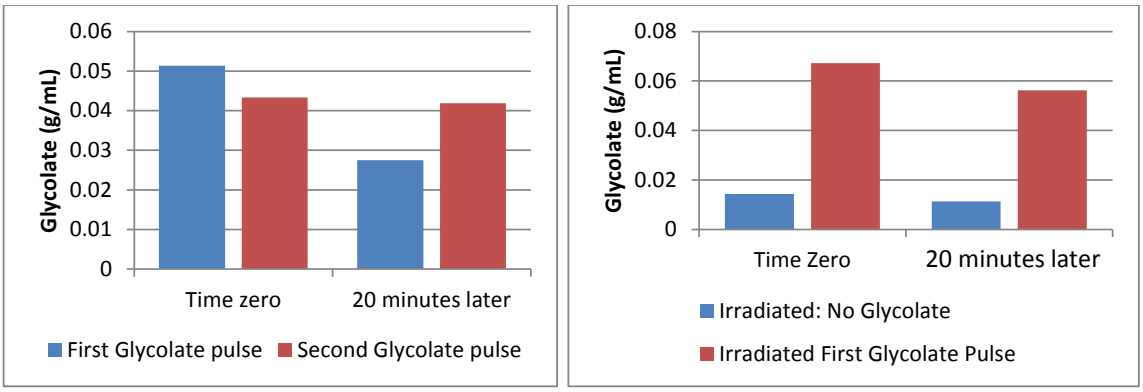

Figure 3-24. Left figure shows the glycolate concentration before and after passing through the coalescer (blue bar first pulse and red bar the second pulse). The figure on the right shows the glycolate concentration before and after passing through an irradiated coalesce (red bar).

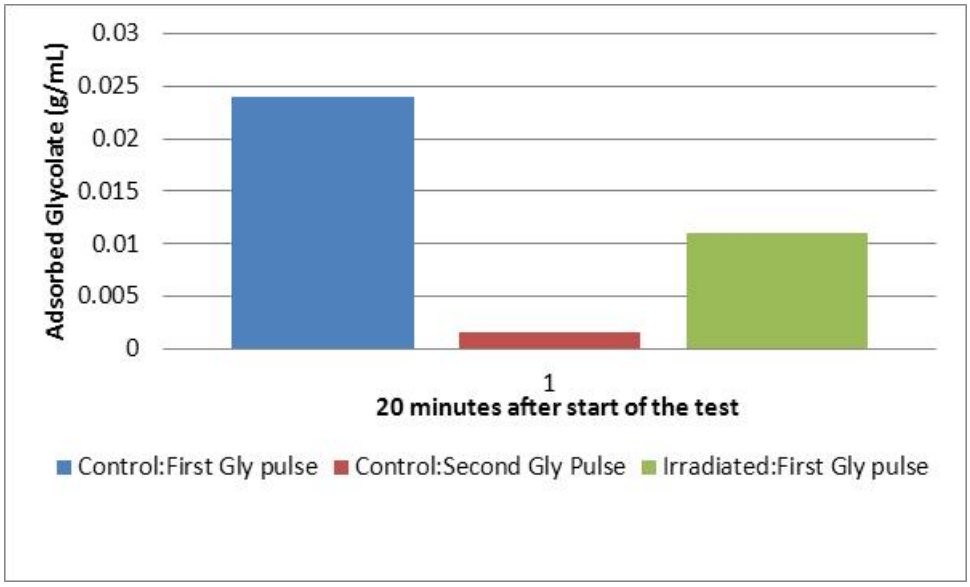

Figure 3-25. Amount of glycolate absorbed by a control coalescer sample and by a gamma irradiated coalescer.

To clarify this issue further, a sorption test was conducted between salt solution containing 10,000 ppm sodium glycolate and the as-received coalescer and in another test with CSSX solvent (using an end-over-end tumbler) for 24 hours. As shown in Table 3-7, negligible glycolate adsorption was observed on the as-received coalescer but approximately 20% of the initial glycolate concentration was lost when it contacted CSSX solvent. Although, the sorption was not as significant as observed in the coalescing tests, the loss of glycolate observed in the coalescing test appears to be due to dilution and sorption onto the CSSX solvent droplets.

Table 3-7. Raman analysis of salt solution containing glycolate (10,000 ppm) that contacted as-received coalescer and CSSX solvent for 24 hours.

Sample	Area Under C-C Stretch
10 mL glycolate solution in contact with ¾ x ¾ x ½ coalescer cut	1.42E5
Glycolate solution (2 grams per 200 mL)	1.48 E5
10 mL glycolate solution in contact with 5 mL of CSSX	1.18 E5

4.0 Conclusions

The presence of glycolate was found to impact the sorption kinetics of both MST and mMST. For MST, the presence of glycolate slowed the removal of both Sr and Pu, while increasing the removal rate of Np. Pre-contacting the MST with glycolate resulted in similar performance as when glycolate was simply present in the simulant.

In the case of mMST, glycolate was found to decrease the removal rates of all three sorbates (Sr, Pu, and Np). However, even in the presence of 10,000 ppm glycolate the mMST outperforms the baseline MST in the absence of glycolate for Pu removal, and has comparable ^{85}Sr removal to MST in the absence of glycolate. As with MST, the pre-contacting of glycolate with the mMST did not appear to have a significant effect on the performance when compared to tests having the same concentration of glycolate in the simulant. Based on these results it is likely that glycolate is impacting the removal rates by forming complexes with the sorbates, and not by fouling the MST or mMST surface.

The impact on DF measured in this report is for a single batch contact. Facility operations involve accumulation of multiple batches of MST. As a result, DF in the facility operations is not directly correlated with the single batch contact values and historically is superior to the laboratory test data. Rather than experimentally assessing the impact of multiple batches, a more practical and cost effective approach is to add glycolate impact to the salt batch qualification program for future batches after the program makes a final selection of process quantities and a better understanding of carryover from DWPF melter operations is known.

Contacting mMST with glycolate did not reduce the concentration of peroxide groups on the solids, suggesting no chemical reaction between the peroxide groups and added glycolate. Analysis of the slurries after 5 months showed minimal amounts of dissolved Ti in solution, suggesting little, if any, impact of glycolate on the dissolution rate for the MST and mMST. Addition of glycolate had a minor impact on the measured particle size distribution for MST, shifting the mean particle size slightly lower. No significant shift in particle size was observed for mMST. FTIR analyses of MST contacted with 10,000 ppm sodium glycolate solutions of varying pH indicated that there is not a strong sorption of glycolate on MST at pH 7 and above. There is sorption of glycolate on MST under acidic conditions due to hydrogen bonding of the protonated glycolic acid and MST.

From the cesium mass transfer test results, we can discern no negative effect of glycolate on the cesium removal efficiency for either the MCU current or NGS solvent. While there is a single anomalous sample result, SRNL does not feel that it affects the conclusion of these tests. A dispersion test with the MCU solvent can also find no negative phase disengagement effects from the presence of glycolate.

Microscopic and coalescing tests demonstrated that salt solution containing 10,000 ppm sodium glycolate had no effect on the coalescing function of the MCU coalescer media. Glycolate had no

effect on the coalescing ability of a gamma irradiated coalescer (8 E6 rad) despite the fact that glycolate adsorption was observed on the irradiated fibers. The observed losses in glycolate concentration are due to solution dilution and sorption onto CSSX solvent droplets.

5.0 Recommendations

Additional testing is recommended to further examine the behavior of glycolate in MCU. Specifically, we propose the following:

1. Measurement of the amount of glycolate that partitions to the solvent during ESS testing.
2. Testing to examine the glycolate – coalescer interactions during stripping (acidic conditions).
3. Material compatibility evaluations to ensure that glycolate does not negatively affect the physical properties of the various polymers used at MCU, including the behavior of irradiated glycolate.

In addition to those recommendations, we also advise that further testing be performed if the glycolate concentration exceeds 10,000 ppm in the DWPF recycle stream.

6.0 References

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Appendix A. Glycolate complexation under alkaline conditions

Complexation Calculations

Glycolate complexation under alkaline conditions

Speciation calculations for glycolate complexation with UO_2^{2+}, NpO_2^{+}, Th^{4+} (used as a 4+ analogue of Pu) and Sr^{2+} have been performed. Stability constants for the metal/glycolate system were selected from the NIST Standard Reference Database.[1] The values were chosen at the highest ionic strengths available that also gave internally consistent data. Typically μ = 1.0 M, with the exception of strontium with μ = 0.1 M. Speciation plots have been prepared using the speciation program HYSS 2009.[2]

The plots shown in Figures A-1 through A-4 display the log of the metal concentration vs. pH. The pH range 2-12 has been chosen for a broader understaning of the system even though the alkaline side is of main interest. The advantage of this display can be found in visuallization of the regions where glycolate will more strongly interact with the metals. Uranyl and Th^{4+} will be typically found as a hydroxide at pH > 7. With neptunyl, this pH increases slightly to where the hydrolysis product begins to dominate at ~ pH = 9. For Sr^{2+}, the free Sr^{2+} dominates across the pH range shown with a lower concentration of a 1:1 strontium glycolate complex. At higher pH, the 1:1 hydrolysis product begins to grow in. Based on these plots, it can be concluded that glycolate will not form a complex with the actinides in any appreciable quantities.

[1] Martell, A. E.; Smith, R. M.; Motekaitis, R. J. NIST Standard Reference Database 46, Version 8.0 – NIST Critically Selected Stability Constants of Metal Complexes, 2004.

[2] a) HYSS 2009 b) Hyperquad simulation and speciation (HySS): a utility program for the investigation of equilibria involving soluble and partially soluble species", *Coordination Chemistry Reviews*, **184** (1999) 311-318.

Table 1. Concentrations of metal and ligand species, of interest in the system, used in speciation calculations.

Metal or ligand of interest	ug/L	M
U	10,000	4.20E-05
Np	500	2.11E-06
Pu	200	8.37E-07
Total Sr		6.85E-06
Total Cs		1.40E-04
Gly		0.133

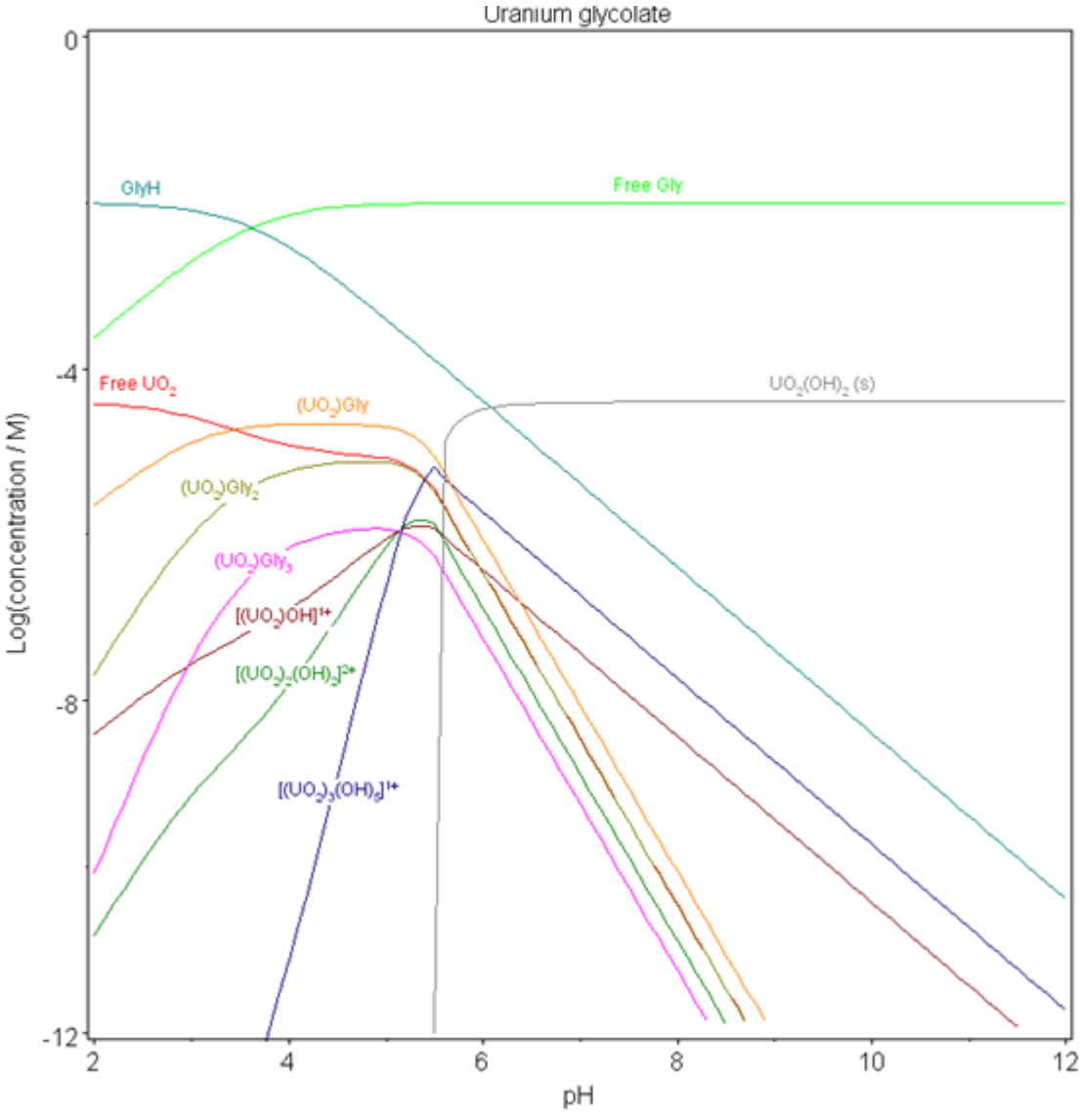

Figure A-1. Speciation plot for uranyl in the presence of glycolate.

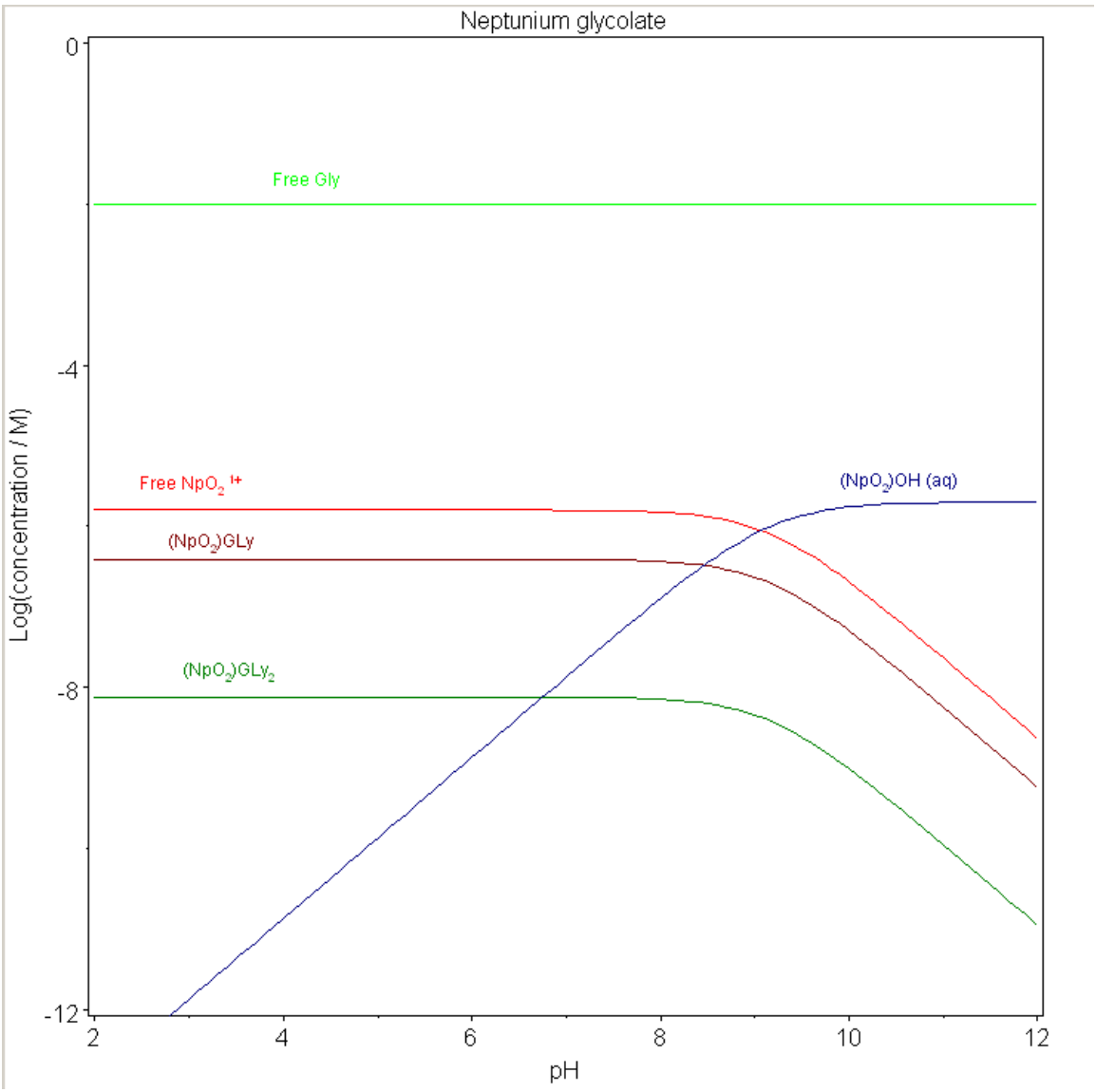

Figure A-2. Speciation plot for neptunyl in the presence of glycolate.

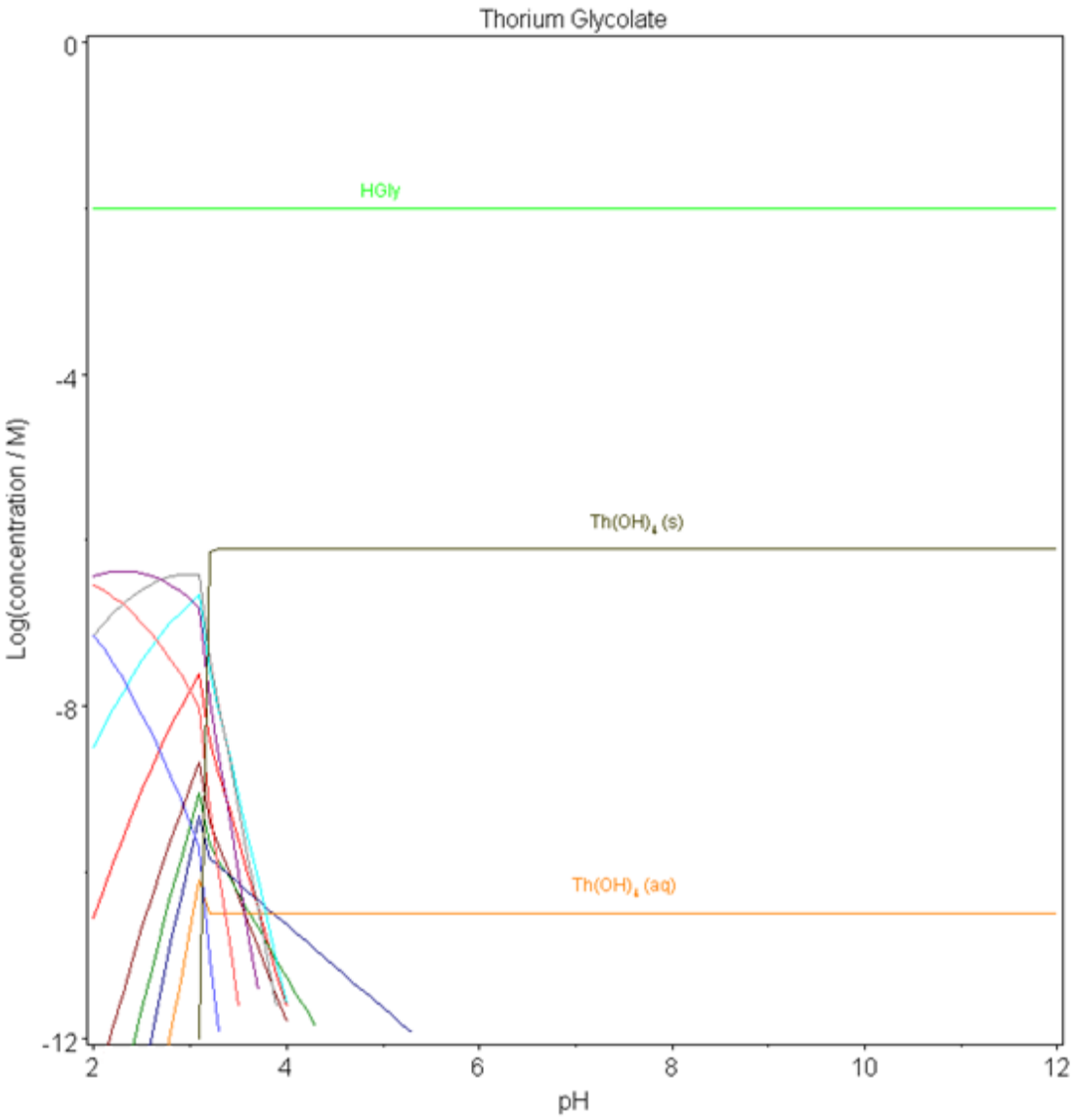

Figure A-3. Speciation plot for Th^{4+} in the presence of glycolate.

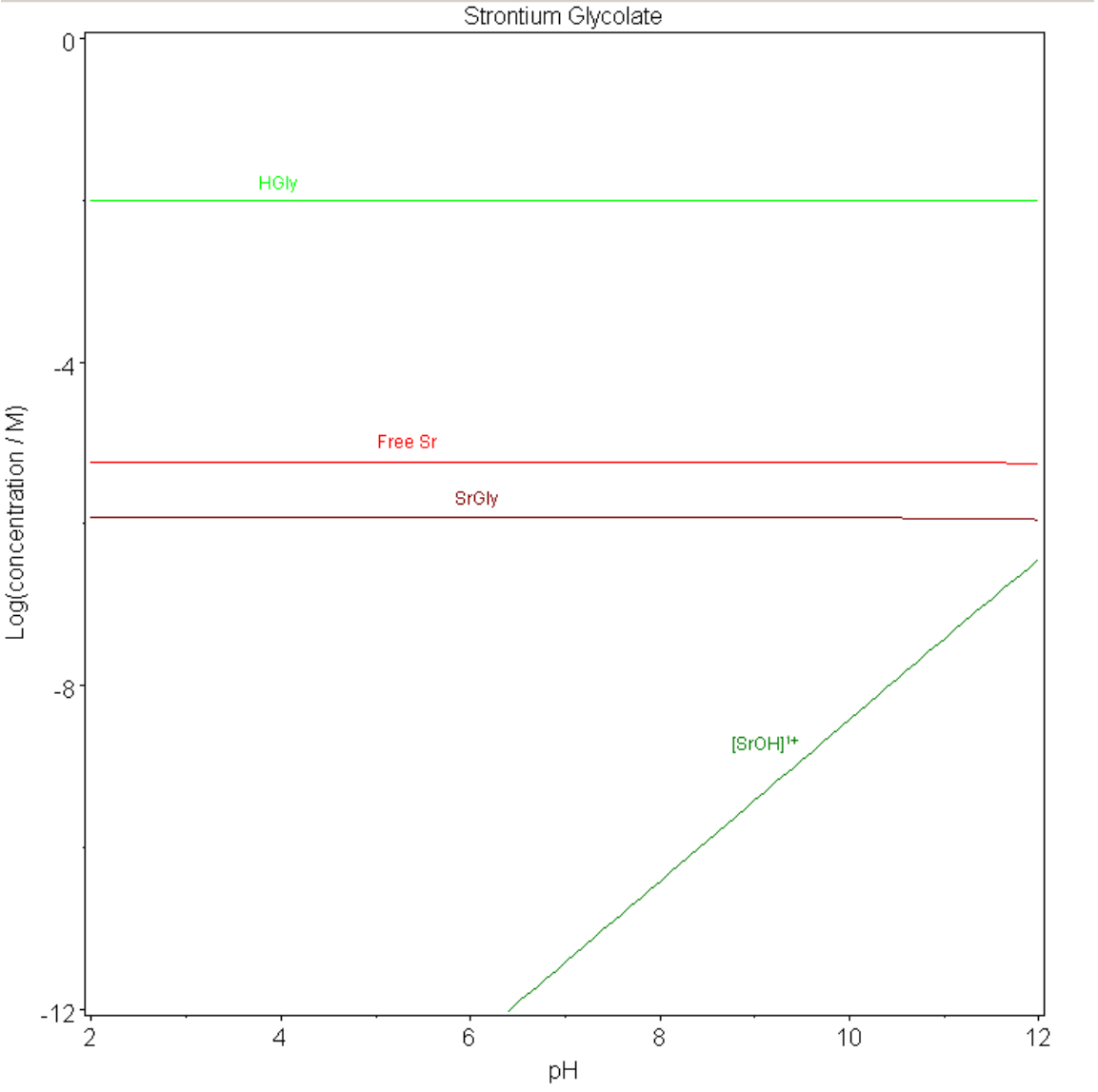

Figure A-4. Speciation plot for Sr^{2+} in the presence of glycolate.

Appendix B. Additional MST/mMST sorption data

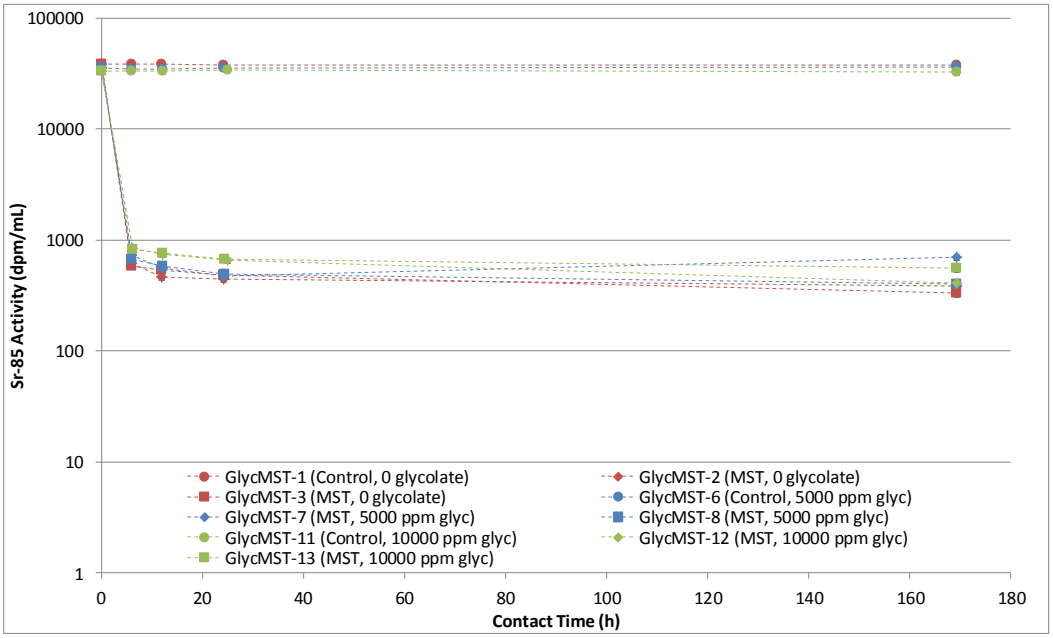

Figure B-1. ^{85}Sr activity versus contact time with MST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

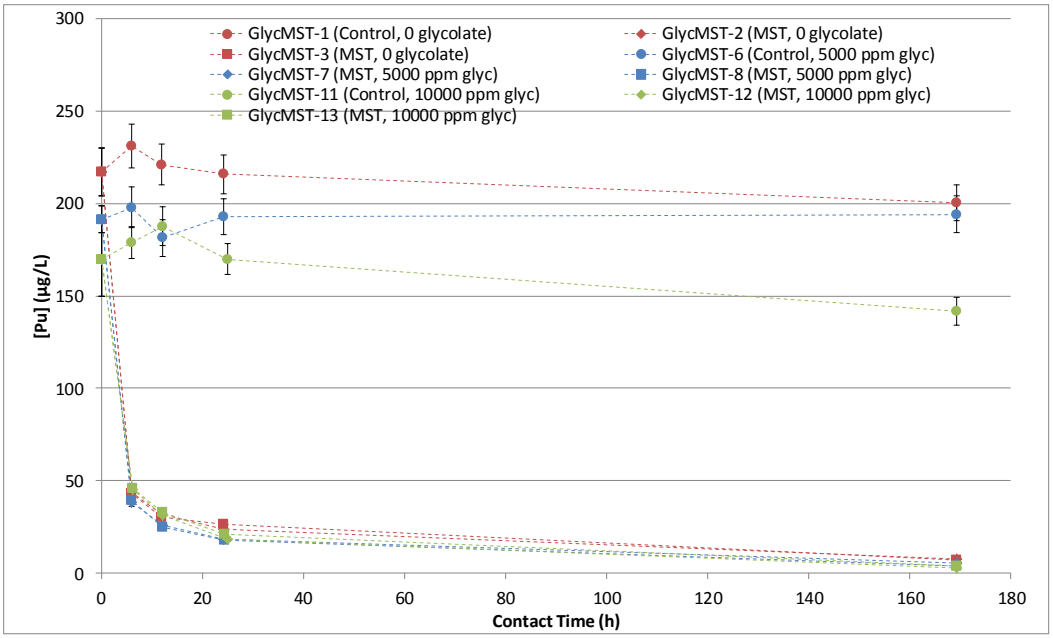

Figure B-2. Pu concentration versus contact time with MST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

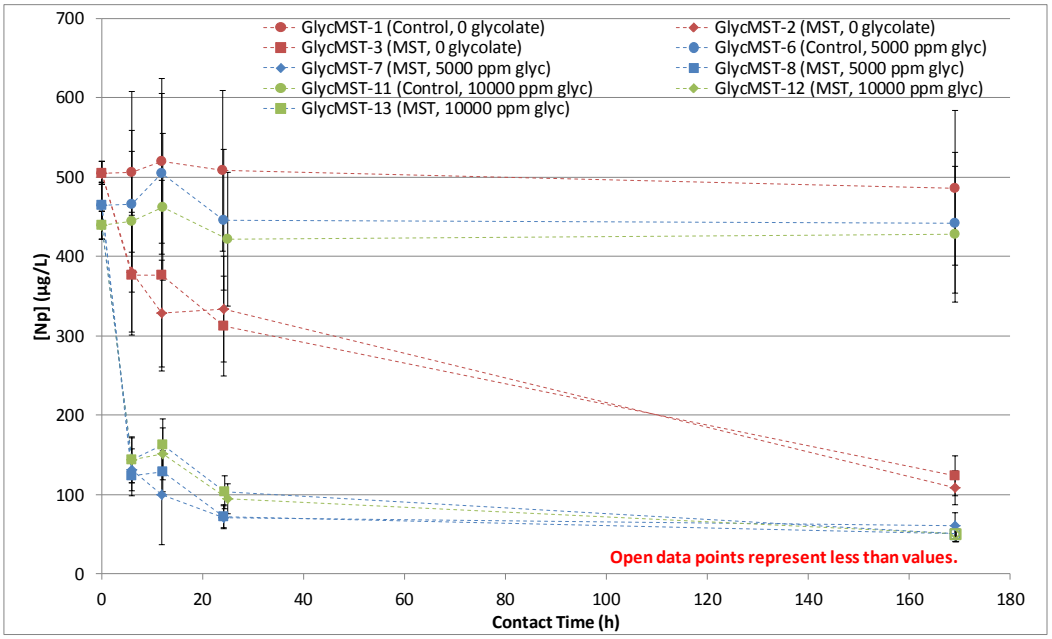

Figure B-3. Np concentration versus contact time with MST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

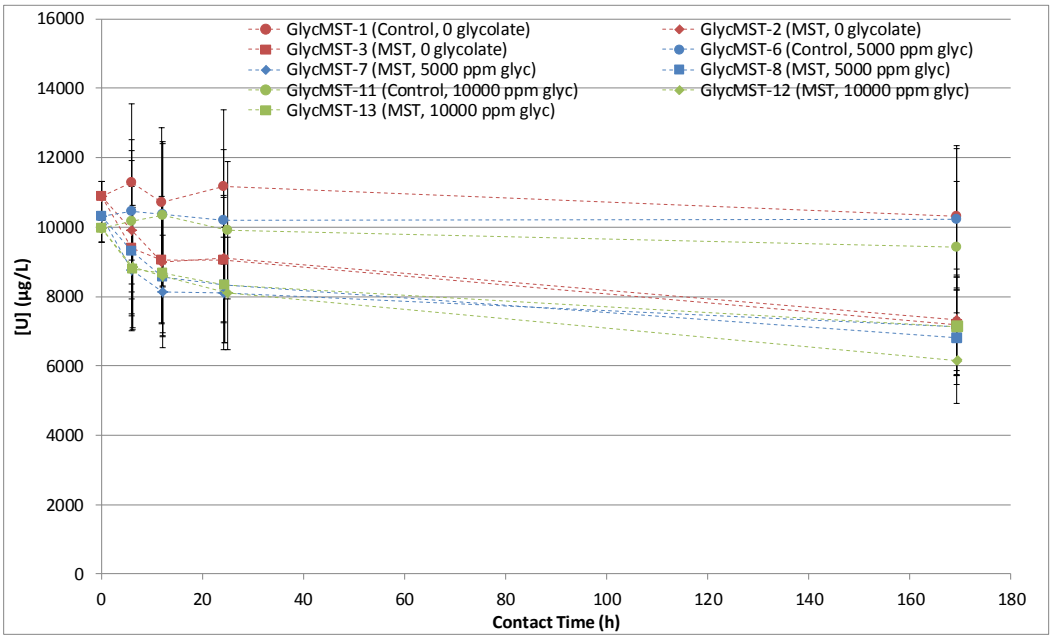

Figure B-4. U concentration versus contact time with MST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

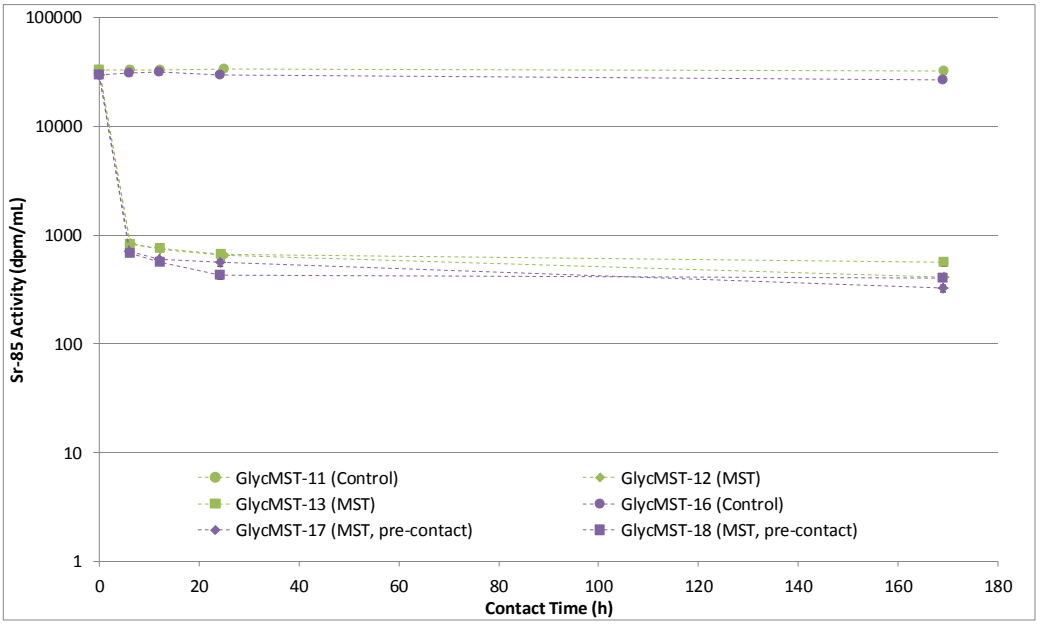

Figure B-5. ^{85}Sr activity versus contact time with MST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the MST.

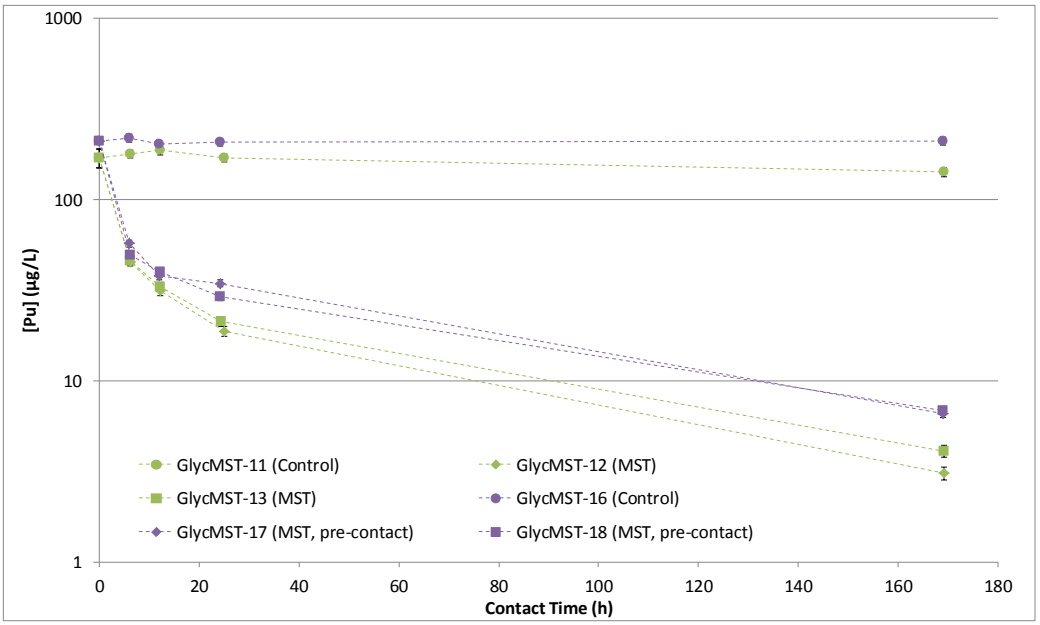

Figure B-6. Pu concentration versus contact time with MST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the MST.

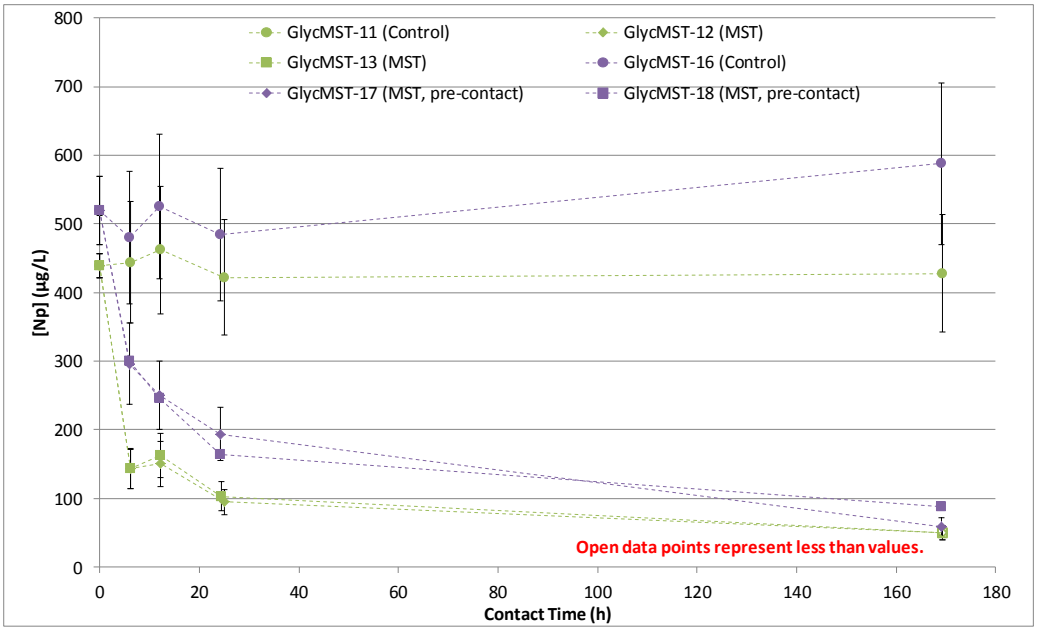

Figure B-7. Np concentration versus contact time with MST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the MST.

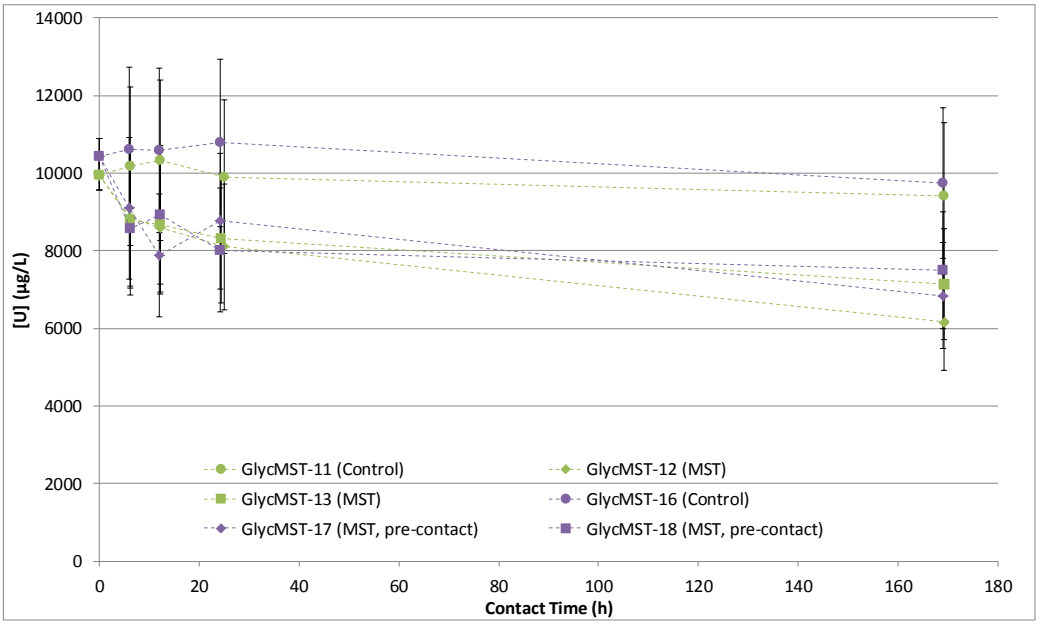

Figure B-8. U concentration versus contact time with MST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the MST.

Table B-1. ^{85}Sr DFs for MST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	64.6 (3.10)	49.7 (2.77)	40.2 (0.663)	44.6 (1.80)
12	76.9 (8.39)	61.3 (1.99)	44.3 (0.686)	54.4 (2.29)
24	81.2 (4.52)	73.6 (2.24)	51.5 (0.723)	62.3 (11.9)
168	106 (11.1)	70.2 (26.6)	68.3 (14.8)	75.4 (11.3)

Table B-2. Pu DFs for MST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	5.31 (0.066)	5.08 (0.141)	3.92 (0.054)	4.14 (0.428)
12	7.07 (0.286)	7.07 (0.200)	5.84 (0.197)	5.18 (0.168)
24	8.60 (0.617)	10.5 (0.256)	8.51 (0.765)	6.61 (0.809)
168	26.8 (0.549)	41.6 (12.1)	40.3 (7.91)	31.4 (0.885)

Table B-3. Np DFs for MST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	1.34 (0.010)	3.67 (0.147)	3.10 (0.006)	1.61 (0.015)
12	1.48 (0.143)	4.48 (0.815)	2.95 (0.155)	2.12 (0.024)
24	1.57 (0.076)	6.22 (0.061)	4.28 (0.263)	2.72 (0.313)
168	4.22 (0.397)	> 8.10 (1.04)	> 8.56 (0.000)	8.39 (2.37)

Table B-4. U DFs for MST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	1.17 (0.046)	1.16 (0.049)	1.15 (0.006)	1.20 (0.052)
12	1.19 (0.006)	1.24 (0.044)	1.20 (0.008)	1.27 (0.111)
24	1.23 (0.006)	1.24 (0.026)	1.21 (0.023)	1.29 (0.080)
168	1.42 (0.019)	1.46 (0.047)	1.42 (0.148)	1.36 (0.089)

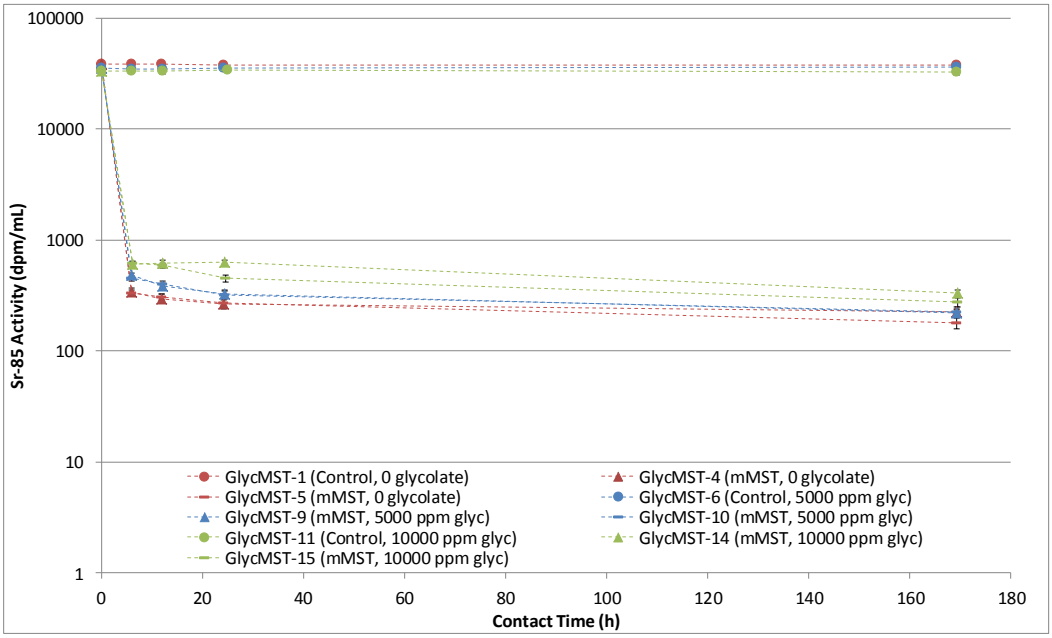

Figure B-9. ^{85}Sr activity versus contact time with mMST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

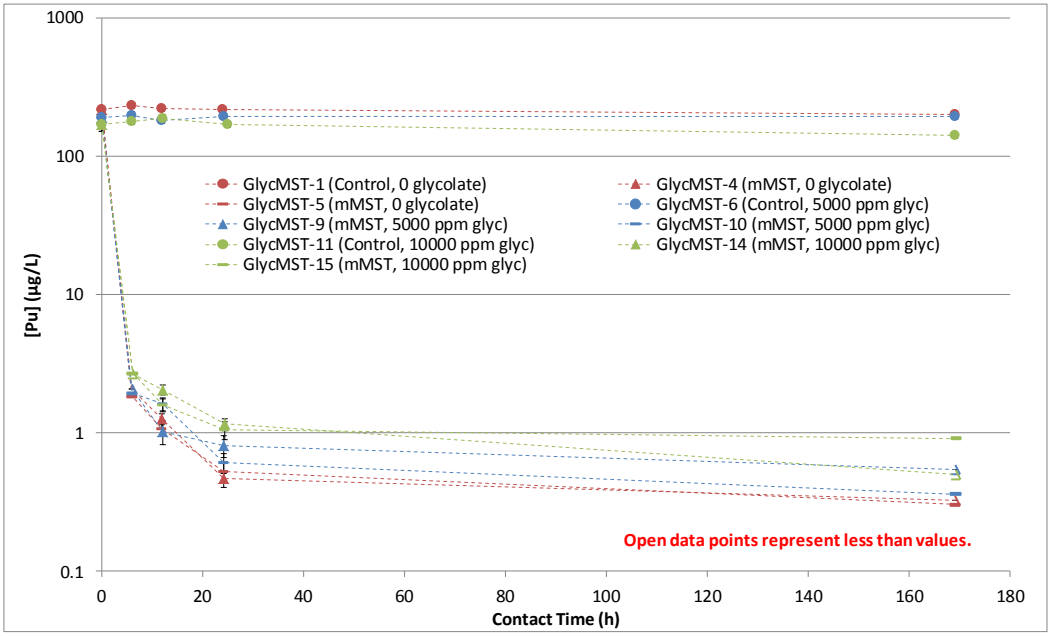

Figure B-10. Pu concentration versus contact time with mMST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

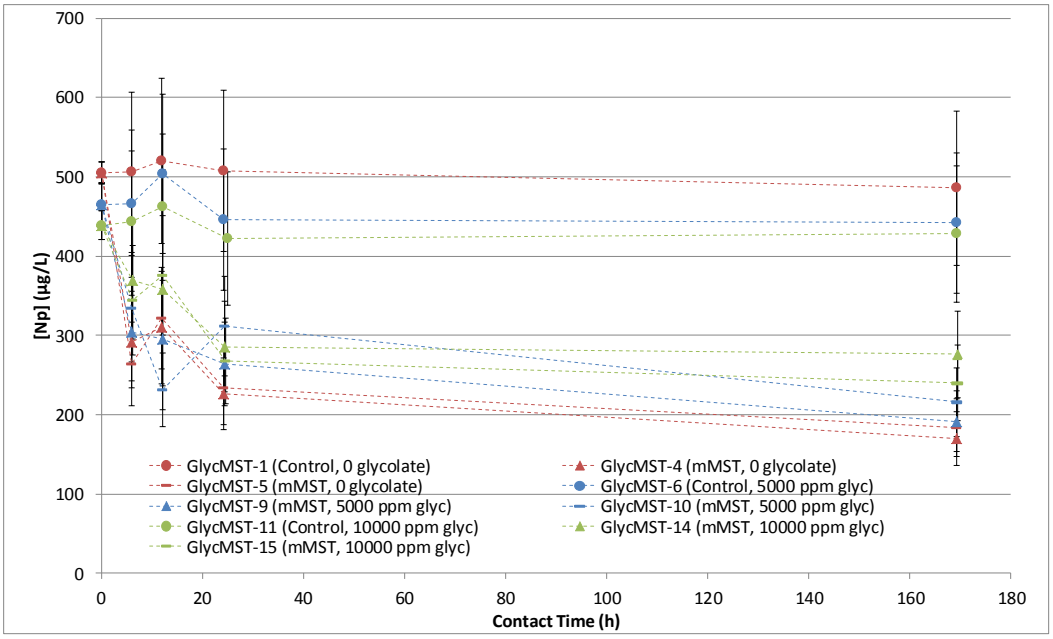

Figure B-11. Np concentration versus contact time with mMST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

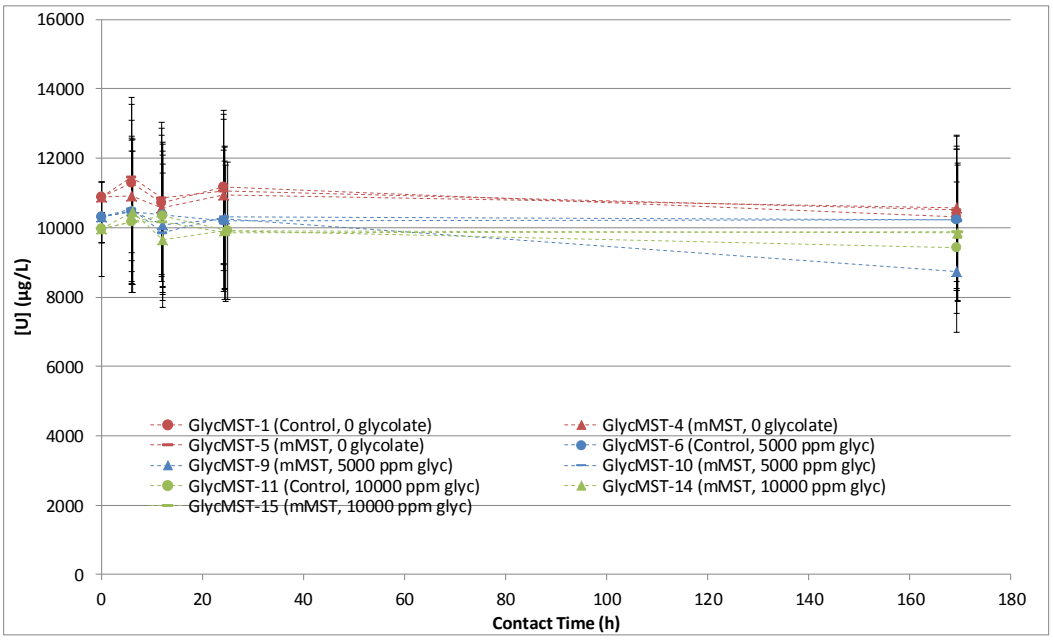

Figure B-12. U concentration versus contact time with mMST in the presence of 0 ppm (red), 5000 ppm (blue), or 10,000 ppm (green) glycolate.

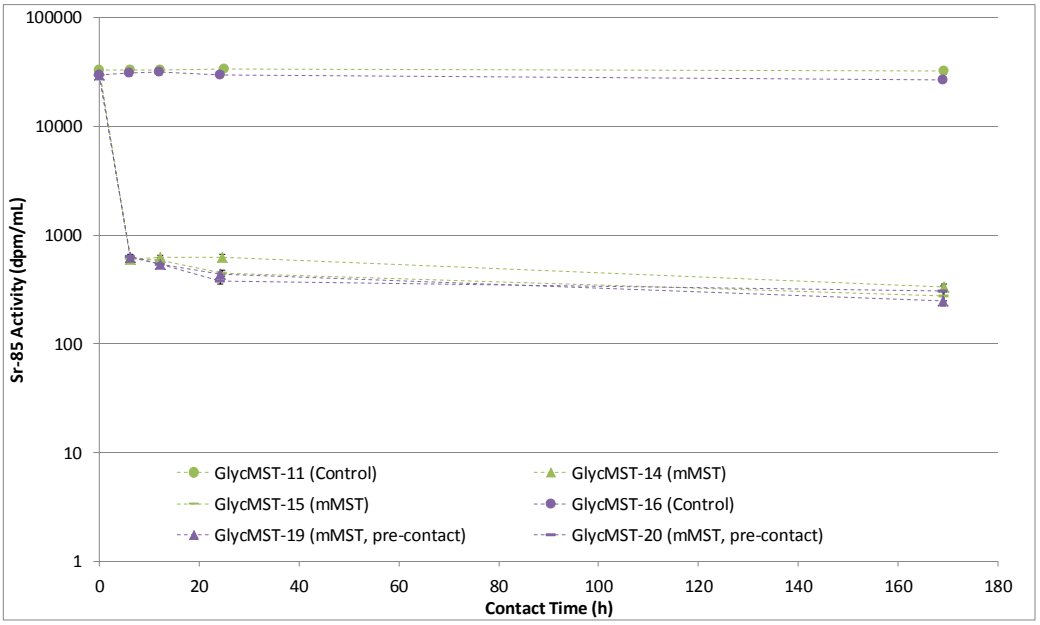

Figure B-13. ^{85}Sr activity versus contact time with mMST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the mMST.

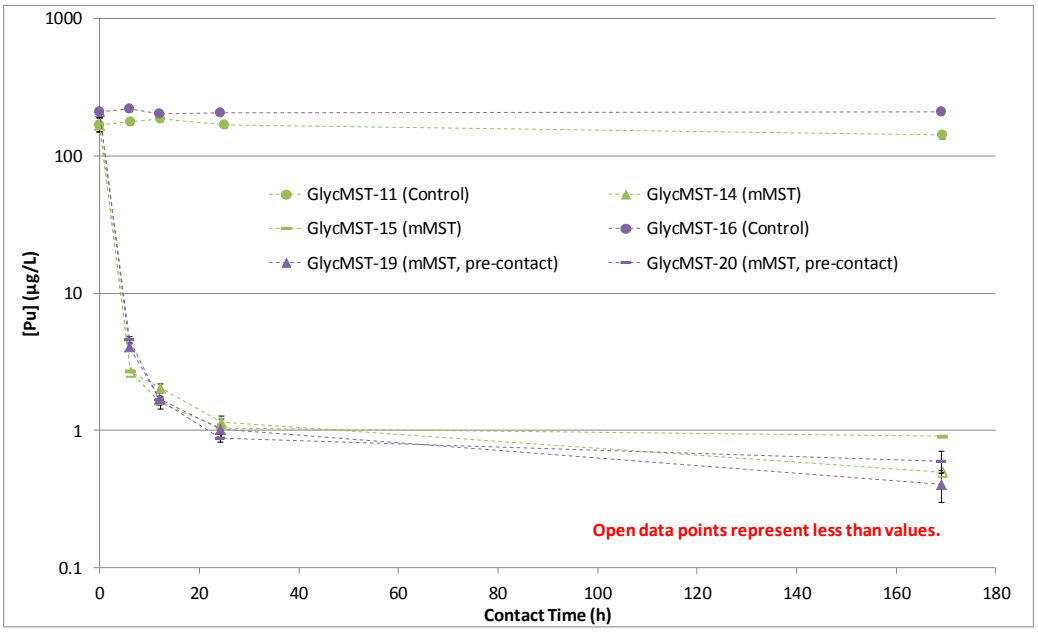

Figure B-14. Pu concentration versus contact time with mMST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the mMST.

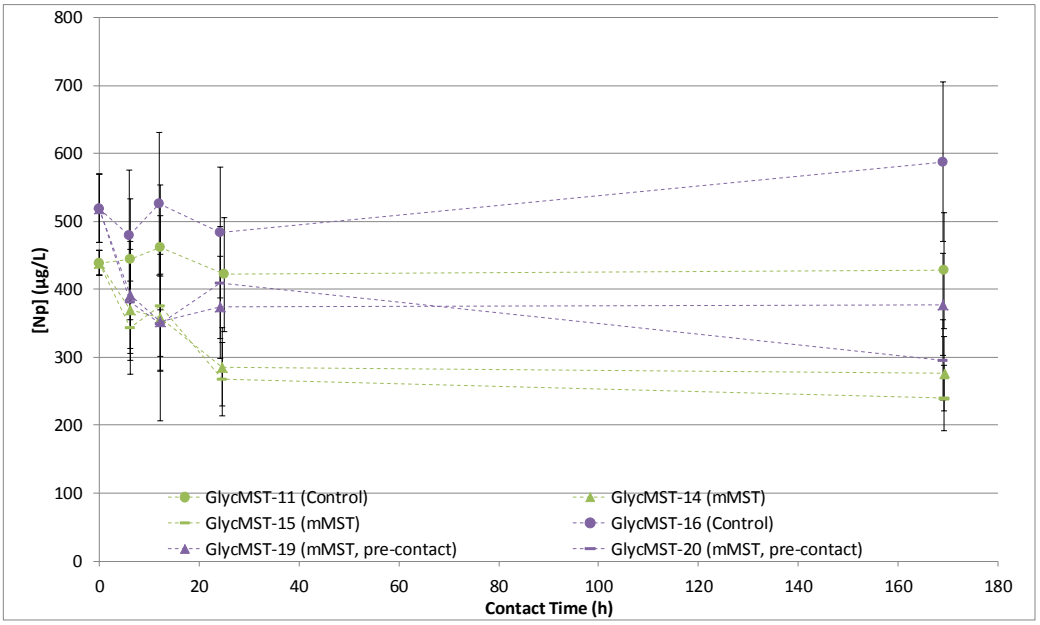

Figure B-15. Np concentration versus contact time with mMST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the mMST.

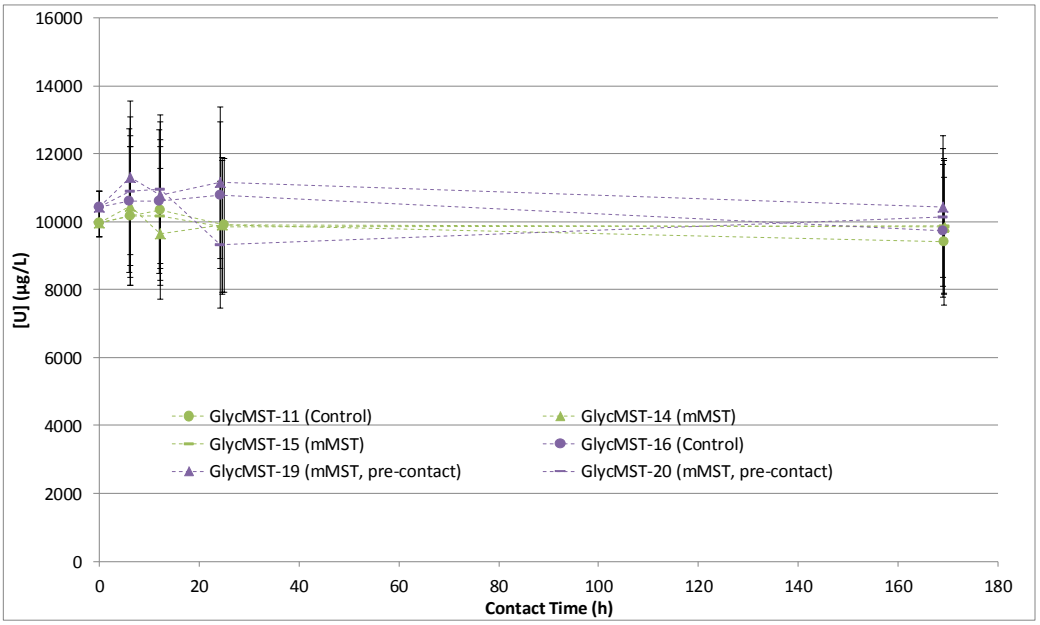

Figure B-16. U concentration versus contact time with mMST in the presence of 10,000 ppm glycolate with (purple) and without (green) pre-contacting the glycolate with the mMST.

Table B-5. ^{85}Sr DFs for mMST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	115 (2.80)	74.7 (2.87)	55.0 (1.02)	49.2 (0.885)
12	126 (3.13)	87.8 (3.30)	54.8 (2.02)	58.5 (0.077)
24	142 (1.93)	109 (1.16)	64.6 (15.3)	74.4 (7.32)
168	190 (28.3)	162 (2.51)	107 (15.2)	98.4 (14.6)

Table B-6. Pu DFs for mMST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	> 119 (9.89)	> 99.2 (4.94)	> 66.7 (0.452)	50.8 (3.70)
12	191 (21.9)	145 (45.7)	105 (18.2)	> 121 (1.28)
24	436 (34.0)	278 (57.1)	155 (10.9)	220 (23.2)
168	> 642 (31.7)	> 449 (128)	> 220 (91.3)	434 (115)

Table B-7. Np DFs for mMST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	1.82 (0.130)	1.46 (0.097)	1.25 (0.064)	1.24 (0.023)
12	1.65 (0.044)	1.94 (0.332)	1.26 (0.044)	1.50 (0.006)
24	2.21 (0.054)	1.56 (0.184)	1.53 (0.070)	1.24 (0.080)
168	2.75 (0.147)	2.18 (0.183)	1.67 (0.164)	1.77 (0.305)

Table B-8. U DFs for mMST in the presence of 0, 5000, and 10,000 ppm glycolate. The values represent the average of two replicate trials, with the standard deviations given in parenthesis.

Time (h)	0 ppm glycolate	5000 ppm glycolate	10,000 ppm glycolate	
			In simulant	Pre-contact w/MST
6	1.01 (0.034)	0.993 (0.004)	0.987 (0.019)	0.957 (0.024)
12	1.00 (0.020)	1.04 (0.016)	1.04 (0.040)	0.974 (0.010)
24	1.01 (0.008)	0.992 (0.003)	1.00 (0.006)	1.06 (0.133)
168	0.977 (0.003)	1.09 (0.122)	0.955 (0.003)	0.947 (0.020)

Distribution:

S. D. Fink, 773-A
K. M. Fox, 999-W
B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)

E. J. Freed, 704-S
J. M. Bricker, 704-27S
T. L. Fellinger, 704-26S
J. M. Gillam, 766-H
B. A. Hamm, 766-H
E. W. Holtzscheiter, 704-15S
J. F. Iaukea, 704-30S
M. T. Keefer, 241-156H
D. P. Lambert, 999-W
D. W. McIlmoyle, 766-H
J. E. Occhipinti, 704-S
D. K. Peeler, 999-W
W. O. Pepper, 704-S
J. W. Ray, 704-S
M. A. Rios-Armstrong, 241-156H
H. B. Shah, 766-H
D. C. Sherburne, 704-S
A. V. Staub, 704-27S
M. E. Stone, 999-W

P. R. Jackson, DOE-SR, 703-46A
K. H. Subramanian, 766-H

K. M. L. Taylor-Pashow, 773-A
T. C. Shehee, 773-A
D. T. Hobbs, 773-A
T. B. Peters, 773-42A
F. F. Fondeur, 773-A
A. L. Washington, 773-42A