PNNL-19015
WTP-RPT-206, Rev 0

U.S. DEPARTMENT OF
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Effect of Antifoam Agent on Oxidative Leaching of Hanford Tank Sludge Simulant

BM Rapko
SA Jones
GJ Lumetta
RA Peterson

February 2010

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Test Specification: 24590-PTF-TSP-RT-06-002, Rev. 0
Test Plan: TP-RPP-WTP-456, Rev. 0.2
Test Exception: 24590-WTP-TEF-RT-08-00012 Rev. 1
R&T focus area: Pretreatment
Test scoping statement(s): 24590-WTP-PL-RT-01-002, Appendix C, B-89

Pacific Northwest National Laboratory
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by Test Plan TP-RPP-WTP-456, Rev 0.2, and ICN- TP-RPP-WTP-456, Rev 0.2. 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

Gordon H. Beeman, Manager
RPP-WTP Support Program

2/26/10
Date

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Acronyms

AEA	alpha energy analysis
AFA	antifoam agent
ASO	Analytical Support Operations
BNI	Bechtel National Incorporated
CUF	Cells Unit Filter
DI	deionized (water)
DOE	U.S. Department of Energy
HDI	"How Do I…?"
IC	ion chromatography
ICP	inductively coupled plasma
LSC	liquid scintillation counting
M&TE	measuring and test equipment
OES	optical emission spectroscopy
PNNL	Pacific Northwest National Laboratory
PNWD	Battelle—Pacific Northwest Division
QA	quality assurance
QAM	Quality Assurance Manual
QAP	Quality Assurance Plan
QARD	Quality Assurance Requirements and Descriptions
QC	quality control
RF	resorcinol-formaldehyde
RPL	Radiochemical Processing Laboratory
RPP	River Protection Project
SOW	statement of work
SP	Support Project
TIC	total inorganic carbon
TOC	total organic carbon
TP	Test Plan
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WTPSP	Waste Treatment Plant Support Project

Testing Summary

Previous studies sponsored by Bechtel National Inc. (BNI) have targeted optimizing the use of sodium permanganate for selectively oxidizing chromium from washed Hanford tank sludge (Rapko et al. 2004; Rapko et al. 2005). The need for the proposed additional testing of the chromium simulant containing antifoam agent (AFA) for leach behavior during oxidative leaching was identified as a result of evaluating AFA effectiveness during oxidative leaching (SCT-MORLE60-00-199-00002 Rev 00A). This report documents the results of those tests.

Objectives

Table S.1 describes the test objectives and whether the objectives were met.

Table S.1. Description of Test Objectives

Test Objective	Objective Met (Y/N)	Discussion
Investigate the efficacy of defined oxidant dosage through bench-scale experimental testing with two actual Hanford tank waste sludges.	Yes	Provided in report WTP-RPT-171, Rev 0.
Demonstrate the oxidative leaching process in the laboratory using actual waste at conditions that represent the anticipated plant flowsheet conditions for all anticipated process systems.	Yes	Provided in report WTP-RPT-171, Rev 0.
Develop information on the fate of Pu and neutron absorbers (B and Cd) for both process design verification and process criticality safety assessment. Specifically, information that will be obtained is the concentration of Pu in the oxidative leachate and the oxidation state(s) of the Pu (using techniques being developed in the current Pu speciation determination activity). Further, changes in the Pu oxidation state will be monitored during the nominal processing steps of the leachate, and material balances will be monitored to assess the potential to precipitate Pu during any of the steps before ion exchange. Finally, the fate of Pu during ion exchange (using spherical resorcinol-formaldehyde) will be determined using feed adjusted to	Yes	Provided in report WTP-RPT-171, Rev 0.

Table S.1. Description of Test Objectives

Test Objective	Objective Met (Y/N)	Discussion
5 M Na (through evaporative concentration or dilution) and elution with 0.5 M HNO_3.		
Investigate the efficacy of defined oxidant dosage through bench-scale experimental testing with two actual Hanford tank waste sludges.	Yes	Provided in report WTP-RPT-171, Rev 0.
Determine the effect of AFA on the oxidative leaching efficiency for chromium (as extent of chromium oxidative leached), using the radioactive simulant developed for the oxidative leaching studies. A radioactive simulant is required to satisfy all the listed objectives.	Yes	A radioactive simulant was prepared and used in all tests. There was no apparent effect of the AFA on oxidative leaching efficiency for chromium as described in Results and Discussion, Section 3.2.
Determine the effects of AFA on dissolution of Pu and other criticality-related elements during oxidative leaching, using the radioactive simulant developed for the oxidative leaching studies.	Yes	Plutonium was included in the radioactive simulant used for testing the AFA effect. There was no apparent effect of AFA on Pu, Am, or U during oxidative leaching.
Verify the results from the radioactive simulant tests above with a minimum of two actual waste tests with the Group 6 S-Saltcake waste solids.	No	Since 1) addition of AFA did not affect the extent of Pu dissolution from the simulant, and 2) the Pu in the actual waste has generally been less amenable to oxidation relative to the Pu in the simulant, a decision was made by BNI not to test with the actual waste. (E-mail from F. Damerow to R. A. Peterson, May 8, 2009, included in Appendix A).[a]

Test Exceptions

The work reported herein is in direct response to Test Exception 24590-WTP-TEF-08-00012. This Test Exception outlines a matrix of experiments to be done to evaluate any impact of the presence of antifoaming agent on the oxidative leaching of a series of criticality-important elements as well as Cr itself from a simulant made through a specified preparation method. The matrix primarily examines the impact of temperature and AFA concentration on the oxidative leaching process. The Test Exception also outlines work to be done with two radioactive Hanford tank sludges and provides that the results of this Test Exception be presented in a stand-alone report. No AFA testing results will be reported for actual tank waste.

(a) Documented in Corrective Action Report CAR #48697.1.

Exceptions to the Test Specifications

This Test Plan followed the guidance provided by the Test Specification 24590-PTF-TSP-RT-06-002, Rev 0 and Test Exception 24590-WTP-TEF-RT-07-00016 with exceptions listed in Table S.2:

Table S.2. Exceptions to the Test Specifications and Their Justification

Exception	**Justification**
1. Inhibited water (0.01 M NaOH + 0.01 M $NaNO_2$) will be used instead of deionized (DI) water to wash the slurry (see Steps 2.6 and 2.10).	1. Slightly basic water is preferred for sludge washing to maintain solubility of aluminum hydroxides.
2. The Cs ion exchange testing will be conducted using the alternative ion exchanger, spherical resorcinol-formaldehyde (RF) resin instead of SL-644.	2. The RF resin is the new baseline resin.
3. Parametric testing with the Cells Unit Filter (CUF) was included in the Test Plan.	3. Additional testing is needed to provide useful filtration data required in Test Specification 24590-PTF-TSP-RT-06-003 Rev 0 for actual waste testing to resolve Issue M12 (24590-WTP-PL-ENG-06-0024).
4. Additional analytical testing was included in Task 5 and Task 6.	4. Additional testing was requested from Engineering (BNI) to support process modeling efforts.
5. Caustic (19 M NaOH) will be added if needed to increase the free hydroxide concentration to the range of 0.1 to 0.25 M.	5. It is possible that the washing alone will be sufficient to bring the free hydroxide to the required range of 0.1 to 0.25 M. In this case, no additional NaOH will be added.
6. Results of the radioactive simulant tests were not verified with actual waste tests with the Group 6 S-Saltcake waste solids.	6. Since 1) addition of AFA did not affect the extent of Pu dissolution from the simulant, and 2) the Pu in the actual waste has generally been less amenable to oxidation relative to the Pu in the simulant, a decision was made by BNI not to test with the actual waste. (E-mail from F. Damerow to R. A. Peterson, May 8, 2009, included in Appendix A).(a)

(a) Documented in Corrective Action Report CAR #48697.1.

Results and Performance Against Success Criteria

Table S.3 describes the success criterion and explains how the criterion was met or not.

Table S.3. Success Criterion

Success Criterion	Explain How the Tests Did or Did Not Meet the Success Criterion
Successful demonstration of the oxidative leaching process using actual waste under conditions that simulate the anticipated plant flowsheet conditions for the anticipated pretreatment process systems: crossflow ultrafiltration, caustic leaching, water washing, oxidative leaching, water washing, ion exchange, and evaporation. This includes demonstration of Cr removal from the solids fraction, effective filtration through the CUF, and no measurable retention of Pu in the ion exchanger.	Provided in report WTP-RPT-171, Rev 0.
Determination of concentrations, closure of mass balances, and speciation of Cr, Pu, and Mn in solution as well as the fate of B, Cd, Cr, Pu, Fe, and Ni in solution for the anticipated pretreatment process systems (crossflow filtration, caustic leaching, water washing, oxidative leaching, water washing, ion exchange, and evaporation).	Provided in report WTP-RPT-171, Rev 0.
Measurement of selected metal concentrations of solids (that are soluble in 2 M nitric acid) associated with the pretreatment-system component surfaces, including Pu (for potential enrichment).	Provided in report WTP-RPT-171, Rev 0.
Determination of selected metal analyte composition, including Pu, of bulk precipitated solids (if any) in the feed evaporator process system (evaporator bottoms and scale).	Provided in report WTP-RPT-171, Rev 0.
Determine the effect of AFA concentration on oxidative leaching of chromium.	This criterion was met and described in Chapter 3 of this report.
Provide a graphical correlation (plot) of the effect of AFA on oxidative leaching of chromium to Process Engineering/Operations.	Met success criterion. Figure 3.4 is a plot of Cr removal from the solids by oxidative leaching at either 25°C or 45°C as a function of AFA concentration.

Table S.3. Success Criterion

Success Criterion	Explain How the Tests Did or Did Not Meet the Success Criterion
Provide a graphical correlation (plot) of the effect of AFA on oxidative leaching of plutonium and the criticality related elements to Process Engineering/Operations. The criticality related elements in the simulant include Fe, Ni, and Mn.	Met success criterion. Figures 3.7 through 3.27 plot results as a function of AFA concentration for iron, nickel, manganese and plutonium. Note that this criterion can only refer to the radioactive simulant tests given in the presence of Pu and U as part of the safety critical elements to be evaluated. The specific elements designated as safety critical are provided in Test Specification 24590-PTF-TSP-RT-06-002, Rev 0, "Process Development for Design of Oxidative Leaching of Hanford Wastes."(a)
Verification of the results from the radioactive simulant tests at a minimum of two test conditions using the waste solids from Group 6 S-Saltcake waste. Verification results will be plotted against Success Criteria 1 and 2 above.	Direction was given by BNI to not test with actual waste.(b)

Quality Requirements

The PNNL Quality Assurance Program is based upon the requirements as defined in the U.S. Department of Energy (DOE) Order 414.1C, *Quality Assurance* and 10 CFR 830, *Energy/Nuclear Safety Management*, Subpart A—*Quality Assurance Requirements* (a.k.a. the Quality Rule). PNNL has chosen to implement the following consensus standards in a graded approach:

- ASME NQA-1-2000, Quality Assurance Requirements for Nuclear Facility Applications, Part 1, *Requirements for Quality Assurance Programs for Nuclear Facilities*
- ASME NQA-1-2000, Part II, Subpart 2.7, Quality Assurance Requirements for Computer Software for Nuclear Facility Applications
- ASME NQA-1-2000, Part IV, Subpart 4.2, Graded Approach Application of Quality Assurance Requirements for Research and Development.

The procedures necessary to implement the requirements are documented in PNNL's "How do I…?" (HDI).(c)

PNNL implemented the RPP-WTP quality requirements by performing work in accordance with the River Protection Project—Hanford Tank Waste Treatment and Immobilization Plant Support Program (RPP-WTP) Quality Assurance Plan (RPP-WTP-QA-001, QAP). Work was performed to the quality requirements of NQA-1-1989 Part I, *Basic and Supplementary Requirements*, NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD)*. These quality requirements were implemented through the River Protection Project—Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Manual (RPP-WTP-QA-003, QAM). The analytical

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(a) PS Sundar. 2006. *Process Development for Design of Oxidative Leaching of Hanford Wastes*. 24590-PTF-TSP-RT-06-002, Rev. 0, Bechtel National, Incorporated, Richland, Washington.

(b) An e-mail note from F Damerow (BNI) to RA Peterson (PNNL) on May 08, 2009, communicated this decision. Email included in Appendix A.

(c) PNNL's system for managing the delivery of laboratory-level policies, requirements, and procedures.

requirements are implemented through RPP-WTP's Statement of Work (WTPSP-SOW-005 and RPP-WTP-QA-005, respectively) with the Radiochemical Processing Laboratory (RPL) Analytical Support Operations (ASO). The requirements of DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions* (QARD) were not required for this work.

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

R&T Test Conditions

Table S.4 lists the research and technology test conditions and whether they were followed.

Table S.4. List of R&T Test Conditions and Whether They Were Followed

List R&T Test Conditions	**Were Test Conditions Followed?**
Actual Waste Selection and Compositing	
4 to 5 L of actual tank waste will be obtained with sludge, saltcake, and supernatant components. Saltcake will be dissolved in water before being added to sludge.	Provided in report WTP-RPT-171, Rev 0.
The tank waste components will be composited in a stainless steel vessel. Mixing will be effected using an overhead stirrer equipped with a suitable stir blade. Homogenization will be evaluated from three subsamples collected from the vessel at three different levels (one sample per level) and will be based on equal (±1.5%) slurry densities.	Provided in report WTP-RPT-171, Rev 0.
Oxidant Dosage Testing	
Nonradioactive simulant was tested with Cr_2O_3 and $Cr(O)(OH) – H_2O$ to determine the effect of mixing conditions, hydroxide concentration, temperature, and initial $[MnO_4]/[Cr]$ ratio on the effectiveness of oxidative leaching. The initial $[MnO_4]/[Cr]$ ratio was varied from 0.75 to 1.25 at 25°C and 45°C in caustic solutions that ranged from 0.25 to 3 M NaOH.	Provided in report WTP-RPT-171, Rev 0.
Demonstration Test	
Nonradioactive simulant was tested with Cr_2O_3 and $Cr(O)(OH) – H_2O$ to determine the effect of mixing conditions, hydroxide concentration, temperature, and initial $[MnO_4]/[Cr]$ ratio on the effectiveness of oxidative leaching. The initial $[MnO_4]/[Cr]$ ratio was varied from 0.75 to 1.25 at 25°C and 45°C in caustic solutions that ranged from 0.25 to 3 M NaOH.	Provided in report WTP-RPT-171, Rev 0.

Simulant Use

A simulant previously developed for monitoring the impact of changing permanganate dosage on chromium(III) conversion to soluble chromate in alkaline solutions was used in these tests. The simulant was designed to evaluate Cr removal as a function of AFA concentration and changing leaching conditions. Chromium(III) oxyhydroxide hydrate was used for the leaching tests. The other components in the simulant were generally based on observed phases present in Hanford tank sludge solids, but their presence, with the exception of Cr, was based primarily on their importance to criticality safety, and concentrations were motivated primarily for detection convenience.

Plutonium nitrate, containing americium-241 in-grown from plutonium-241 decay, was mixed with ferric nitrate in a nitric acid solution. The iron and plutonium plus americium were coprecipitated by neutralizing the nitric acid with sodium hydroxide. A mixture of U(IV) and U(VI) oxides was mixed with the oxide/hydroxides of chromium, nickel, and zinc to form the simulant slurry used in testing. No manganese was added to the initial simulant preparation. During simulant characterization, some aliquots indicated the presence of traces of Mn that may have been present as impurities or were an analytical artifact of the complex sample matrix. The antifoam agent was added to individual aliquots of the slurry and allowed to equilibrate for at least 24 hours before leach testing.

Discrepancies and Follow-On Tests

The Test Exception and Test Plan specified stirring during the caustic leaching sufficient to maintain suspended solids. Due to experimental difficulties, the caustic leaching system was agitated by rotary shaking at 200 rpm. It is not believed that this change impacted the conclusions in this report. All tests were conducted using the same method for mixing, so the relative effects are consistent. In addition, the conclusions are based solely on the radioactive simulant and were not verified with actual waste testing. Confirmation of the simulant conclusions with actual waste would be useful.

The four tests with an Mn/Cr ratio of 1.25 were inadvertently carried out at a ratio of 1.0. The result of this action is that all tests were conducted with the Mn/Cr ratio of 1.0, i.e., no excess permanganate.

Due to interference from the analytical preparation method for solids dissolution, nickel was not measured in the solids and all conclusions were based on the result that nickel was not observed in the leachate solutions. Additional testing would be required to confirm these conclusions based on analysis of solids.

The Test Exception requested confirmatory tests with actual waste samples. However, based on the limited impact observed from the simulant studies, a decision was made by WTP to not carry out the actual waste tests. This was documented in an e-mail message from Fred Damerow on May 08, 2009.

Acknowledgements

The authors would like to thank Pacific Northwest National Laboratory staff members Wayne Cosby for his editorial support and Bruce McNamara for obtaining the uranium compounds used in this report. The authors also would like to thank Parameshwaran Sundar of Bechtel National Inc. for his technical insights and much helpful discussion and support.

1.0 Introduction

Oxidative leaching of simulant tank waste using permanganate as the oxidant in 0.25 M NaOH solutions reduces the chromium content of the sludge. An antifoam agent (AFA) is added to the waste treatment process to reduce foaming. The AFA, Dow Corning Q2-3183A, is a surface-active polymer that consists of polypropylene glycol, polydimethylsiloxane, octylphenoxy polyethoxy ethanol, treated silica, and polyether polyol. Some of the Hanford Tank Waste Treatment and Immobilization Plant (WTP) waste slurries contain high concentrations of undissolved solids that would exhibit undesirable behavior without AFA addition.

These tests were conducted to determine the effect of the AFA on oxidative leaching of Cr(III) in waste by permanganate. Previous studies have demonstrated that the Cr(III) oxidation can be used to produce soluble chromium (Cr(VI)) according to the reaction:

$$\mathrm{Cr(OH)_3 + MnO_4^- + \quad OH^-} \rightleftharpoons \mathrm{CrO_4^{2-} + MnO_2 + 2\,H_2O} \tag{1.1}$$

It has not previously been determined what effect AFA has on the permanganate reaction. This study was conducted to determine the effect AFA has on the oxidation of the chromium, plus plutonium and other criticality-related elements, specifically Fe, Ni, and Mn. During the oxidative leaching process, Mn is added as liquid permanganate solution and is converted to an insoluble solid that precipitates as MnO_2 and becomes part of the solid waste. Caustic leaching was performed followed by an oxidative leach at either 25°C or 45°C. The test conditions for these tests are shown in Table 1.1.

Table 1.1. Test Conditions for Radioactive Simulant Testing

Test Number	Temperature, °C	AFA Level, mg/L[(a)]	Mn:Cr
1	25	175	1.0
2	25	350	1.0
3	25	350	1.0
4[(b)]	25	700	1.0
5	45	350	1.0[(c)]
6	45	700	1.0[(c)]
7	45	350	1.0[(c)]
8[(b)]	25	0	1.0
9	45	700	1.0
10	25	700	1.0[(c)]

(a) AFA concentration in the feed simulant before caustic leaching step.
(b) Test carried out in triplicate to assess experimental variance.
(c) Test Exception called for 1.25 ratio.

Details of the testing are presented in the Experimental Section of this report. An overview is presented here. The simulant was prepared using a previously developed recipe and was aliquoted into individual test items. Water and sodium hydroxide were added to each test item to give a leachate volume equal to three times the simulant sludge volume with a caustic concentration of 3 M NaOH. AFA was mixed with a small quantity of NaOH solution and was added to the items as specified in Table 1.1.. The simulant and supernate/leachate were mixed for 24 hours at room temperature to equilibrate with the

3 M NaOH, after which the solids and liquids were sampled to determine the starting concentrations of elements of interest. These steps are shown in Figure 1.1.

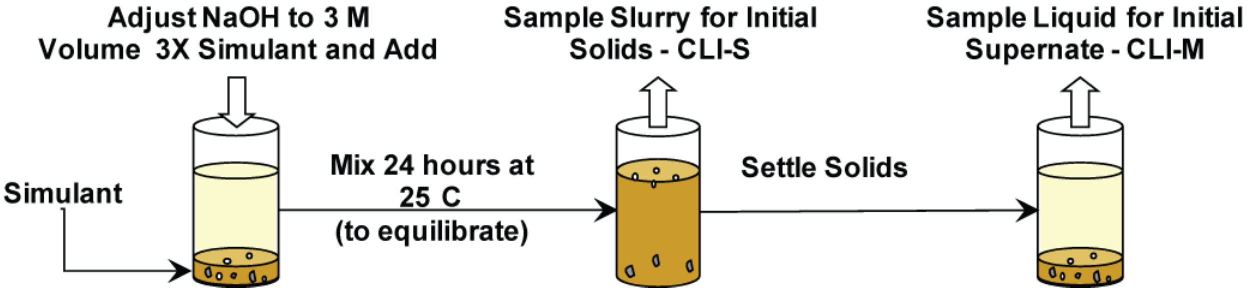

Figure 1.1. Initial Equilibration of Simulant Sludge with 3 M NaOH

Each test item was subjected to a caustic leach process by heating and mixing the items at 85±5°C for 8 hours. The caustic was 3 M NaOH. After 8 hours at 85±5 °C, the test items were cooled overnight to room temperature, and any lost water was replenished. The amount of water lost was less than 1 mL in all cases, so the caustic concentration increased by no more than 0.8% due to water loss. The samples were mixed, the solids were sampled and the supernatant leachate was separated from the solids. The solids were washed twice with 0.01 M NaOH and the wash solutions were added to the supernate. Previous testing for oxidative leaching used three washes and it was observed that the highly-colored chromate was mostly removed after two washes. Reducing the number of washes to two, reduced the combined volume of washes and supernate. The reduced volume inproved detectability of analytes present at very low concentrations. After thorough mixing, the supernate/wash solution was sampled. These steps are depicted in Figure 1.2.

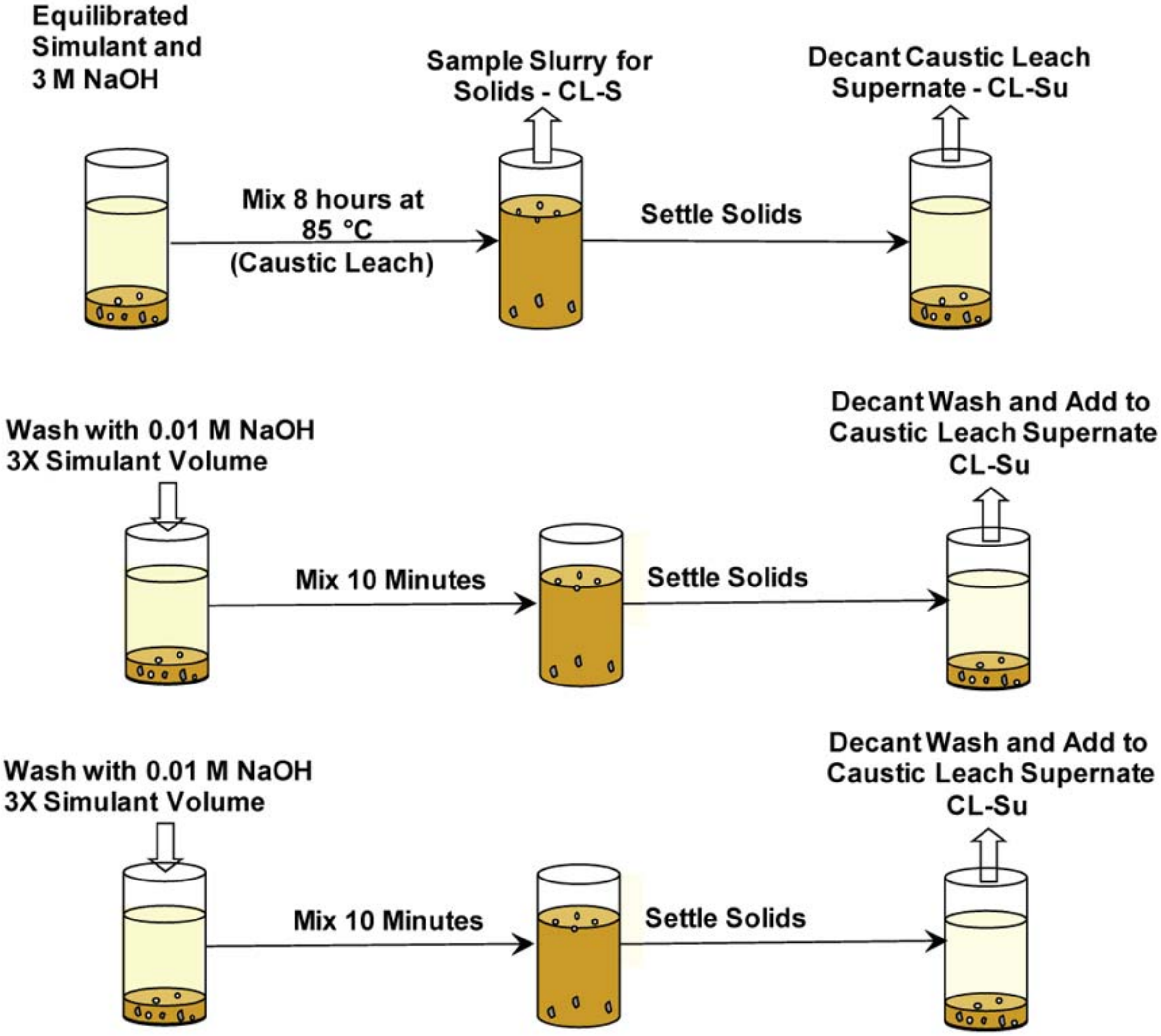

Figure 1.2. Caustic-Leach and Wash of Simulant Sludge

The caustic leached and washed solids were combined with water, NaOH, and $NaMnO_4$ to produce an oxidative-leach solution that contained 0.25 M NaOH and permanganate at a concentration to produce a 1:1 chromium to permanganate ratio. The test item was heated to the temperature specified in Table 1.1. for six hours with mixing. At the end of this time, the solids were settled and the supernate/leachate was decanted, the solids were washed twice with 0.01 M NaOH, and the wash solutions were combined with the oxidative-leach supernate. After mixing well, the leachate/wash solution was sampled. The solids were dried and submitted for analysis. The oxidative-leach steps are shown in Figure 1.3.

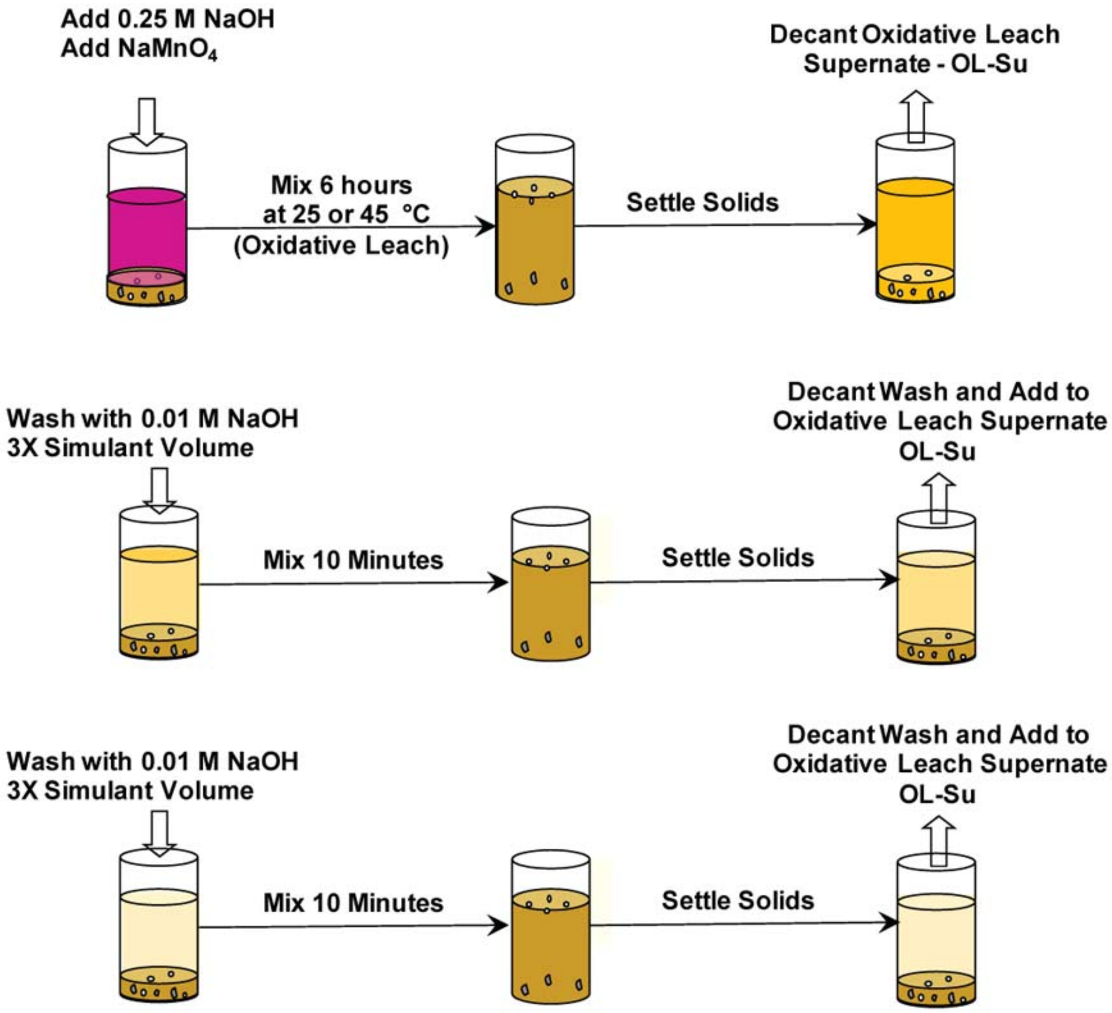

Figure 1.3. Oxidative-Leach Steps

In this document, the test item is a radioactive simulant solid material in liquid. The solid may be suspended into a slurry or settled into compacted solids. The settling may be accomplished by gravity or induced by centrifugation. When the maximum amount of settling has resulted in the minimum volume of solids, the solids are considered settled solids or compacted solids. Liquid is added to the solids for the purpose of either leaching material out with a leachate or washing the solids to remove interstitial leachate. When the solids are settled, the liquid may be referred to as either leachate or supernate interchangeably. Mixing settled solids and leachate or wash solution by agitation results in a slurry. When all of the liquid is removed, the residue is dried solids.

In summary, this report describes work focused on determining the effect of AFA on chromium oxidation by permanganate with Hanford sludge simulant.

2.0 Experimental Section

The experimental details relevant to the work described in this report are provided in this section, which begins with general equipment and analytical information. It then proceeds to the details of producing and characterizing the Cr(III)-containing solids to be used in this work.

2.1 General Experimental Information

All commercially-supplied chemicals were of reagent grade unless otherwise specified. The uranium and plutonium were obtained from in-house stores. The hydroxide concentrations in the stock sodium hydroxide solutions were verified by titration with primary standard acid solutions and were performed by the Radiochemical Processing Laboratory's (RPL's) Analytical Support Operations (ASO) using standard procedures. The permanganate concentrations in stock sodium permanganate solutions were verified by titration against standards-grade sodium oxalate according to a literature procedure (Jeffery et al. 1989).

The ASO at PNNL performed all sample analyses, hydroxide concentration determination by titration, uranium concentrations by inductively coupled plasma-optical emission spectroscopy (ICP-OES), plutonium and americium by alpha energy analysis (AEA), total metals by ICP-OES, anions by ion chromatography (IC), total inorganic carbon (TIC), and total organic carbon (TIC) by the hot persulfate method. The measurements using standard procedures were performed under statement of work SOW-RPP-WTP-QA-005.

2.2 Preparation of Radioactive Simulant

The composition of the solids constituents for preparing the radioactive simulant was in accordance with those described in a prior publication (Rapko et al. 2007). However, adding $Fe(OH)_3$ and Pu(IV) to the simulant sludge was accomplished differently than before, specifically by neutralizing a solution containing both $Fe(NO_3)_3$ and $Pu(NO_3)_4$ from an acidic medium with NaOH. Furthermore, it was not recommended that this coprecipitated $Fe(OH)_3 + Pu(OH)_4$ be washed to separate the nitrate anion. Instead, BNI accepted the small amount of this nitrate anion in the simulant supernate composition. At this point, the simulant supernate should consist only of hydroxide anion and a residual concentration of nitrate anion from Fe and Pu(IV) coprecipitation. This procedure for adding Pu(IV) deviated from the addition of $Pu(NO_3)_4$ solution into a heterogeneous mixture of $Fe(OH)_3$, CrO(OH), and other constituents in an alkaline medium. In this regard, the procedure for adding Pu(IV) follows that described in a prior WTP publication (Sinkov 2007). The chromium component added to prepare the simulant consisted of chromium oxyhydroxide $CrOOH$-nH_2O only. The CrOOH was added as a slurry without the drying and milling steps in its preparation described in the publication cited above (Rapko et al. 2007).

Table 2.1 provides information regarding the commercially procured chemicals used in preparing radioactive chromium leaching simulant.

Table 2.1. Commercially-Procured Phases Used in the Simulant

Chemical Phase	Chemical Formula	Manufacturer	Lot. No.
Ferric Nitrate	$Fe(NO_3)\cdot 9H_2O$	JT Baker	34460
Zinc Hydroxide	$Zn(OH)_2$	Wintersun Chemical	051201
Nickel Hydroxide	$Ni(OH)_2$	Alfa Aesar®	C24R009

2.2.1 Preparation of the Radioactive Chromium Leaching Simulant

The initial components of the simulant were combined in the amounts listed in Table 2.2. Plutonium nitrate, containing americium-241 in-grown from plutonium-241 decay, was mixed with ferric nitrate in a nitric acid solution. The iron and plutonium plus americium were coprecipitated by neutralizing the nitric acid with sodium hydroxide. The precipitated iron, plutonium and americium were combined with a mixture of U(IV) and U(VI) oxides that were mixed with the oxide/hydroxides of chromium, nickel, and zinc to form the simulant slurry used in testing. No manganese was added to the initial simulant preparation. During simulant characterization, some aliquots indicated the presence of traces of Mn that may have been present as impurities or were an analytical artifact of the complex sample matrix.

Table 2.2. Initial Components Combined for the Radioactive Simulant

Component	Amount Added, g	Amount As Oxide, g
CrO(OH)	14.917[(a)]	13.336
$Fe(OH)_3$	25.007[(b)]	19.046
$Ni(OH)_2$	0.7014	0.565
UO_2	0.563	0.563
β-U_3O_8	1.047	1.047
$Zn(OH)_2$	0.2241	0.219
$Pu(OH)_4$	0.022[(c)]	0.019
Total Mass:	42.4815	34.795

(a) The CrO(OH) was obtained as a slurry and the mass was calculated from characterization data.

(b) Iron was prepared as 131.25g of $Fe(NO_3)_3\cdot 9H_2O$ in 250 mL. Only 180 mL was used and it was neutralized by dropwise addition of NaOH.

(c) A solution containing 17 mg Pu(IV) in nitric acid was neutralized with NaOH and coprecipitated with $Fe(OH)_3$. The form of hydroxide or oxyhydroxide species was not determined.

The plutonium added to the test samples was weapons-grade material with the isotopic distribution shown in Table 2.3. The americium concentration was calculated based on the known plutonium isotopic distribution and observed ratio of the alpha activity of (^{238}Pu+^{241}Am) relative to (^{239}Pu+^{240}Pu). The alpha energy analysis (AEA) used to measure plutonium in the samples detects a single energy peak for (^{239}Pu+^{240}Pu) and another peak for (^{238}Pu+^{241}Am). The quantities of the individual isotopes cannot be directly determined by this technique. Data for both (^{239}Pu+^{240}Pu) and (^{238}Pu+^{241}Am) is presented in this report. Any systematic differences between the results for these can be attributed to different behavior by plutonium compared to americium.

Table 2.3. Isotopic Distribution for Plutonium Used in Simulant

Isotope	Isotopic Abundance (wt%)	Specific Alpha Activity (Ci/g)	Alpha Activity (Ci/g Pu)
^{238}Pu	0.0068	1.70E+01	1.16E-03
^{239}Pu	93.8957	6.20E-02	5.82E-02
^{240}Pu	6.0034	2.30E-01	1.38E-02
^{241}Pu	0.06924	1.00E+02	
^{242}Pu	0.0247	3.90E-03	9.63E-07
^{241}Am	0.0057	3.4	1.93E-04
^{238}Pu+^{241}Am	0.0124	--	1.35E-05
^{239}Pu+^{240}Pu	99.9	--	7.20E-04

2.2.2 Division of the Radioactive Simulant for Oxidative Leaching Experiments

The following steps were taken to aliquot test samples containing nearly equal amounts of waste simulant. The simulant slurry was mixed with an overhead mechanical mixer. Using a pipetter equipped with a tip that had been trimmed at the end to prevent plugging, 31 mL of the mixed slurry was transferred to each of fourteen 250-mL plastic bottles plus an extra bottle that contained approximately 27 mL of the mixed slurry. This amount of slurry was calculated to give 3.2 g of simulant solids in each bottle.

2.3 Caustic-Leach Preparation

The amount of reagents needed was calculated based on a 3:1 ratio of volume leachate to volume compacted solids. The radioactive simulant had an initial caustic concentration of 1.86 M. To attain the 3:1 leachate volume at a concentration of 3 M NaOH, deionized (DI) water was first added and followed by 10 M NaOH stock solution. To mix the leachate with the sludge simulant, the slurries with added leachate were mixed for at least 10 minutes on a shaker table at 200 rpm and room temperature. The volumes of compacted solids slurry and added leach solution are shown in Table 2.4.

Table 2.4. Caustic-Leach Volumes Used with Radioactive Simulant Testing

Test	Total Initial Slurry Volume (mL)	Total Volume Leach Solution (mL)
1	31.58	126
2	30.79	123
3	31.20	125
4a	31.03	124
4b	31.09	124
4c	31.19	125
5	30.68	123
6	31.02	124
7	30.72	123
8a	30.91	124
8b	31.07	124
8c	30.71	123
9	31.09	124
10	30.71	123

2.4 Test Sample Preparation and Characterization

The AFA testing used Dow Corning® Q2-3183A AFA at levels of 0 to 700 ppm (0 to 700 mg/L). This AFA consists of polypropylene glycol, polydimethylsiloxane, octylphenoxy polyethoxy ethanol, treated silica, and polyether polyol. The designated amount of AFA was added to each test slurry as indicated in Table 2.5. The target was based on a multiplier to produce a final concentration of AFA in mg/L of slurry. The WTP process uses a nominal 350 ppm AFA concentration. The selected test concentrations are equal to 0, 0.5, 1, and 2 times the plant concentration. Table 2.5 contains the specific amounts of AFA added to each test item.

Table 2.5. AFA Concentrations in Radioactive Simulant

Test	Total Slurry Volume (mL)	AFA target (mg/L)	AFA added (mg)
1	31.58	175	5.5
2	30.79	350	10.8
3	31.20	350	10.9
4a	31.03	700	21.7
4b	31.09	700	21.8
4c	31.19	700	21.8
5	30.68	350	10.7
6	31.02	700	21.7
7	30.72	350	10.8
8a	30.91	0	0.0
8b	31.07	0	0.0
8c	30.71	0	0.0
9	31.09	700	21.8
10	30.71	700	21.5

The test mixtures containing sludge simulant, 3 M NaOH leachate, and AFA at the targeted level were placed on an orbital shaker table at room temperature, and the slurries were agitated at 200 rpm for 24 hours. Solids were allowed to settle until a clear supernate was observed. Samples of the supernate were collected and filtered. From each test item, one sample was submitted for free hydroxide, anions by IC, TIC, and TOC analyses. A second sample of the supernate was submitted for metals analysis by ICP-OES and plutonium (Pu) by AEA. The slurries were mixed vigorously, and a third sample of the solids was collected as a slurry. The solids were washed with 0.01 M NaOH, dried, and analyzed for metals and Pu. The washing of these solids was incomplete because of the small sample size and the solids that settled poorly.

The test containers were loosely capped and placed in the heated shaker table. Slurries were agitated by rotary shaking at 200 rpm and heated at 85°C±5°C for 8 hours. After 8 hours at 85±5°C, the test items were cooled overnight to room temperature, and any lost water was replenished. The amount of water lost was less than 1 mL in all cases, so the caustic concentration increased by no more than 0.8% due to water loss. The samples were mixed, evaporated liquid was replenished with DI water, stirring was stopped, and the sample was allowed to settle. Supernate was decanted into tared plastic bottles and weighed. The leached solids remaining in each caustic leachate bottle were washed by adding 0.01 M NaOH (in an equivalent amount to the leach solution) into each test container and agitated for at least 10 minutes, and again the solids were allowed to settle. The wash solution was decanted into the appropriate tared plastic bottle containing the previous supernate. The wash with 0.01 M NaOH was repeated for a second wash.

After agitating each supernate- and washes-containing bottle for at least 5 minutes at 200 rpm on the shaker table to confirm that the contents were well mixed, an aliquot of sample from each bottle was removed and filtered through a 0.2-μm syringe filter into a vial. From each test caustic-leach item, one sample was submitted for free hydroxide, TIC, and TOC analyses. A second sample of the supernate was submitted for metals analysis by ICP-OES and Pu by AEA. The slurries were mixed vigorously, and a third sample of the solids was collected as a slurry. The solids were washed with 0.01 M NaOH, dried, and analyzed for metals and Pu. Concentrations of key components in the radioactive simulant are shown in the Results section. All of the ICP results for the initial sample characterization are reported in Appendix B.

2.5 Oxidative-Leach Procedure

The amount of reagents needed was calculated based on a 3:1 volume leachate to volume compacted solids for each oxidative-leach test. The amount of NaOH needed to achieve the desired 0.25 M NaOH concentration was calculated based on the amounts of residual 3 M NaOH and volumes of 0.01 M NaOH wash solution used in the caustic-leach and wash steps. Water was added first, then the 10 M NaOH to achieve a final concentration of 0.25 M NaOH, and then 0.967 M $NaMnO_4$ stock solution was added to yield a Mn:Cr ratio of 1.0. The measured amounts of chromium present in each test item and the amount of permanganate added is shown in Table 2.6. Each test solution was agitated at the target temperature of 25°C or 45°C for 6 hours. Each reaction bottle was centrifuged for at least 5 minutes, and each supernate was decanted into a labeled and tared plastic bottle. The leached solids remaining in each caustic leachate bottle were washed by adding 0.01 M NaOH (in an equivalent amount to the leach solution) into each test container and agitated for at least 10 minutes. The wash solution was at ambient temperature. The sample was centrifuged for at least 5 minutes, and the supernate was decanted into the appropriate tared plastic bottle containing the previous supernate. The washing process was repeated for a second wash.

Table 2.6. Quantities of Chromium and Manganese Present in Test Items at Beginning of Oxidative Leaching of Simulated Tank Waste Solids

Test	AFA target (mg/L)	Initial Mass Cr in Solids (µg)	Initial Cr (moles)	Initial Mass Mn (µg)[(a)]	MnO_4^- Added (moles)	Mass Mn Added (µg)	Cr:Mn Molar Ratio
1	175	540,000	0.010	1,100	0.011	610,000	0.95
2	350	530,000	0.010	1,100	0.011	610,000	0.93
3	350	570,000	0.011	930	0.011	610,000	0.99
4a	700	570,000	0.011	2,000	0.011	610,000	1.00
4b	700	570,000	0.011	1,400	0.011	610,000	0.99
4c	700	570,000	0.011	900	0.011	610,000	0.99
5	350	580,000	0.011	3,500	0.011	610,000	1.00
6	700	770,000	0.015	[560]	0.011	610,000	1.30
7	350	560,000	0.011	[380]	0.011	610,000	0.98
8a	0	560,000	0.011	[240]	0.011	610,000	0.98
8b	0	560,000	0.011	[70]	0.011	610,000	0.98
8c	0	550,000	0.011	<72	0.011	610,000	0.96
9	700	700,000	0.013	<60	0.011	610,000	1.20
10	700	550,000	0.011	<40	0.011	610,000	0.95

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

After agitating each bottle containing supernate and washes to confirm that the contents were well mixed, an aliquot of sample from each bottle was removed and filtered through a 0.2-µm syringe filter into a vial. The solution density was determined by measuring the weight of three 1 mL aliquots of these filtered solutions. An aliquot from each filtered solution was transferred into a tared and labeled 20 mL glass liquid scintillation counting (LSC) vial containing 9 mL of 1 M HNO_3 for ICP and Pu (by AEA) analysis. Another aliquot from each filtered solution was submitted for hydroxide, TOC, and TIC analysis.

The centrifuged solids were dried to incipient dryness at 80°C and then dried at 105°C overnight. The solids were submitted for potassium hydroxide (KOH) fusion, metals content by ICP-AES, Pu (by AEA), TIC, and TOC.

2.6 Quality Assurance

PNNL's QA program is based on requirements defined in DOE Order 414.1C, Quality Assurance and 10 CFR 830, Energy/Nuclear Safety Management, Subpart A—Quality Assurance Requirements (a.k.a. the Quality Rule). As of September 2008, PNNL has chosen to implement the following consensus standards in a graded approach:

- ASME NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*, Part 1, *Requirements for Quality Assurance Programs for Nuclear Facilities*.
- ASME NQA-1-2000, Part II, Subpart 2.7, *Quality Assurance Requirements for Computer Software for Nuclear Facility Applications.*
- ASME NQA-1-2000, Part IV, Subpart 4.2, *Graded Approach Application of Quality Assurance Requirements for Research and Development.*

PNNL implements these requirements by integrating them into the laboratory's management systems and daily operating processes. The procedures necessary to implement the requirements are documented through PNNL's "How do I…?" (HDI).(a)

PNNL implemented the RPP-WTP quality requirements by performing work in accordance with the *River Protection Project—Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Plan* (RPP-WTP-QA-001, QAP). Work was performed to the quality requirements of NQA-1-1989 Part I, Basic and Supplementary Requirements, NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD).* These quality requirements are implemented through the *River Protection Project—Waste Treatment Plant Support Program (RPP-WTP) Quality Assurance Manual* (RPP-WTP-QA-003, QAM). The analytical requirements are implemented through RPP-WTP's Statement of Work (RPP-WTP-QA-005) with the RPL-ASO.

The quality assurance requirements of DOE/RW-0333P, Rev 13, *Quality Assurance Requirements and Descriptions (QARD)* and DOE Order 414.1C were not identified as a requirement for this work in the Test Specification.

2.7 Conduct of Experimental and Analytical Work

Experiments that were not method-specific were performed in accordance with PNNL's procedures QA-RPP-WTP-1101 "Scientific Investigations" and QA-RPP-WTP-1201 "Calibration Control System,"

(a) System for managing the delivery of laboratory-level policies, requirements, and procedures.

verifying that sufficient data were taken with properly calibrated measuring and test equipment (M&TE) to obtain quality results.

A test guidance, 53019-AFA-1 “Alkaline Oxidative Leaching of Cr from a Hanford Tank Waste Simulant” was used to describe the work to be performed. Operational aspects of this work were governed by procedure RPL-OP-001 and the applicable permits and supporting procedures referenced therein.

Reported hydroxide analyses were performed by RPL-ASO according to procedure RPG-CMC-228, Rev 1. No QC issues were associated with these data.

Solution metal concentrations (including uranium) were determined by ICP-OES according to procedure RPG-CMC-211. The controlling documents for these analyses were ASO-QAP-001 and RPP-WTP-QA-005, Rev 3. All QC checks were within the acceptance criteria.

Reported metals and uranium for the solids from oxidative leaching of the radioactive simulant were dissolved according to PNL-ALO-115 and analyzed by ICP-OES according to RPG-CMC-211. No QC issues were associated with these data.

Radionuclides were obtained for solids and prepared solutions. Solids were prepared according to PNL-ALO-115. Plutonium was measured according to RPG-CMC-496, Rev 0 and RPG-CMC-422, Rev 2. No QC issues were associated with these data.

Anions by IC were measured using PNL-ALO-212. Samples were prepared by bench dilution. There were no QC issues associated with these data.

TIC and TOC were determined using a hot persulfate method according to RPG-CMC-385 Rev 0. No QC issues were associated with these data.

Additional M&TE that were used included clocks, programmable heater/stirrers, tachometers, thermocouple-meters, and balances. The clocks, tachometers, and programmable heater-stirrers were standard laboratory equipment for use as indicators only. The thermocouple-meter combination was calibrated by the PNNL Instrument Calibration Facility. The thermometers were calibrated June 2008. Balances are calibrated annually by a certified contractor, QC Services, Portland, Oregon. A balance performance check was conducted each day the balance was used.

2.8 Internal Data Verification and Validation

PNNL addressed internal verification and validation activities by conducting an independent technical review of the final data report in accordance with PNNL’s procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and the reported work satisfied the Test Plan objectives. This review procedure is part of PNNL’s RPP-WTP Quality Assurance Manual.

3.0 Results and Discussion

3.1 Test Item Characterization

The initial concentrations of elements of interest were determined after equilibrating the simulant tank waste slurries in 3 M NaOH containing the specified amounts of AFA. Amounts of key components in the radioactive simulant are shown in Table 3.1. These masses are the amounts of each component that were present in the approximately 3.2 grams of sample used in each test. Although no Mn was included in the simulant recipe, some of the samples tested positive for small amounts of Mn. These results show a wide variability and are believed to be either trace contaminants in the chemicals used for simulant or artifacts of the analytical method. The samples were prepared using a KOH-KNO_3 fusion in a nickel crucible, so nickel concentrations were not obtained for the solid samples. Nickel concentrations in the supernate were all below detection limits.

Table 3.1. Measured Amounts of Key Components in the Radioactive Simulant Solids Equilibrated with 3 M NaOH Before Heating for Caustic Leaching

Test	Cr (µg)	Fe (µg)	Mn(a) (µg)	Ni(b) (µg)	U (µg)	$^{239+240}$Pu (µCi)	^{238}Pu + ^{241}Am (µCi)
1	540,000	750,000	5,000	30,000	[9,100]	63	1.3
2	530,000	740,000	7,600	30,000	[8,000]	68	1.9
3	570,000	800,000	4,900	30,000	[4,100]	63	1.3
4a	570,000	770,000	11,000	30,000	[8,900]	77	1.9
4b	570,000	760,000	6,700	30,000	[9,300]	69	1.7
4c	570,000	790,000	5,300	30,000	[6,600]	68	1.8
5	580,000	800,000	20,000	30,000	[11,000]	70	1.7
6	770,000	1,000,000	[4200]	30,000	[18,000]	92	2.5
7	560,000	750,000	[2300]	30,000	[7,600]	68	1.7
8a	560,000	750,000	[1500]	30,000	[12,000]	73	2.3
8b	560,000	740,000	[440]	30,000	[14,000]	69	1.7
8c	550,000	720,000	<690	30,000	[10,000]	67	1.8
9	700,000	930,000	<370	30,000	[22,000]	79	1.9
10	550,000	720,000	<220	30,000	[11,000]	69	2.0
Average	580,000	790,000	5,000	-	11,000	71	1.8
Stdev	65,000	84,000	680	-	4,700	7	0.3
RSD	11%	11%	120%	-	43%	18%	17%

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

(b) Ni crucibles were used in the analytical fusions procedure to dissolve solids samples for analysis. These values are calculated based on simulant makeup and solution analyses.

The equilibrated leach solution for each slurry was filtered and analyzed for key components. For all of the leach solutions, the nickel concentration was below the instrument detection limit. Values for analytes are shown in Table 3.2. These quantities represent the total amount of each component in each test item containing approximately 125 mL of test slurry and leachate.

Table 3.2. Initial Amounts of Key Components in Equilibrated Leachate Solution Before Heating for Caustic Leaching[(a)]

Test	Cr (μg)	Fe (μg)	Mn (μg)	Ni (μg)	U (μg)	$^{239+240}$Pu (μCi)	^{238}Pu + ^{241}Am (μCi)
1	2,300	[16]	<2.7	<68	[2,900]	4.5E-2	8.9E-4
2	2,200	[28]	<2.6	<67	[2,500]	4.9E-2	9.6E-4
3	2,100	[34]	<2.6	<67	[2,900]	2.5E-1	4.9E-3
4a	2,200	[22]	<2.6	<67	[2,800]	4.5E-2	7.8E-4
4b	2,200	[21]	<2.6	<67	[2,800]	5.0E-2	9.1E-4
4c	2,100	[33]	<2.6	<67	[2,800]	9.3E-2	1.7E-3
5	2,100	<1.3	<2.6	<67	[2,400]	5.1E-2	9.1E-4
6	2,200	[18]	<2.6	<67	[2,500]	5.5E-2	1.1E-3
7	2,200	[18]	<2.6	<67	[2,800]	5.3E-2	9.6E-4
8a	2,200	[20]	<2.6	<68	[3,000]	5.5E-2	9.8E-4
8b	2,200	[21]	<2.6	<67	[3,000]	5.1E-2	9.6E-4
8c	2,200	[18]	<2.6	<67	[2,900]	5.3E-2	9.0E-4
9	2,200	<1.3	<2.6	<68	[3,200]	4.9E-2	8.4E-4
10	2,200	[26]	<2.6	<67	[3,000]	5.1E-2	1.0E-3
Average	2,200	20			2,800	6.8E-2	1.3E-3
Stdev	53	10			240	5.5E-2	1.1E-3
RSD	2%	48%			8%	80%	84%

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Two triplicate samples (total six test items) were tested to determine variability in the method. These triplicates were test items 4a-c that contained AFA at the 700 ppm level and items 8a-c that contained no AFA. Results, averages, standard deviations, and relative standard deviations for each of the elements of interest in the initial test item solids equilibrated with 3 M NaOH are shown in Table 3.3.

Table 3.3. Statistical Analysis of Triplicate Test Items at Two AFA Concentration Levels in the Simulant

		Quantities Measured in Initial Supernate						
AFA Level (mg/L)		Cr (µg)	Fe (µg)	Mn (µg)	U (µg)	Zn (µg)	$^{239+240}$Pu (µCi)	^{238}Pu+^{241}Am (µCi)
0	Average	2,300	20	0	3,094	368	5.3E-02	9.4E-04
	Stdev	83	1	0	158	3	2.2E-03	4.3E-05
	RSD	3.6%	5.6%	0.0%	5.1%	0.8%	4.2%	4.5%
700	Average	2,100	25	0	2,707	317	6.2E-02	1.1E-03
	Stdev	100	6	0	82	45	2.6E-02	4.8E-04
	RSD	4.8%	23.8%	0.0%	4.8%	14.2%	42.5%	43.0%

		Quantities Measured in Initial Solids						
AFA Level (mg/L)		Cr (µg)	Fe (µg)	Mn (µg)	U (µg)	Zn (µg)	$^{239+240}$Pu (µCi)	^{238}Pu+^{241}Am (µCi)
0	Average	560,000	735,639	126	19,000	6,800	70	1.9
	Stdev	7,200	10,575	95	1,900	670	3.2	0.34
	RSD	1.3%	1.4%	75.3%	10%	9.7%	4.5%	18%
700	Average	570,000	770,000	1,400	12,000	7,200	71	1.8
	Stdev	3,000	15,000	540	2,100	390	0.11	0.11
	RSD	0.5%	1.9%	37%	17%	5.5%	7.0%	5.9%

Samples of the filtered supernate were collected after the simulant slurry had been equilibrated with the 3 M NaOH leachate, but before the simulant was heated for the caustic-leach. A separate aliquot of the same initial equilibrated leachate solution was submitted for anions, hydroxide and TIC/TOC analysis. The results of the supernate analyses are shown in Table 3.4. The hydroxide concentration in Test 3 was high because of an error during initial leachate preparation.

Table 3.4. Anions, Hydroxide, Inorganic and Organic Carbon in Supernate Before Heating for Caustic-Leach

Test	Cl^-, ppm	NO_3^-, ppm	OH^-, M	TIC, µg C/mL	TOC, µg C/mL
1	1,500	31,000	2.55	710	710
2	1,500	31,000	2.55	710	770
3	2,100	25,000	4.59	1900	<560
4a	1,400	31,000	3.06	710	1,500
4b	1,400	30,000	2.55	610	<560
4c	1,700	29,000	3.57	610	<560
5	1,500	31,000	3.06	920	<560
6	1,500	31,000	2.55	1,000	<560
7	1,500	31,000	3.06	1,100	<1,600
8a	1,500	31,000	3.06	510	<1,600
8b	1,500	31,000	3.06	560	<1,600
8c	1,700	31,000	3.06	870	<1,600
9	1,500	31,000	3.06	410	<1,600
10	1,500	31,000	3.06	260	<1,600

Carbon was measured as TIC and TOC with the results reported in Table 3.5. Organic carbon concentration was below the detection limit of the analytical method.

Table 3.5. Inorganic and Organic Carbon in Solids Before Caustic-Leach

Test	TIC, µg	TOC, µg
1	63,000	<5,000
2	61,000	<3,600
3	80,000	<5,100
4a	51,000	<4,100
4b	41,000	20,000
4c	58,000	<4,700
5	56,000	<4,600
6	220,000	<200
7	320,000	<11,000
8a	200,000	<7,700
8b	61,000	<8,700
8c	53,000	<7,400
9	230,000	<24,000
10	120,000	<12,000

Some of the test items equilibrated with 3 M NaOH may be seen in Figure 3.1. The solids in this image are not completely settled.

Figure 3.1. Some of the Test Items Equilibrated with 3 M NaOH Before Heating for Caustic-Leach

3.2 Caustic-Leach Results

Table 3.6 shows the quantities of key components that partition to the caustic leachate after heating the sample slurry-leachate mixtures to 85±5°C for 8 hours. These quantities are based on the analysis of the supernate solution combined with the wash solutions. The uranium values in the leachate are unrealistically high compared to the initial solids uranium value. The initial solids analysis for uranium returned a value that averaged 20% of the calculated preparation concentration. As will be seen later for the final solids analysis, the final uranium value is in line with expected values based on the simulant preparation. It must be concluded that there was a strong bias in either the sample collection or the sample analysis for the initial value of uranium in the slurry solids.

Manganese and nickel were not detected in any of the initial equilibrated or the caustic leachate solutions. The detection limit for the method combined with the leachate volumes are included in Table 3.6 and have had the sample dilution factors applied.

Table 3.6. Amounts of Key Components in Caustic Leachate

Test	Cr (μg)	Fe[a] (μg)	Mn (μg)	Ni (μg)	U (μg)	Zn (μg)	$^{239+240}$Pu (μCi)	^{238}Pu + ^{241}Am (μCi)
1	4,500	[160]	<4.9	<130	[3,200]	[930]	3.6E-03	1.1E-04
2	4,900	[83]	<5.0	<130	[3,500]	[1,000]	3.8E-03	1.0E-04
3	4,900	[81]	<4.9	<130	[4,400]	[590]	5.8E-03	1.1E-04
4a	3,700	[49]	<5.4	<140	[4,400]	[790]	6.0E-03	1.4E-04
4b	3,100	[52]	<4.6	<120	[3,000]	[590]	4.3E-03	1.1E-04
4c	4,100	[62]	<4.9	<130	[4,200]	[640]	6.8E-03	9.9E-05
5	4,300	[76]	<4.9	<130	[3,500]	[790]	4.3E-03	1.2E-04
6	4,300	[67]	<4.9	<130	[3,500]	[770]	3.6E-03	8.1E-05
7	2,800	[53]	<4.4	<110	[3,700]	[680]	6.4E-03	9.9E-05
8a	3,200	[82]	<5.0	<130	[4,000]	[870]	6.0E-03	1.2E-04
8b	3,800	[54]	<4.5	<110	[5,400]	[450]	4.4E-03	6.3E-05
8c	4,000	<23	<4.7	<120	[3,100]	[420]	3.3E-03	5.7E-05
9	4,300	[82]	<4.2	<110	[3,600]	[740]	3.5E-03	6.1E-05
10	2,800	[59]	<4.7	<120	[3,800]	[630]	1.0E-02	1.7E-04

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

The fractions of each key element removed by the caustic-leach of the simulant tank waste are shown in Figure 3.2. These results are from AEA, which cannot distinguish between ^{238}Pu and ^{241}Am, so the results are reported together. Caustic leaching removed less than 1% of all elements with the exceptions of uranium and zinc. A second graph of the elements in the lower 1% range is shown in Figure 3.3 with the scale expanded and converted to a logarithmic scale. Manganese and nickel concentrations are not shown in either figure because their concentrations were below the method detection limit.

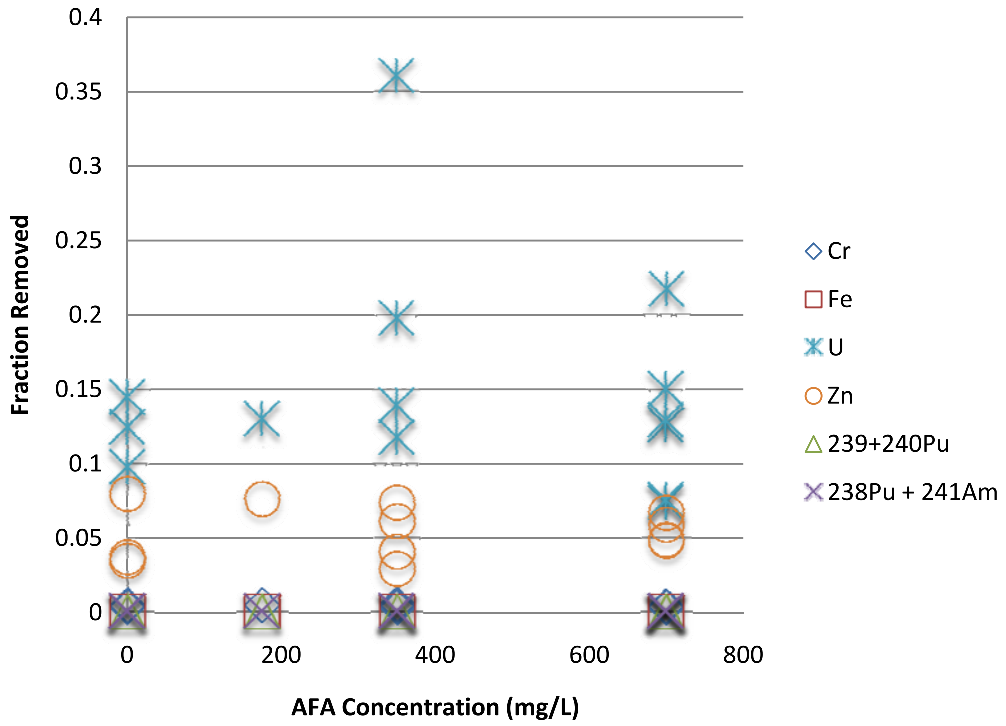

Figure 3.2. Impact of AFA Concentration on Key Elements During Caustic Leaching

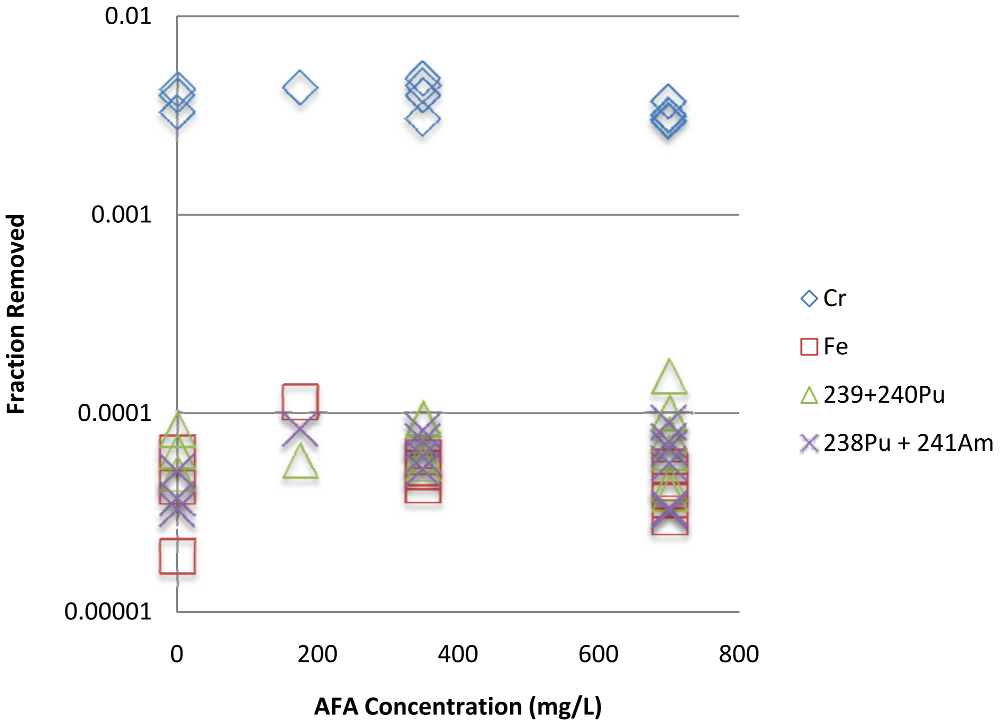

Figure 3.3. Expanded Caustic-Leach Results

3.3 Oxidative-Leach Results

3.3.1 Chromium Results

Removal of chromium from simulated tank sludge by oxidative leaching resulted in 83 to 97% of initial chromium being removed as seen in Table 3.7. These values are based on analysis of washed and dried solids.

Table 3.7. Chromium Removal from Simulated Tank Sludge by Oxidative Leaching

Test	Initial Cr (µg/g)	Initial Total Cr (µg)	Final Cr (µg/g)	Final Total Cr (µg)	Fraction Cr Removed
1	120,000	540,000	29,000	90,000	0.83
2	79,000	530,000	37,000	110,000	0.79
3	110,000	570,000	22,000	65,000	0.89
4a	110,000	570,000	23,000	68,000	0.88
4b	120,000	570,000	27,000	81,000	0.86
4c	97,000	570,000	8,800	26,000	0.95
5	100,000	580,000	6,300	19,000	0.97
6	100,000	770,000	8,000	24,000	0.97
7	92,000	560,000	8,700	27,000	0.95
8a	86,000	560,000	7,800	24,000	0.96
8b	88,000	560,000	8,800	27,000	0.95
8c	57,000	550,000	7,200	22,000	0.96
9	110,000	700,000	10,000	31,000	0.96
10	99,000	550,000	6,700	21,000	0.96

It should be noted that, although most of the chromium is leached from the waste, the overall mass of waste does not decrease because MnO_2 replaces the chromium in the solids.

Evaluation of the chromium removal from the solids as a function of AFA concentration is summarized in Table 3.8 and the fraction chromium removed as a function of AFA concentration is shown in Figure 3.4. A least square fit of the data at 25°C yields $y = -3E\text{-}05x + 0.915$ with $R^2 = 0.0275$ and at 45°C yields $y = 8E\text{-}06x + 0.9565$ with $R^2 = 0.0391$. These data show no correlation between the concentration of AFA and the effectiveness of the oxidative leaching for removal of chromium from the simulated tank sludge.

The concentration of chromium in the solids initially present in the simulated tank waste and the amount of chromium remaining after oxidative leaching is shown in Figure 3.5 for the 25°C tests and in Figure 3.6 for the 45°C tests.

Table 3.8. Summary of Cr Removal by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Fraction Cr Removed	Data Points	Average	Stdev	RSD
25°C	0	0.95				
25°C	0	0.96				
25°C	0	0.96	3@25, 0	0.96	0.0042	0.44%
25°C	175	0.83	1@25,175	0.83	NA	NA
25°C	350	0.79				
25°C	350	0.89	2@25, 350	0.84	0.066	7.9%
25°C	700	0.86				
25°C	700	0.88				
25°C	700	0.95				
25°C	700	0.96	4@25, 700	0.91	0.052	5.7%
45°C	350	0.95				
45°C	350	0.97	2@45, 350	0.96	0.011	1.1%
45°C	700	0.96				
45°C	700	0.97	2@45, 700	0.96	0.0093	0.96%

NA = Not Applicable

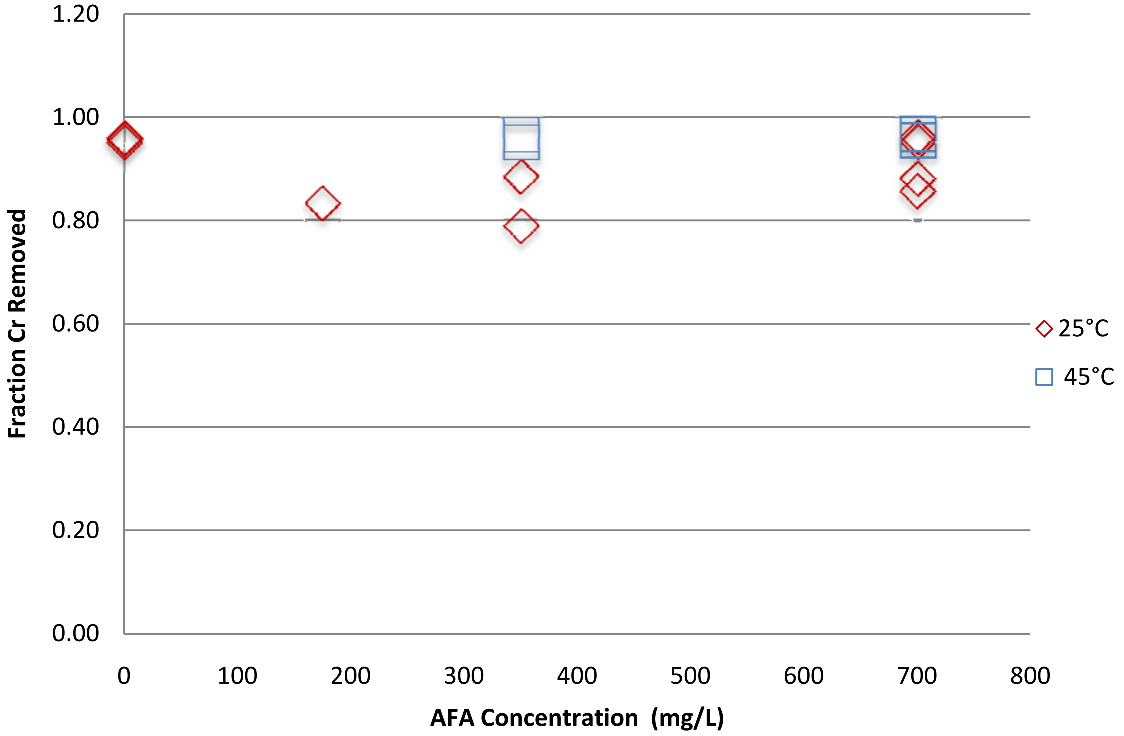

Figure 3.4. Chromium Removed from Simulated Tank Sludge by Oxidative Leaching with Permanganate

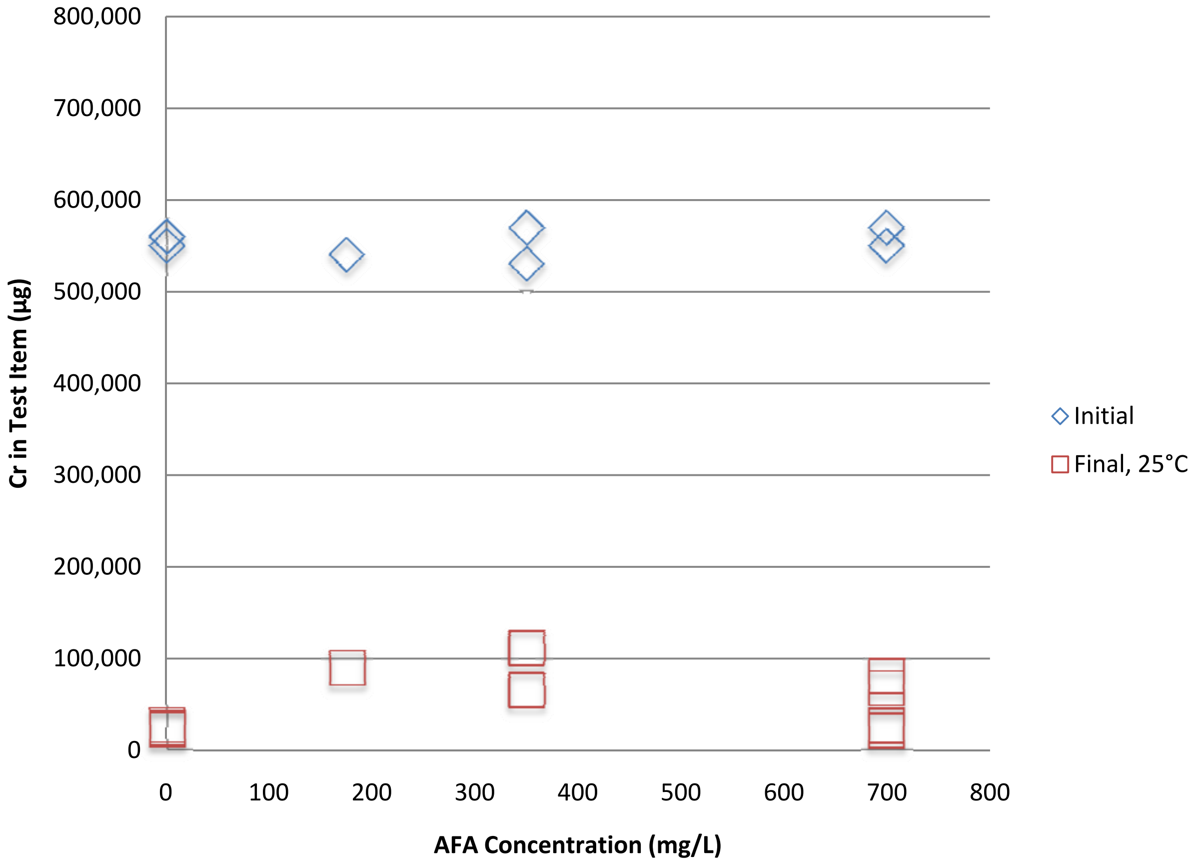

Figure 3.5. Quantity of Chromium in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 25°C

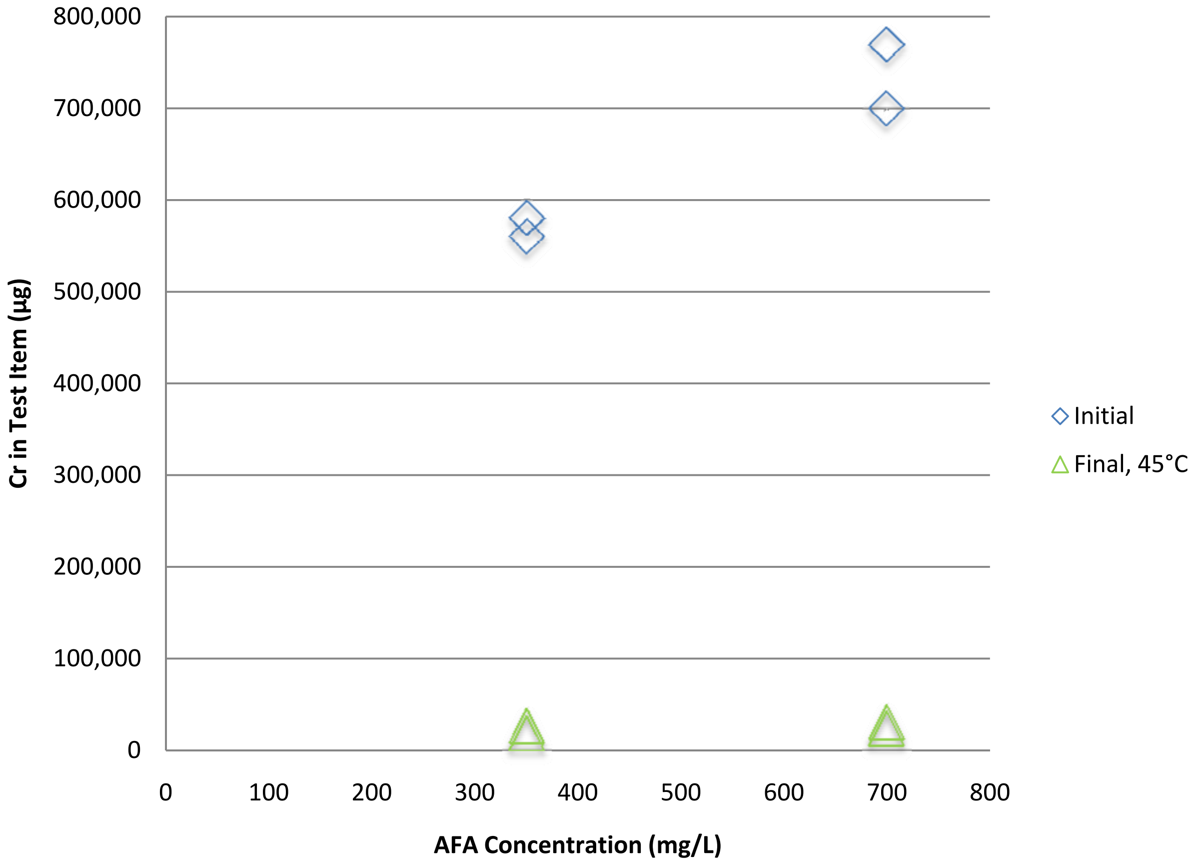

Figure 3.6. Quantity of Chromium in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 45°C

3.3.2 Iron Results

Removal of iron from simulated tank sludge by oxidative leaching resulted in 0.0032% to 0.026% of initial iron being removed as seen in Table 3.9. These values are based on analysis of leachate solution compared to washed and dried residual solids.

Table 3.9. Iron Removal from Simulated Tank Sludge by Oxidative Leaching

Test	Initial Fe (µg/g)	Initial Total Fe (µg)	Final Fe (µg/g)	Final Total Fe (µg)	Fraction Fe Removed
1	160,000	750,000	260,000	790,000	0.000060
2	110,000	740,000	230,000	680,000	0.000086
3	150,000	800,000	250,000	760,000	0.000092
4a	140,000	770,000	280,000	830,000	0.000061
4b	160,000	760,000	240,000	740,000	0.000062
4c	140,000	790,000	250,000	760,000	0.000032
5	150,000	800,000	260,000	790,000	0.000091
6	140,000	1,000,000	240,000	700,000	0.000046
7	120,000	750,000	250,000	760,000	0.000043
8a	110,000	750,000	250,000	750,000	0.000049
8b	120,000	740,000	250,000	780,000	0.000042
8c	76,000	720,000	200,000	590,000	0.000049
9	150,000	930,000	260,000	810,000	0.00026
10	130,000	720,000	250,000	750,000	0.000045

Evaluation of the iron removal as a function of AFA concentration is summarized in Table 3.10 and the fraction iron removed as a function of AFA concentration is shown in Figure 3.7. A least square fit of the data at 25°C yields $y = 3E\text{-}10x + 6E\text{-}05$ with $R^2 = 2E\text{-}05$ and at 45°C yields $y = 2E\text{-}07x - 2E\text{-}05$ with $R^2 = 0.2343$. These data show no correlation between the concentration of AFA and removal of iron by oxidative leaching of the simulated tank sludge.

The concentration of iron initially present in the simulated tank waste and the amount of iron remaining after oxidative leaching is shown in Figure 3.8 for the 25°C tests and in Figure 3.9 for the 45°C tests.

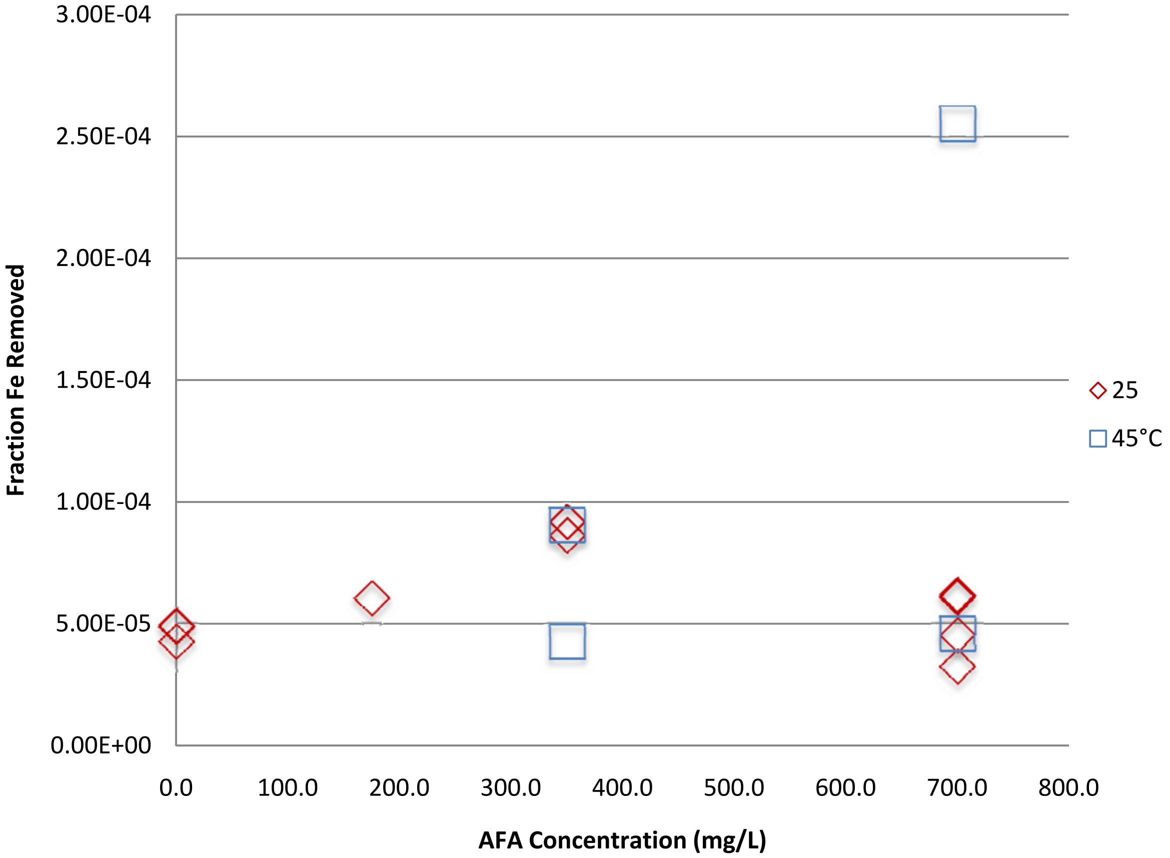

Figure 3.7. Fraction of Fe Removed by Oxidative Leaching at Either 25°C or 45°C

Table 3.10. Summary of Fe Removal by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Fraction Fe Removed	Data Points	Average	Stdev	RSD
25°C	0	4.85E-05				
25°C	0	4.24E-05				
25°C	0	4.89E-05	3@25,0	4.66E-05	3.67E-06	7.9%
25°C	175	6.03E-05	1@25,175	6.03E-05	NA	NA
25°C	350	8.61E-05				
25°C	350	9.17E-05	2@25, 350	8.89E-05	3.95E-06	4.4%
25°C	700	6.07E-05				
25°C	700	6.16E-05				
25°C	700	3.22E-05				
25°C	700	4.51E-05	4@25, 700	5.41E-05	1.91E-05	35.3%
45°C	350	9.05E-05				
45°C	350	4.26E-05	2@45, 350	6.65E-05	3.39E-05	51.0%
45°C	700	4.58E-05				
45°C	700	2.56E-04	2@45, 700	1.51E-04	0.000148	98.4%

NA = Not Applicable

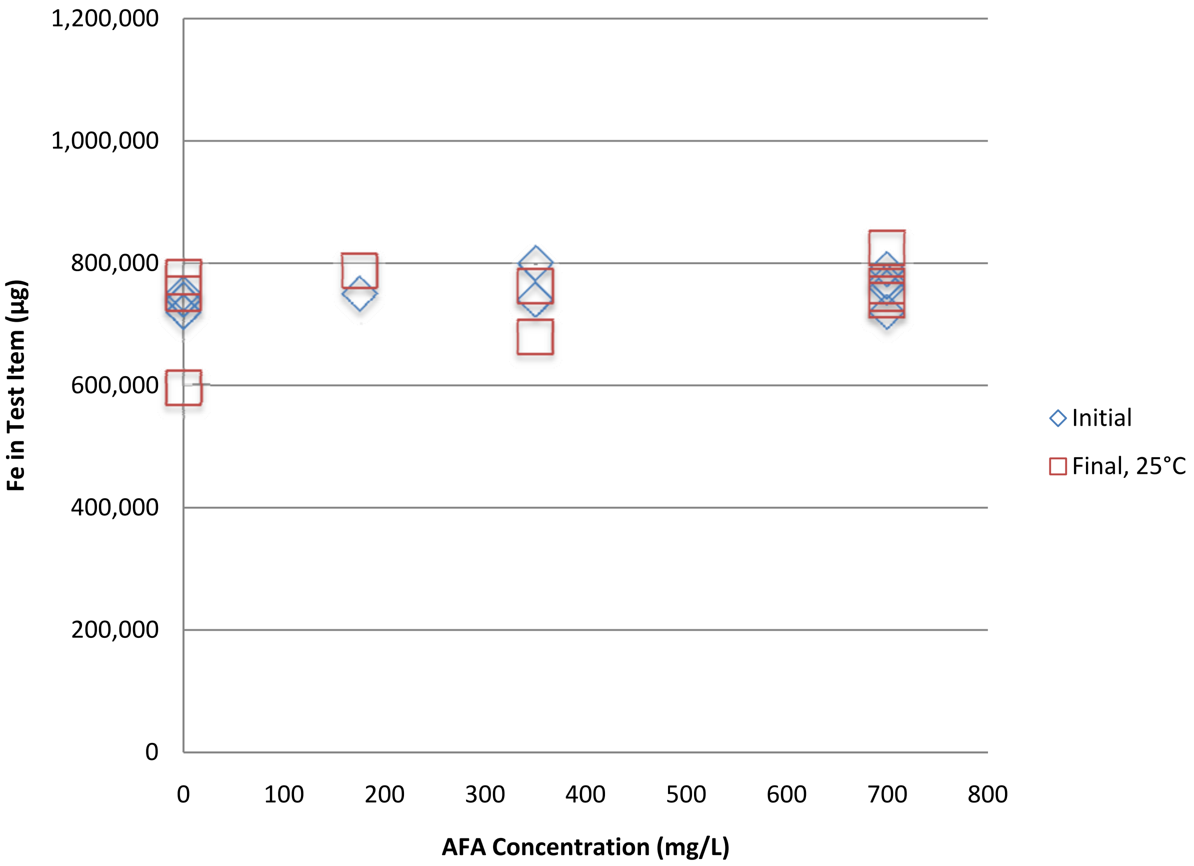

Figure 3.8. Quantity of Iron in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 25°C

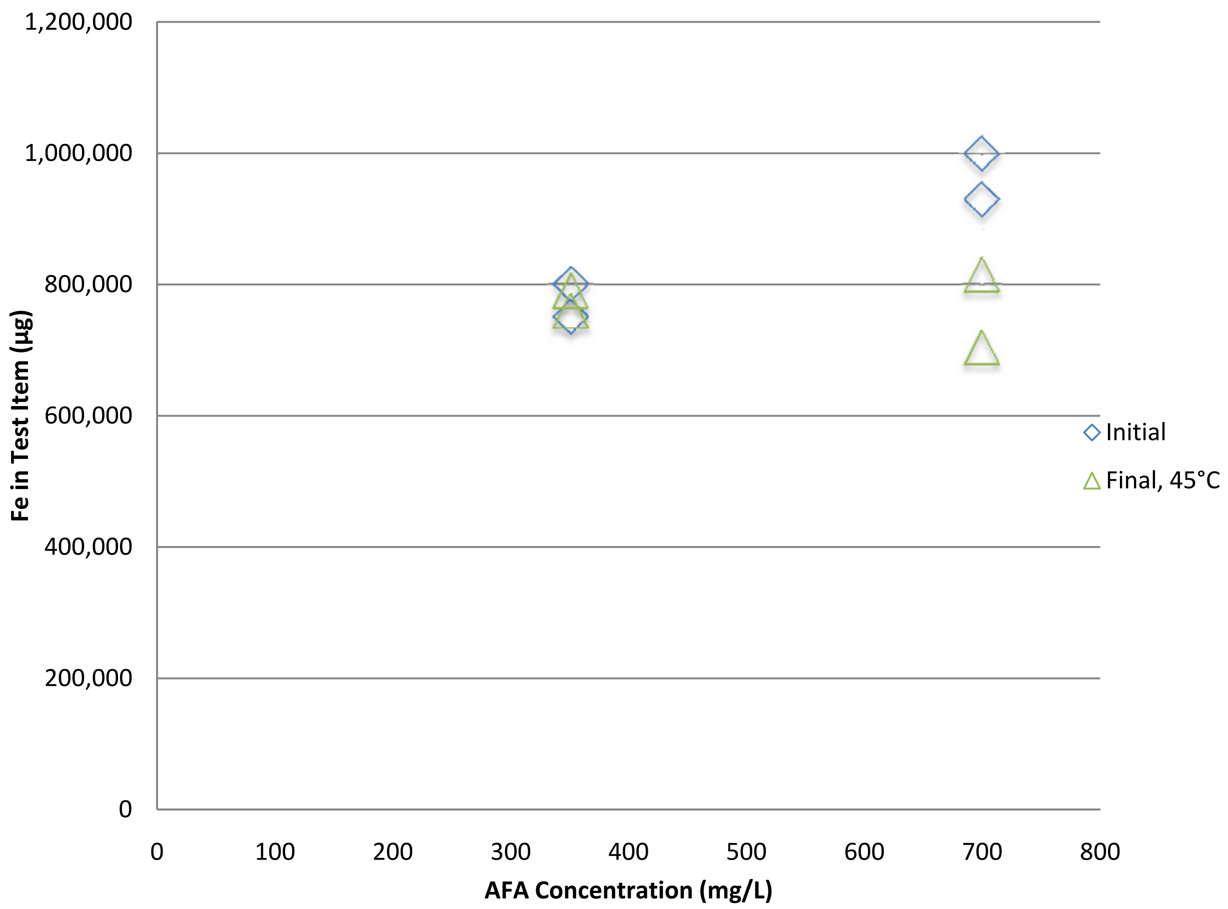

Figure 3.9. Quantity of Iron in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 45°C

3.3.3 Manganese Results

Manganese was not one of the oxides or hydroxides that was included in the simulant during preparation. Analysis of the initial solids reported some manganese in some of the solids. The relatively small amounts are considered either impurities or artifacts from the analytical method of the complex matrix. During the oxidative leaching process, Mn is added as liquid permanganate solution and is converted to an insoluble solid that precipitates as MnO_2 and becomes part of the solids. All of the solutions and solids were analyzed for manganese and the results are shown in Table 3.11. Manganese was not detected in the initial 3 M NaOH or the caustic leachate. There were small amounts detected in the initial solids. There is insufficient data to determine if these trace quantities in the solids are impurities in the starting materials or artifacts of the analytical method. Comparisons of the starting solids and the solids after oxidative-leach are listed in Table 3.12. Essentially all of the added permanganate is reduced and reports to the solid phase.

Table 3.11. Amounts of Manganese Present During Testing

Test	AFA (mg/L)	Mn (μg) Initial Leachate	Caustic-Leach Supernate + Washes	Solids After Caustic-Leach	Oxidative-Leach Supernate + Washes	Solids After Oxidative-Leach
1	175	<2.7	<4.9	1,100	<11	560,000
2	350	<2.6	<5.0	1,100	<11	500,000
3	350	<2.6	<4.9	930	<11	570,000
4a	700	<2.6	<5.4	2,000	<11	620,000
4b	700	<2.6	<4.6	1,400	<11	560,000
4c	700	<2.6	<4.9	900	<10	590,000
5	350	<2.6	<4.9	3,500	1,400	610,000
6	700	<2.6	<4.9	560	<12	600,000
7	350	<2.6	<4.4	380	<12	600,000
8a	0	<2.6	<5.0	240	<11	590,000
8b	0	<2.6	<4.5	70	<10	600,000
8c	0	<2.6	<4.7	<0.11	4,200	650,000
9	700	<2.6	<4.2	<0.067	<1.4	610,000
10	700	<2.6	<4.7	<0.061	2,700	590,000

Table 3.12. Manganese in Solids from Simulated Tank Sludge by Oxidative Leaching

Test	Initial Mn (μg/g)	Initial Total Mn (μg)	Final Mn (μg/g)	Final Total Mn (μg)	Fraction Mn Removed
1	240	1,100	180,000	560,000	NA[(b)]
2	170	1,100	170,000	500,000	NA
3	180	930	190,000	570,000	NA
4a	370	2,000	210,000	620,000	NA
4b	300	1,400	180,000	560,000	NA
4c	160	900	200,000	590,000	NA
5	640	3,500	200,000	610,000	NA
6	76	[560][(a)]	200,000	600,000	NA
7	62	[380]	200,000	600,000	NA
8a	36	[240]	200,000	590,000	NA
8b	11	[70]	200,000	600,000	NA
8c	8	<72	220,000	650,000	NA
9	10	<60	200,000	610,000	NA
10	7	<40	190,000	590,000	NA

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

(b) NA = Not Applicable

The concentration of manganese initially present in the simulated tank waste and the amount of manganese remaining after oxidative leaching is shown in Figure 3.10 for the 25°C tests and in Figure 3.11 for the 45°C tests. The manganese results separated by AFA concentration and temperature are summarized in Table 3.13 with the relative differences between multiples of the same sample types ranging between 1.2% and 9.3%. The 45°C tests both had duplicate samples that had relative differences of 1.2% between the two. These values are essentially identical.

Table 3.13. Summary of Mn Concentration by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Mn in Final Solids (µg)	Data Points	Average	Stdev	RSD
25°C	0	590,000				
25°C	0	600,000				
25°C	0	650,000	3@25,0	610,000	32,000	5.2%
25°C	175	560,000	1@25,175	560,000	NA	NA
25°C	350	500,000				
25°C	350	570,000	2@25, 350	540,000	49,000	9.3%
25°C	700	620,000				
25°C	700	560,000				
25°C	700	590,000				
25°C	700	590,000	4@25, 700	600,000	19,000	3.2%
45°C	350	610,000				
45°C	350	600,000	2@45, 350	600,000	7,100	1.2%
45°C	700	600,000				
45°C	700	610,000	2@45, 700	600,000	7,100	1.2%

NA = Not Applicable

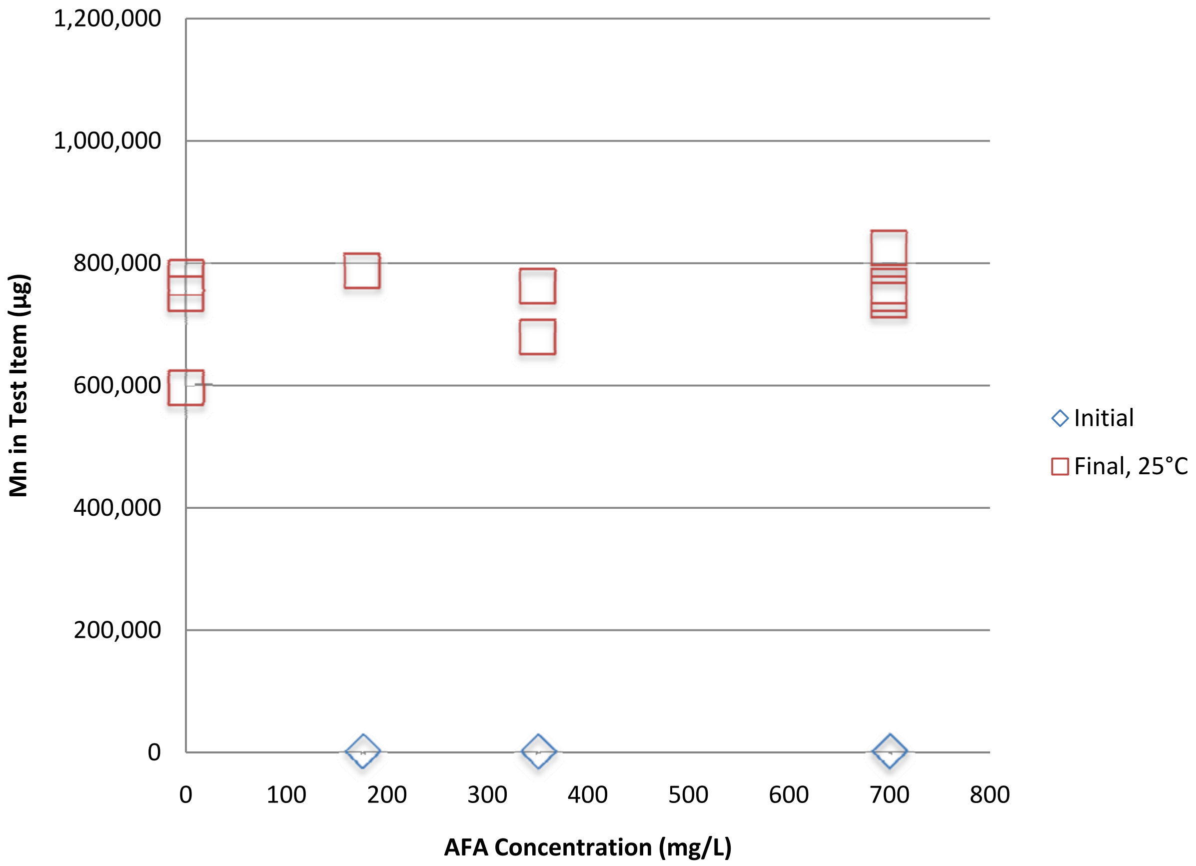

Figure 3.10. Quantity of Manganese in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 25°C

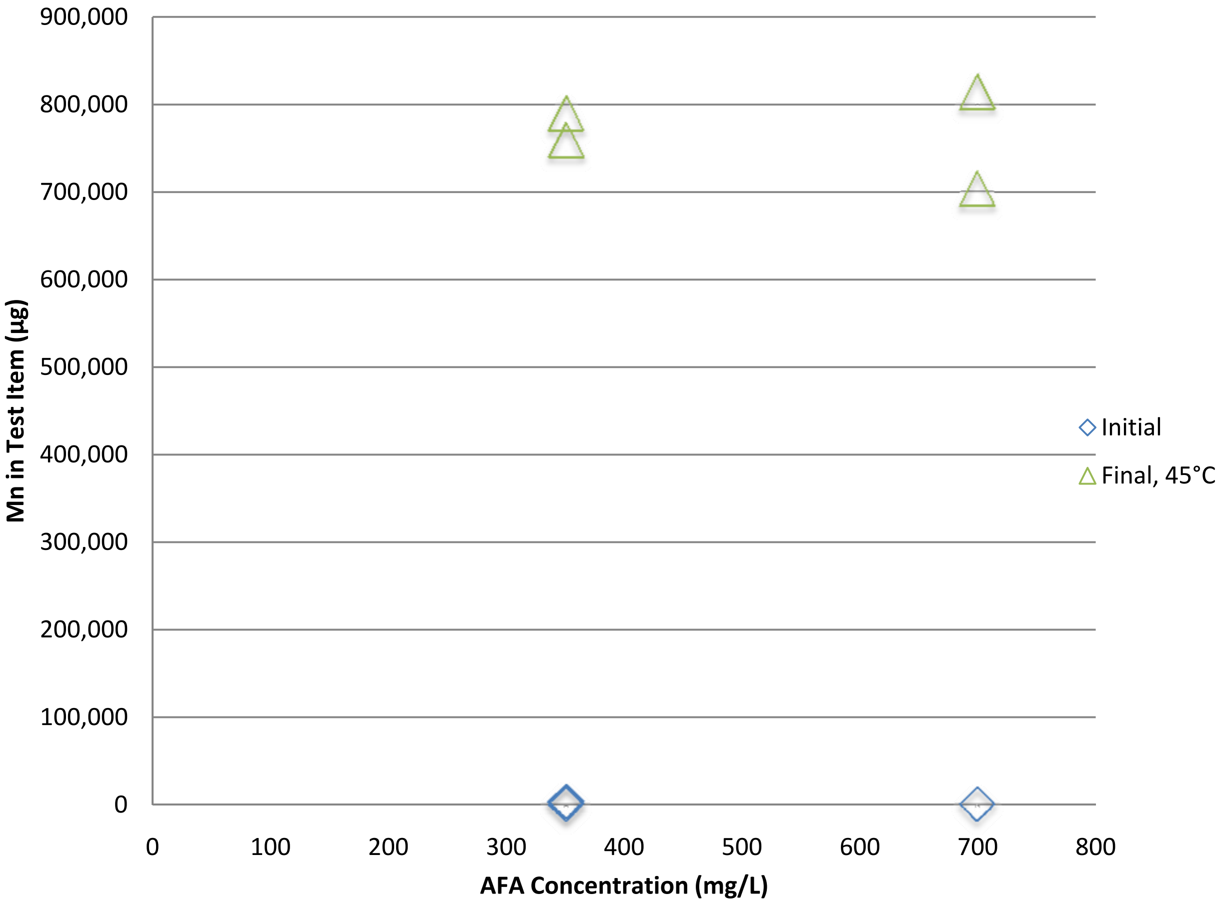

Figure 3.11. Quantity of Manganese in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 45°C

3.3.4 Nickel Results

Removal of nickel from simulated tank sludge by oxidative leaching resulted in no detectable nickel being removed. Only solutions were analyzed and all values measured for the solution nickel concentrations were below instrument detection limits. The fraction nickel removed calculation is based on the reported "less than" value from the oxidative-leach solution analysis. Results are shown in Table 3.14. Based on the amount of nickel not detected in the solution, the fraction of nickel removed is less than 0.03% of the total nickel present.

Table 3.14. Nickel Removal from Simulated Tank Sludge by Oxidative Leaching

Test	Initial Ni[(a)] (µg/g)	Initial Total Ni (µg)	Final Ni[(a)] (µg/g)	Final Total Ni (µg)	Fraction Ni Removed
1	6,600	30,000	9,900	30,000	<0.00027
2	4,400	30,000	9,900	30,000	<0.00028
3	5,700	30,000	9,900	30,000	<0.00028
4a	5,500	30,000	10,000	30,000	<0.00028
4b	6,300	30,000	9,800	30,000	<0.00029
4c	5,200	30,000	10,000	30,000	<0.00027
5	5,300	30,000	9,800	30,000	<0.00031
6	4,000	30,000	10,000	30,000	<0.00030
7	4,900	30,000	9,600	30,000	<0.00031
8a	4,600	30,000	9,900	30,000	<0.00027
8b	4,700	30,000	9,800	30,000	<0.00026
8c	3,100	30,000	9,800	30,000	<0.00028
9	4,900	30,000	9,700	30,000	<0.00030
10	5,300	30,000	9,700	30,000	<0.00027

(a) Ni crucibles were used in the analytical fusions procedure to dissolve solids samples for analysis. These values are calculated based on simulant makeup and solution analyses.

Because no nickel was detected in any of the leach solutions, there was no indication of a correlation between the concentration of AFA and removal of nickel by oxidative leaching of the simulated tank sludge. The values are plotted in Figure 3.12.

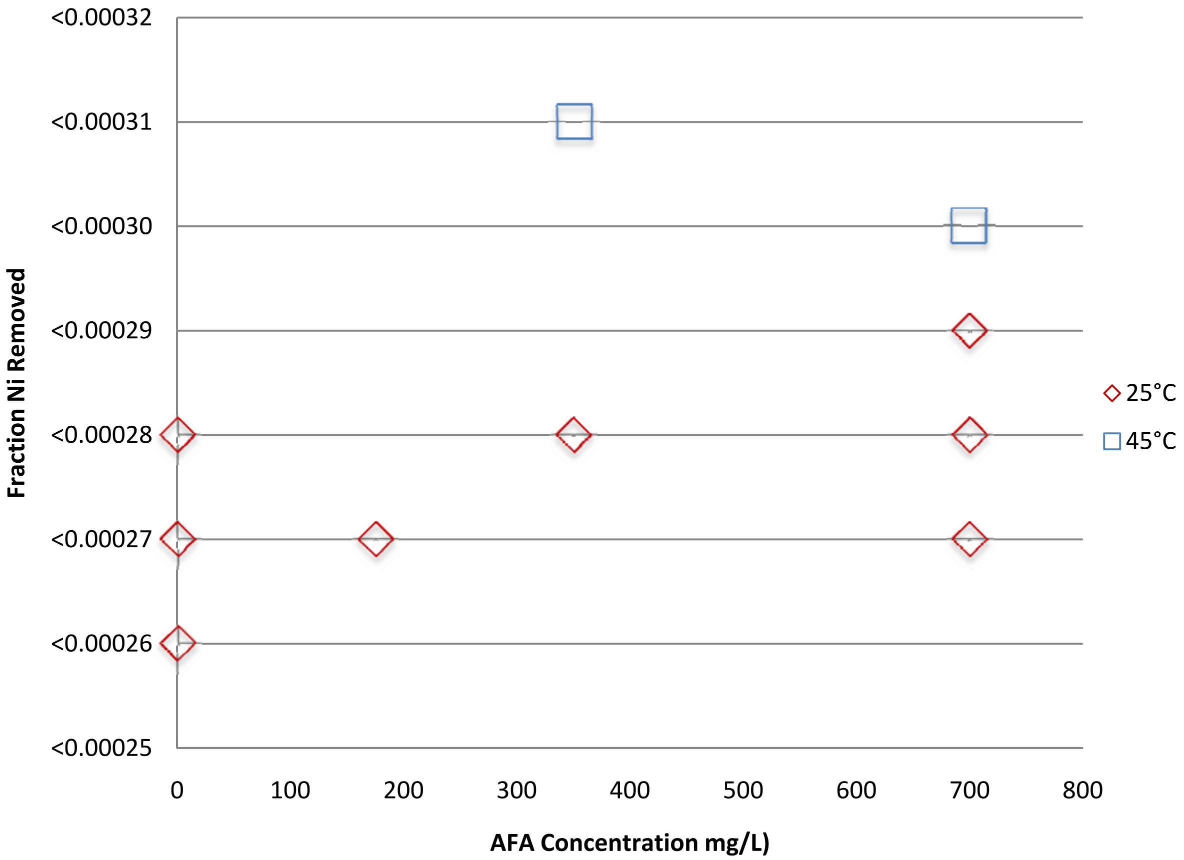

Figure 3.12. Fraction of Nickel Removed from Simulated Tank Waste by Permanganate Oxidative Leaching

3.3.5 Zinc Results

Removal of zinc from simulated tank sludge by oxidative leaching resulted in -34% to +59% of the initial zinc being removed as seen in Table 3.15. These values are based on analysis of washed and dried solids.

Table 3.15. Zinc Removal from Simulated Tank Sludge by Oxidative Leaching

Test	Initial Zn (µg/g)	Initial Total Zn (µg)	Final Zn (µg/g)	Final Total Zn (µg)	Fraction Zn Removed
1	1,400	6,400	1,900	5,700	0.11
2	1,100	7,200	1,600	4,800	0.34
3	1,400	7,500	1,800	5,600	0.26
4a	1,400	7,600	2,100	6,300	0.17
4b	1,500	7,200	1,700	5,200	0.28
4c	1,200	6,800	1,900	5,600	0.17
5	2,600	14,000	2,000	5,900	0.59
6	1,200	8,800	2,000	6,000	0.32
7	1,100	6,800	2,000	6,100	0.10
8a	960	6,300	2,000	5,900	0.06
8b	1,200	7,600	2,000	6,100	0.20
8c	700	6,700	1,700	5,000	0.25
9	1,200	7,600	2,200	6,900	0.10
10	980	5,400	2,400	7,300	-0.34

Evaluation of the zinc removal as a function of AFA concentration is summarized in Table 3.16 and the fraction zinc removed as a function of AFA concentration is shown in Figure 3.13. A least square fit of the data at 25°C yields $y = -0.0001x + 0.2006$ with $R^2 = 0.0534$ and at 45°C yields $y = -0.0004x + 0.4825$ with $R^2 = 0.1143$. The fraction of Zn removed values of 0.59 and -0.34 lie outside the 95% confidence interval and appear to be the result of unusually high and low initial values, respectively. Removal of these two values and fitting the remaining data gives a least square fit of the data at 25°C of $y = 0.0003x - 0.0088$ with $R^2 = 0.2438$ and at 45°C yields $y = 7E\text{-}05x + 0.179$ with $R^2 = 0.0712$. These data show no correlation between the concentration of AFA and removal of zinc by oxidative leaching of the simulated tank sludge.

The concentration of zinc initially present in the simulated tank waste and the amount of zinc remaining after oxidative leaching is shown in Figure 3.14 for the 25°C tests and in Figure 3.15 for the 45°C tests.

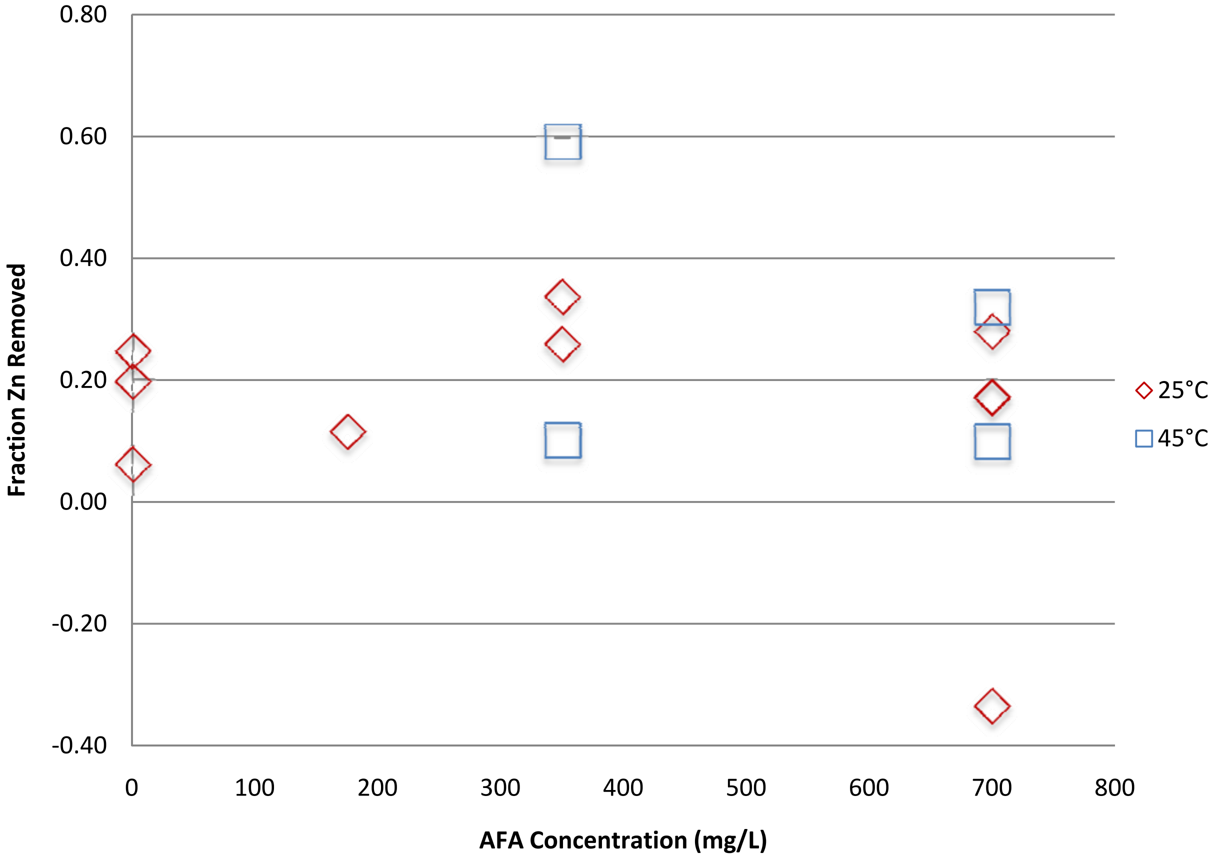

Figure 3.13. Fraction of Zn Removed by Oxidative Leaching at Either 25°C or 45°C

Table 3.16. Summary of Zn Removal by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Fraction Zn Removed	Data Points	Average	Stdev	RSD
25°C	0	0.06				
25°C	0	0.20				
25°C	0	0.25	3@25,0	0.17	0.10	57%
25°C	175	0.11	1@25,175	0.11	NA	NA
25°C	350	0.34				
25°C	350	0.26	2@25, 350	0.30	0.05	18.5%
25°C	700	0.17				
25°C	700	0.28				
25°C	700	0.17				
25°C	700	-0.34	4@25, 700	0.18	0.28	152%
45°C	350	0.59				
45°C	350	0.10	2@45, 350	0.35	0.35	101%
45°C	700	0.32				
45°C	700	0.10	2@45, 700	0.21	0.16	75%

NA = Not Applicable

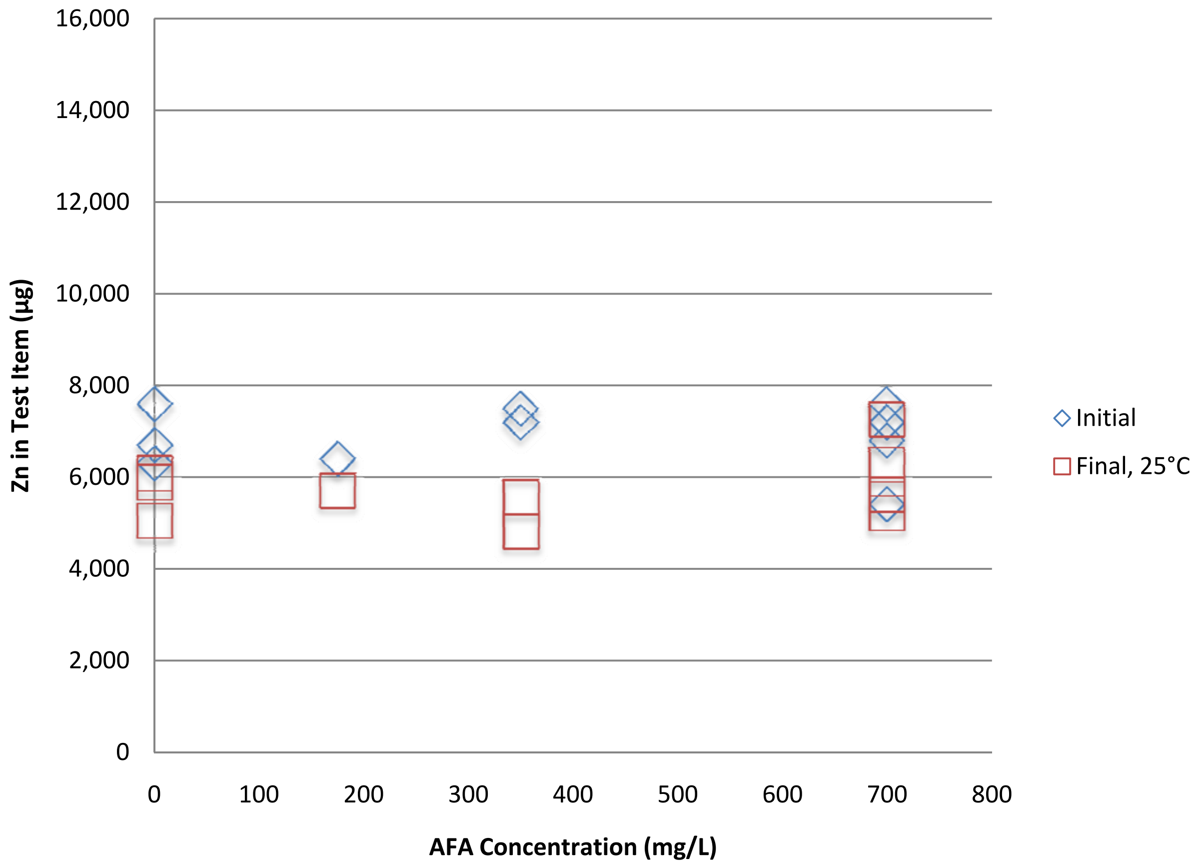

Figure 3.14. Quantity of Zinc in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 25°C

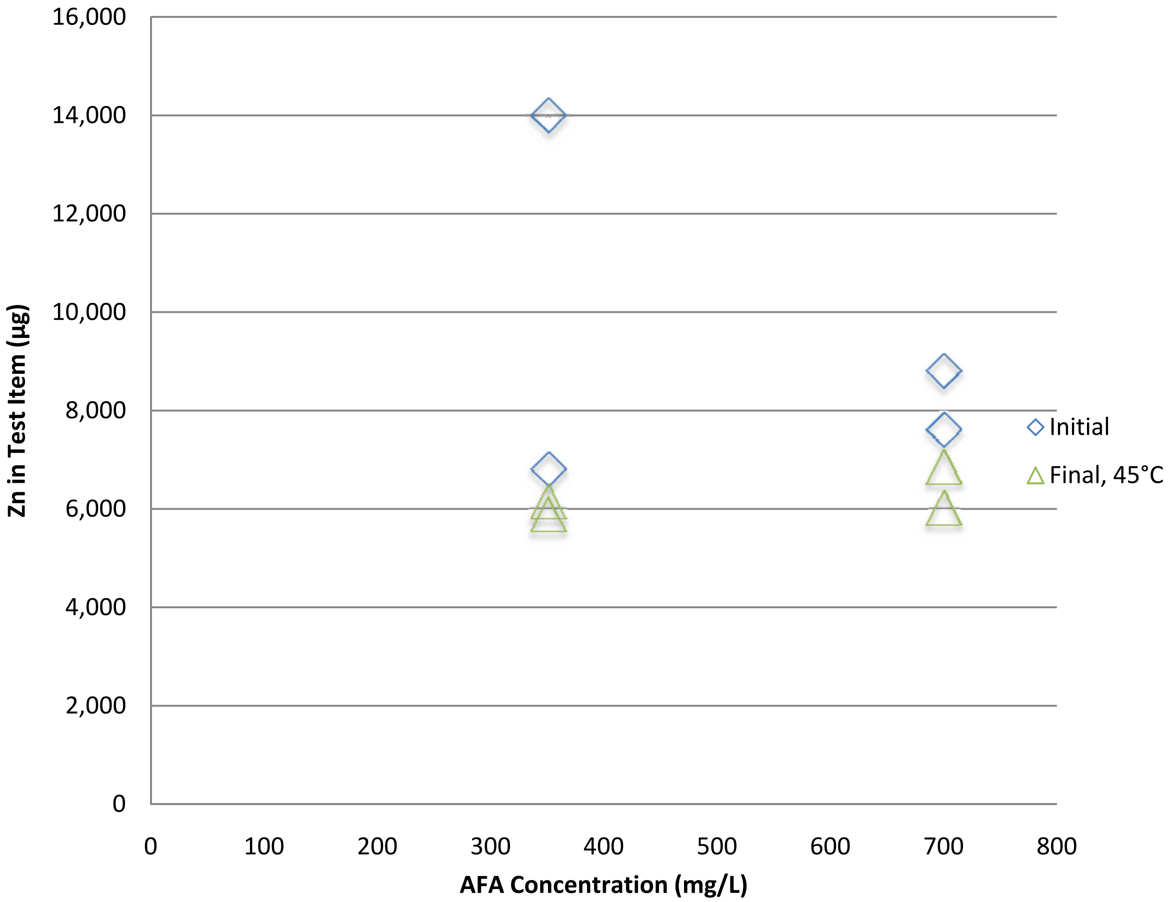

Figure 3.15. Quantity of Zinc in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 45°C

3.3.6 Uranium Results

Removal of uranium from simulated tank sludge by oxidative leaching resulted in 0.40% to 3.9% of initial uranium being removed as seen in Table 3.17. These values are based on analysis of leachate solution compared to washed and dried residual solids.

Table 3.17. Uranium Removal from Simulated Tank Sludge by Oxidative Leaching

Test	Initial U (µg/g)	Initial Total U (µg)	Final U (µg/g)	Final Total U (µg)	Fraction U Removed
1	[2,800]	[12,880]	15,000	46,000	0.010
2	[2,000]	[13,409]	15,000	44,000	0.008
3	[1,200]	[6,334]	14,000	43,000	0.039
4a	[2,500]	[13,488]	20,000	59,000	0.012
4b	[2,900]	[13,770]	14,000	43,000	0.015
4c	[1,700]	[9,920]	32,000	97,000	0.014
5	[2,800]	[15,490]	14,000	42,000	0.023
6	[3,500]	[26,002]	33,000	99,000	0.010
7	[1,900]	[11,575]	23,000	71,000	0.026
8a	[2,800]	[18,302]	24,000	72,000	0.015
8b	[3,400]	[21,616]	23,000	71,000	0.007
8c	[1,900]	[18,227]	37,000	110,000	0.004
9	[4,700]	[28,791]	14,000	42,000	0.013
10	[3,000]	[16,602]	28,000	87,000	0.006

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Evaluation of the uranium removal as a function of AFA concentration is summarized in Table 3.18 and the fraction uranium removed as a function of AFA concentration is shown in Figure 3.16. A least square fit of the data at 25°C yields $y = 1E\text{-}06x + 0.0168$ with $R^2 = 0.001$ and at 45°C yields $y = 1E\text{-}05x + 0.0016$ with $R^2 = 0.3474$. These data yield lead to conflicting conclusions. At 25°C, the data show no correlation between the concentration of AFA and removal of uranium by oxidative leaching of the simulated tank sludge, while at 45°C, there appears to be a slight correlation, with more uranium removed at higher AFA concentration.

The concentration of uranium initially present in the simulated tank waste and the amount of uranium remaining after oxidative leaching is shown in Figure 3.17 for the 25°C tests and in Figure 3.18 for the 45°C tests.

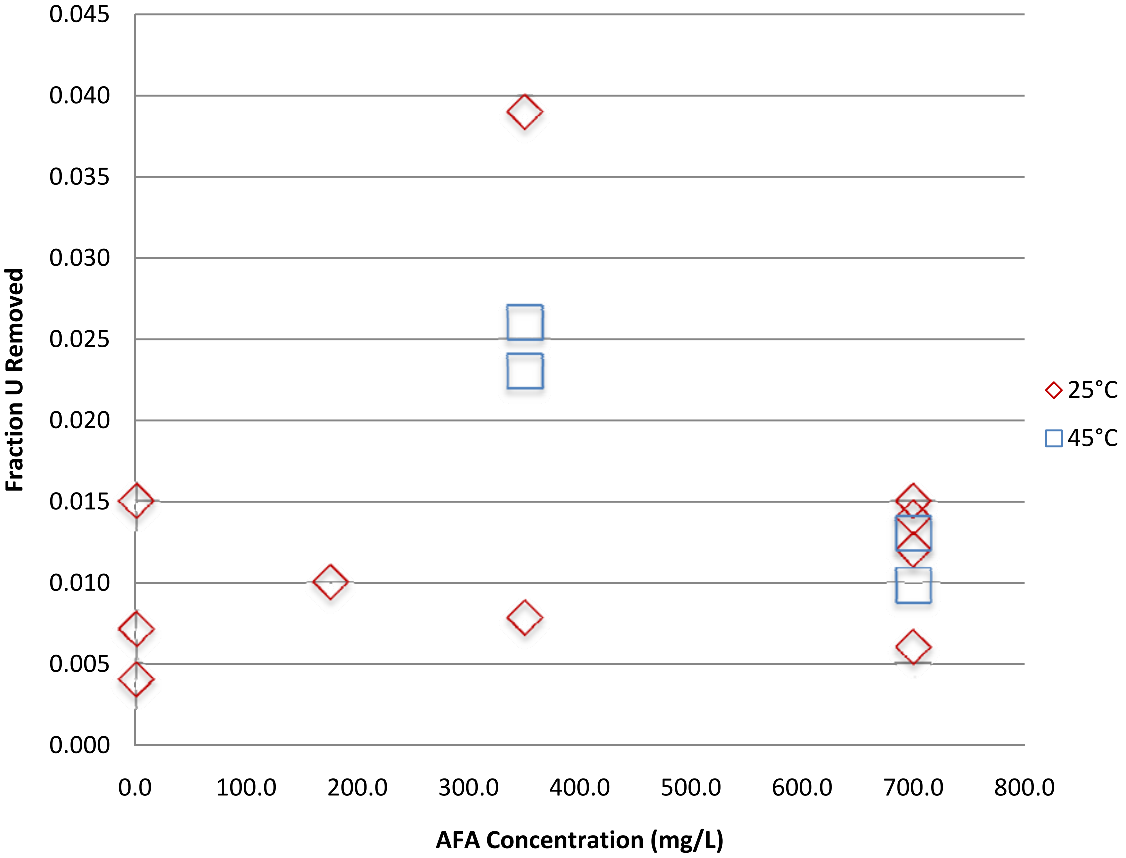

Figure 3.16. Fraction of U Removed by Oxidative Leaching at Either 25°C or 45°C

Table 3.18. Summary of U Removal by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Fraction U Removed	Data Points	Average	Stdev	RSD
25°C	0	0.015				
25°C	0	0.007				
25°C	0	0.0040	3@25, 0	0.0087	0.0057	65%
25°C	175	0.010	1@25,175	0.010	NA	NA
25°C	350	0.0078				
25°C	350	0.039	2@25, 350	0.023	0.022	94.3%
25°C	700	0.012				
25°C	700	0.015				
25°C	700	0.014				
25°C	700	0.0060	4@25, 700	0.015	0.0071	47%
45°C	350	0.023				
45°C	350	0.026	2@45, 350	0.025	0.0021	9%
45°C	700	0.010				
45°C	700	0.013	2@45, 700	0.011	0.0023	20%

NA = Not Applicable

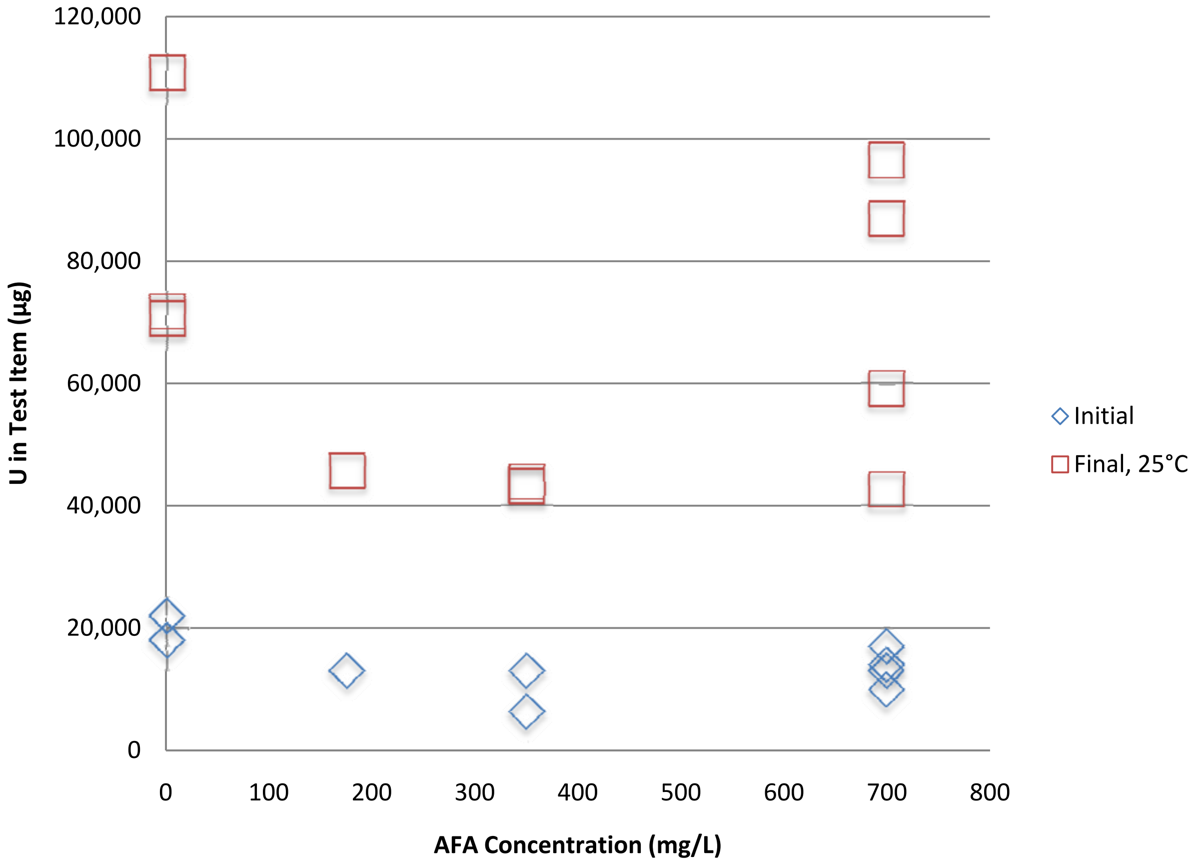

Figure 3.17. Quantity of Uranium in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 25°C

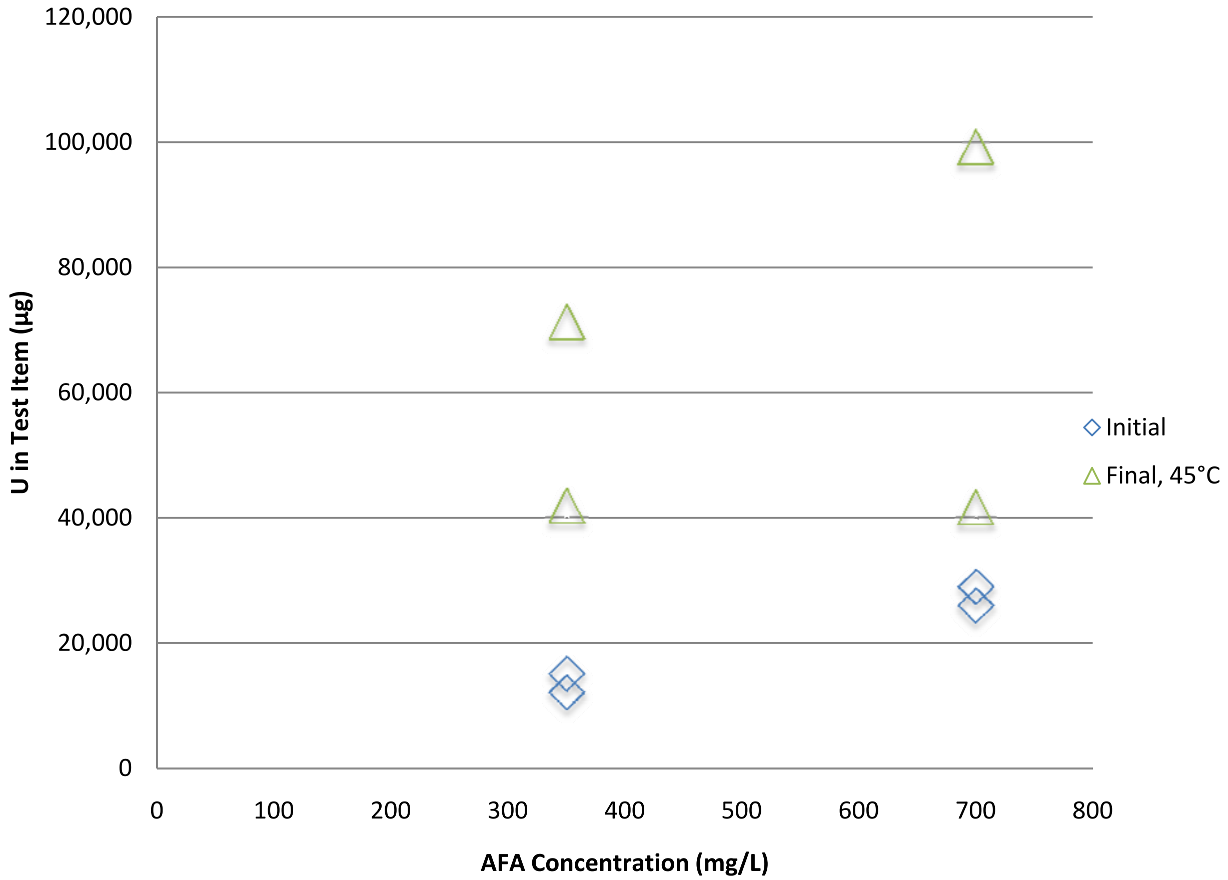

Figure 3.18. Quantity of Uranium in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 45°C

3.3.7 $^{239+240}$Pu Results

Removal of $^{239+240}$Pu from simulated tank sludge by oxidative leaching resulted in 0.00040% to 0.24% of initial plutonium being removed as seen in Table 3.19. These values are based on analysis of leachate solution compared to washed and dried residual solids.

Table 3.19. $^{239+240}$Pu Removal from Simulated Tank Sludge by Oxidative Leaching

Test	Initial $^{239+240}$Pu (µCi/g)	Initial Total $^{239+240}$Pu (µCi)	Final $^{239+240}$Pu (µCi/g)	Final Total $^{239+240}$Pu (µCi)	Fraction $^{239+240}$Pu Removed
1	14	63	20	60	2.7E-04
2	10	68	21	62	1.8E-04
3	12	63	21	65	1.2E-04
4a	14	77	21	63	1.0E-04
4b	15	69	23	70	4.5E-05
4c	12	68	24	73	1.8E-05
5	13	70	25	75	2.4E-03
6	12	92	24	73	2.9E-05
7	11	68	25	77	1.5E-03
8a	11	73	23	70	8.7E-06
8b	11	69	23	72	4.0E-06
8c	7	67	22	68	3.4E-04
9	13	79	24	74	4.9E-06
10	12	69	24	73	3.1E-04

Evaluation of the $^{239+240}$Pu removal as a function of AFA concentration is summarized in Table 3.20 and the fraction $^{239+240}$Pu removed as a function of AFA concentration is shown in Figure 3.19. A least square fit of the data at 25°C yields $y = 9E\text{-}07x + 2E\text{-}05$ with $R^2 = 0.2277$ and at 45°C yields $y = 7E\text{-}09x + 0.0001$ with $R^2 = 0.0001$. These data show no correlation between the concentration of AFA and removal of $^{239+240}$Pu by oxidative leaching of the simulated tank sludge.

The concentration of $^{239+240}$Pu initially present in the simulated tank waste solids and the amount of $^{239+240}$Pu remaining after oxidative leaching is shown in Figure 3.20 for the 25°C tests and in Figure 3.21 for the 45°C tests.

There is a lot of scatter in these data that is likely due to very small particles of plutonium oxyhydroxide that are not removed by filtration. An evaluation of the solids was considered as an alternative method for quantifying the amount of $^{239+240}$Pu removed by oxidative leaching. The $^{239+240}$Pu in each solid test item initially ranged from 63 to 92 µCi per item with an average of 71 µCi and an RSD of 11%. The final solids contained from 60 to 75 µCi per test item with an average of 70 and an RSD of 8%. When the initial solids were compared to the final solids to determine the fraction of $^{239+240}$Pu removed by oxidative leaching, the values ranged from -0.13 to +0.21 with an average of 0.013 and an RSD of 764%. The large relative deviations appear to be due to comparing small differences between relatively large numbers. Within a group of analyses of solids, the deviations within the group are relatively small (8% and 11%). However, when comparing initial and final values for individual test items, the small differences produce very large relative differences and unreasonable values with approximately half of the samples having more $^{239+240}$Pu in the final samples than was present in the initial test items.

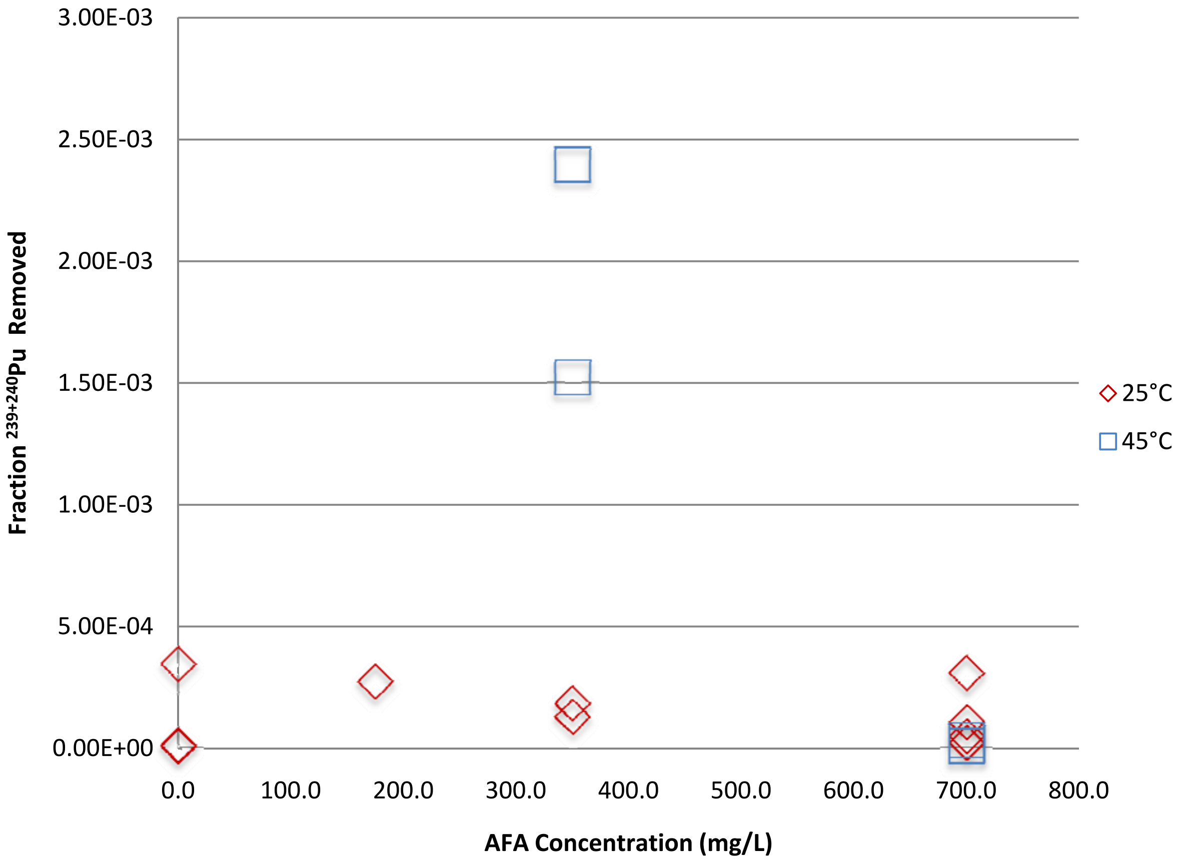

Figure 3.19. Fraction of $^{239+240}$Pu Removed by Oxidative Leaching at Either 25°C or 45°C

Table 3.20. Summary of $^{239+240}Pu$ Removal by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Fraction $^{239+240}Pu$ Removed	Data Points	Average	Stdev	RSD
25°C	0	8.66E-06				
25°C	0	4.00E-06				
25°C	0	3.41E-04	3@25,0	1.2E-04	1.9E-04	164%
25°C	175	2.70E-04	1@25,175	2.7E-04	NA	NA
25°C	350	1.80E-04				
25°C	350	1.25E-04	2@25, 350	1.5E-04	3.9E-05	25.6%
25°C	700	1.02E-04				
25°C	700	4.52E-05				
25°C	700	1.76E-05				
25°C	700	3.05E-04	4@25, 700	6.3E-04	9.5E-04	150%
45°C	350	2.40E-03				
45°C	350	1.52E-03	2@45, 350	2.0E-03	6.2E-04	32%
45°C	700	2.89E-05				
45°C	700	4.89E-06	2@45, 700	1.7E-05	1.7E-05	100%

NA = Not Applicable

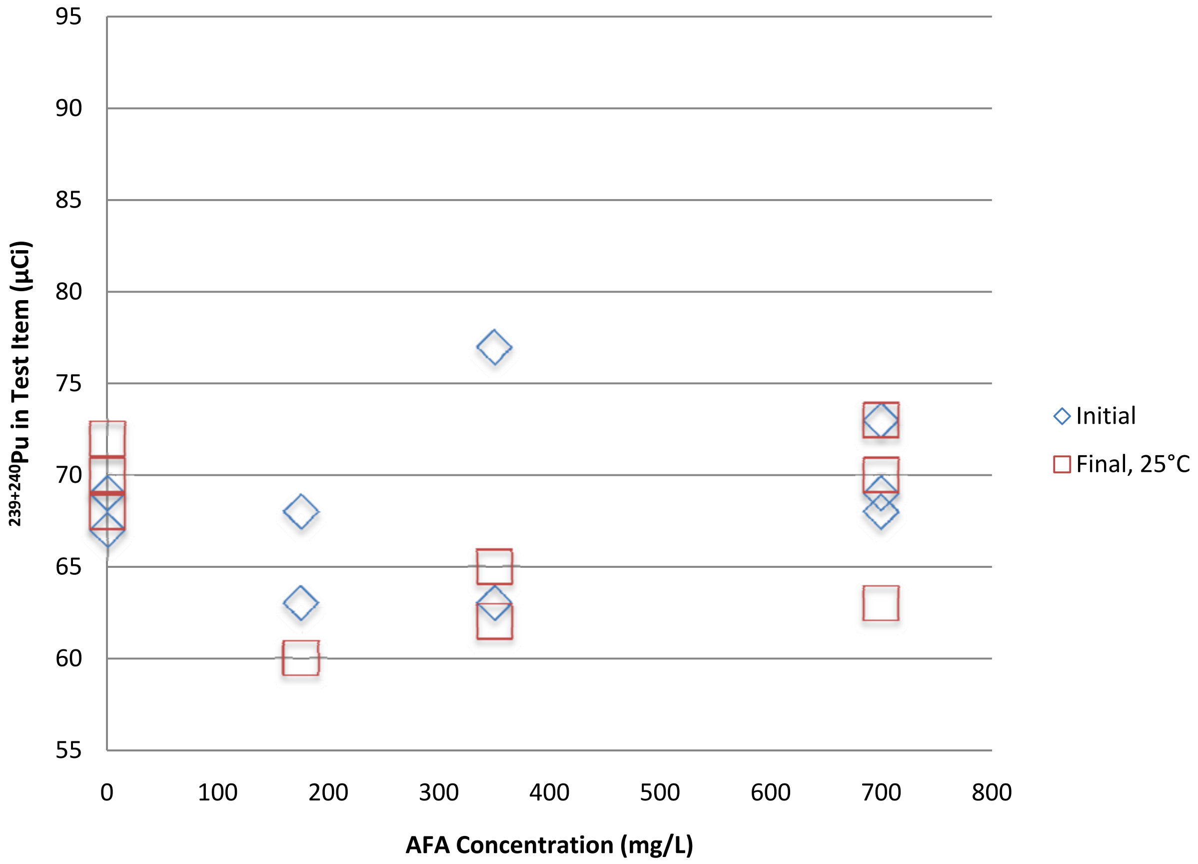

Figure 3.20. Quantity of ${}^{239+240}$Pu in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 25°C

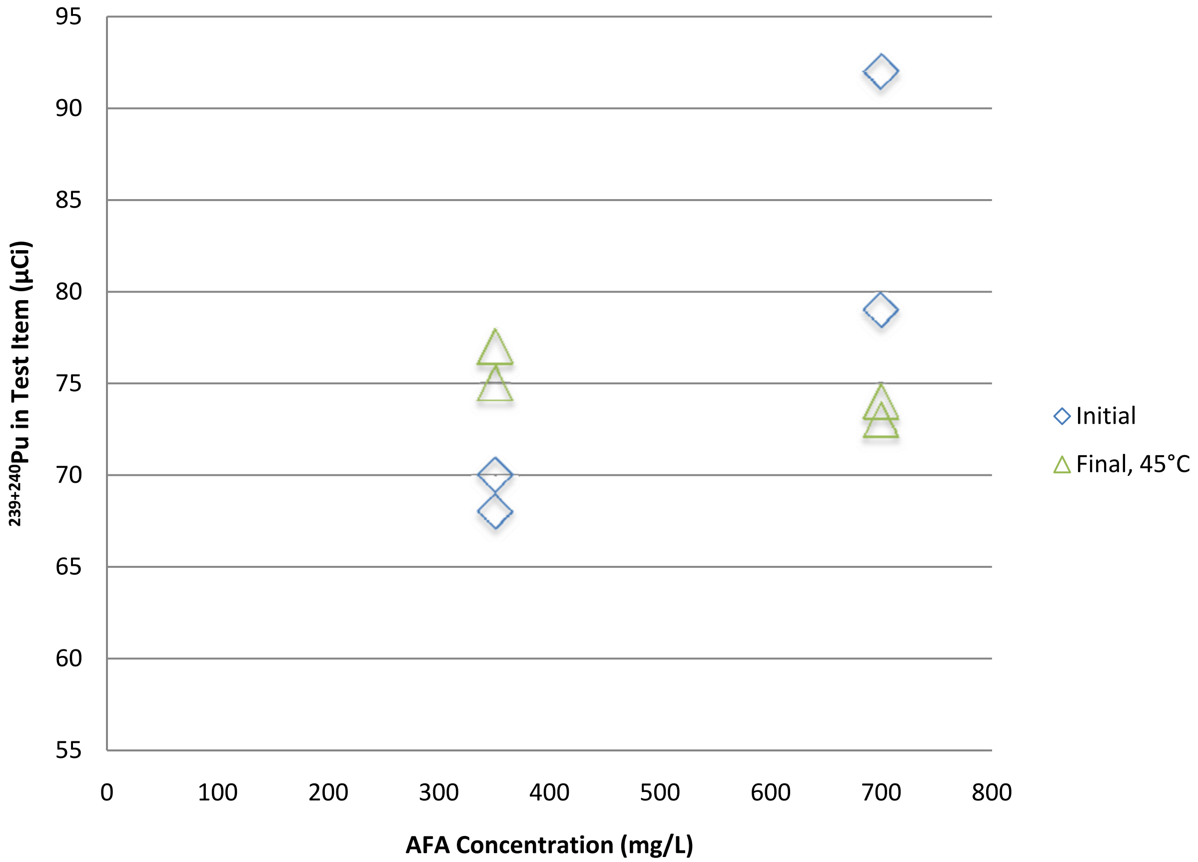

Figure 3.21. Quantity of $^{239+240}$Pu in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 45°C

3.3.8 ^{238}Pu+^{241}Am Results

Removal of ^{238}Pu+^{241}Am from simulated tank sludge by oxidative leaching resulted in 0.0032% to 0.026% of initial plutonium being removed as seen in Table 3.21. These values are based on analysis of leachate solution compared to washed and dried residual solids.

Table 3.21. ^{238}Pu+^{241}Am Removal from Simulated Tank Sludge by Oxidative Leaching

Test	Initial ^{238}Pu+^{241}Am (µCi/g)	Initial Total ^{238}Pu+^{241}Am (µg)	Final ^{238}Pu+^{241}Am (µg/g)	Final Total ^{238}Pu+^{241}Am (µg)	Fraction ^{238}Pu+^{241}Am Removed
1	0.28	1.3	0.59	1.8	1.96E-04
2	0.29	1.9	0.55	1.6	1.37E-04
3	0.25	1.3	0.58	1.8	9.46E-05
4a	0.36	1.9	0.59	1.8	<8.E-05
4b	0.36	1.7	0.61	1.9	<9.E-05
4c	0.31	1.8	0.64	1.9	<3.E-05
5	0.31	1.7	0.64	1.9	1.80E-03
6	0.33	2.5	0.56	1.7	<3.E-05
7	0.27	1.7	0.63	1.9	1.08E-03
8a	0.36	2.3	0.58	1.7	<3.E-05
8b	0.27	1.7	0.58	1.8	<3.E-05
8c	0.19	1.8	0.60	1.8	2.25E-04
9	0.31	1.9	0.63	1.9	<1.E-05
10	0.35	2.0	0.58	1.8	2.43E-04

Evaluation of the ^{238}Pu+^{241}Am removal as a function of AFA concentration is summarized in Table 3.22 and the fraction ^{238}Pu+^{241}Am removed as a function of AFA concentration is shown in Figure 3.22. A least square fit of the data at 25°C yields $y = 9E\text{-}07x + 2E\text{-}05$ with $R^2 = 0.2277$ and at 45°C yields $y = 7E\text{-}09x + 0.0001$ with $R^2 = 0.0001$. These data show no correlation between the concentration of AFA and removal of ^{238}Pu+^{241}Am by oxidative leaching of the simulated tank sludge.

The concentration of ^{238}Pu+^{241}Am initially present in the simulated tank waste solids and the amount of ^{238}Pu+^{241}Am remaining after oxidative leaching is shown in Figure 3.23 for the 25°C tests and in Figure 3.24 for the 45°C tests.

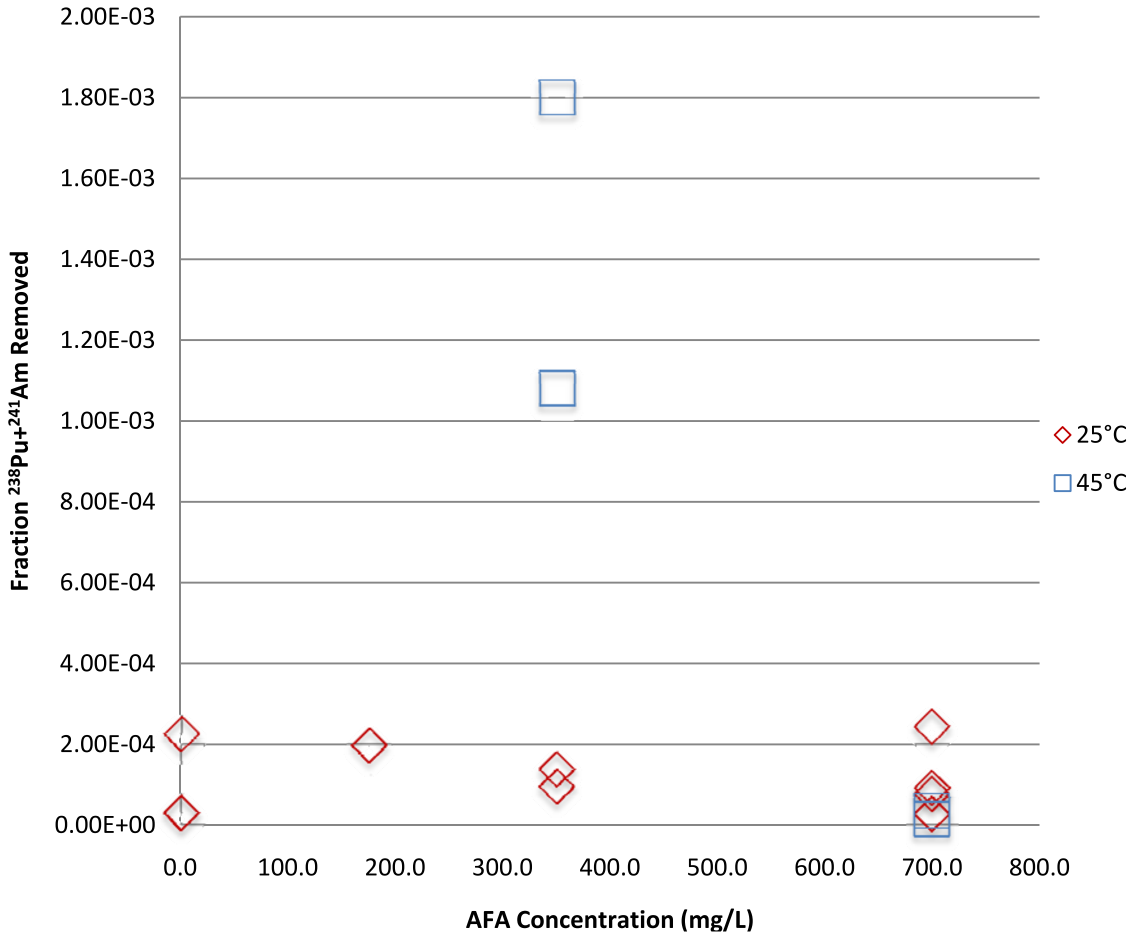

Figure 3.22. Fraction of ^{238}Pu+^{241}Am Removed by Oxidative Leaching at Either 25°C or 45°C

Table 3.22. Summary of ^{238}Pu+^{241}Am Removal by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Fraction ^{238}Pu+^{241}Am Removed	Data Points	Average	Stdev	RSD
25°C	0	<3.0.E-05				
25°C	0	<2.8.E-05				
25°C	0	2.25E-04	3@25,0	9.4E-05	1.1E-04	120%
25°C	175	1.96E-04	1@25,175	2.0E-04	NA	NA
25°C	350	1.37E-04				
25°C	350	9.46E-05	2@25, 350	1.2E-04	3.0E-05	26.0%
25°C	700	<7.6.E-05				
25°C	700	<9.1.E-05				
25°C	700	<2.7.E-05				
25°C	700	2.43E-04	4@25, 700	4.8E-04	6.9E-04	145%
45°C	350	1.80E-03				
45°C	350	1.08E-03	2@45, 350	1.4E-03	5.1E-04	35%
45°C	700	<3.5.E-05				
45°C	700	<1.5.E-05	2@45, 700	2.5E-05	1.4E-05	57%

NA = Not Applicable

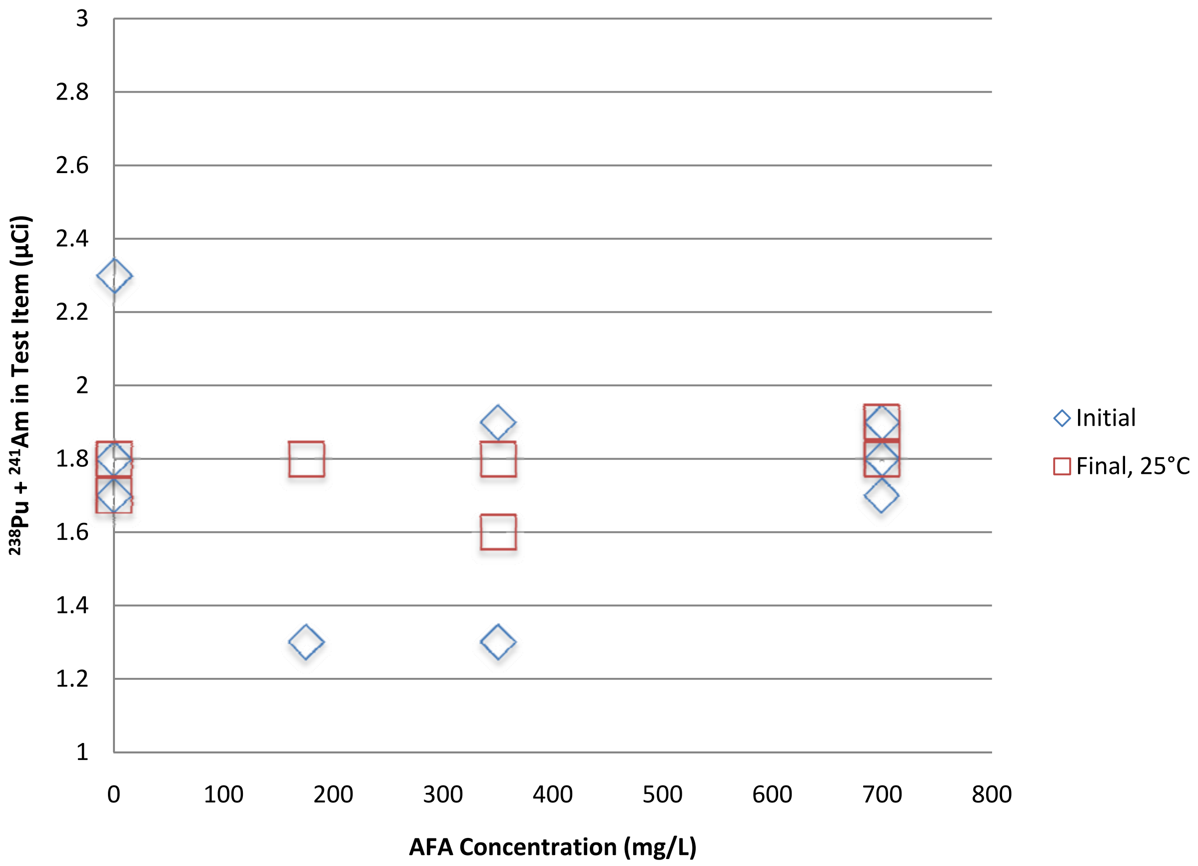

Figure 3.23. Quantity of ^{238}Pu + ^{241}Am in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 25°C

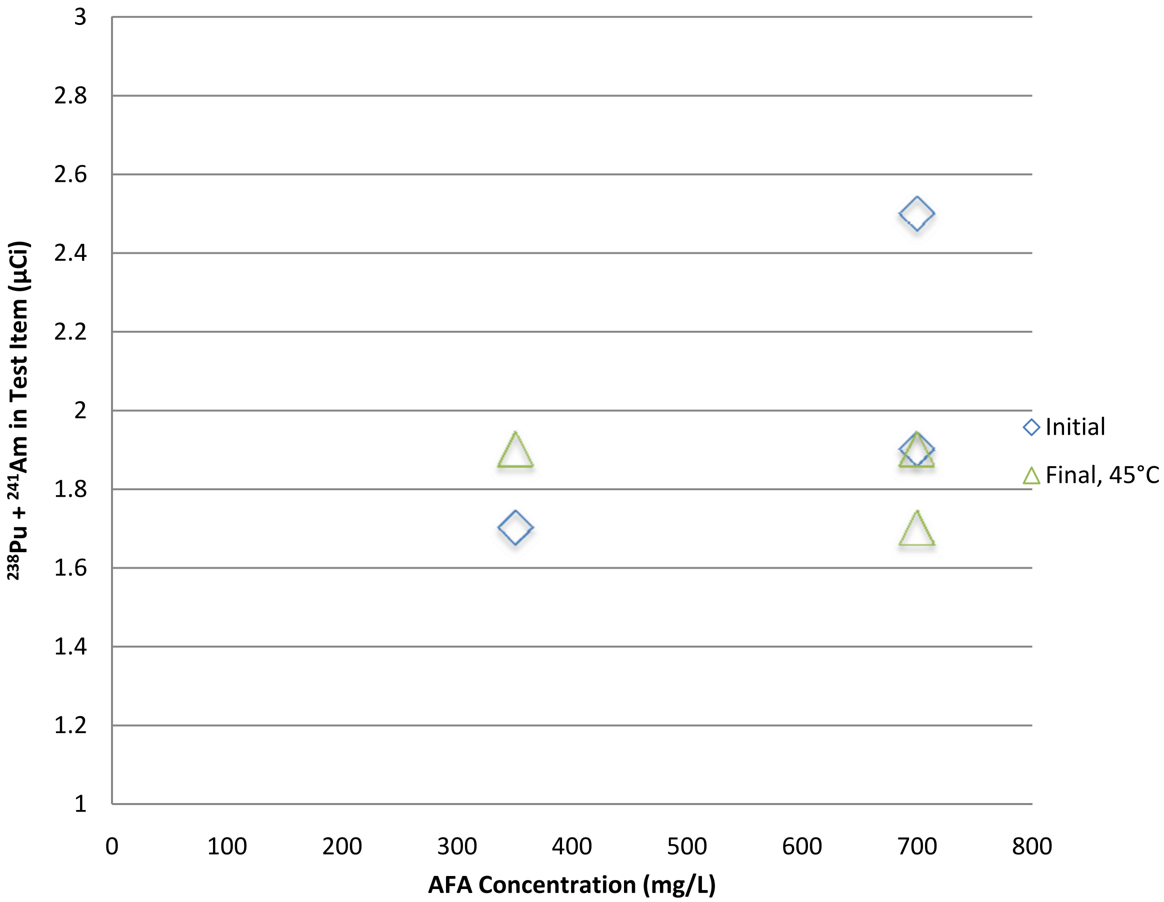

Figure 3.24. Quantity of ^{238}Pu + ^{241}Am in the Solids Before Oxidative Leaching (Initial) and After Oxidative Leaching (Final) at 45°C

Although plutonium and americium were not analyzed separately, by comparing the results for $^{239+240}$Pu with the results for ^{238}Pu + ^{241}Am any differences in behavior of americium compared to plutonium may be inferred. In Figure 3.25, The fraction of $^{239+240}$Pu removed plotted against the fraction of ^{238}Pu + ^{241}Am removed yields a straight line with a slope of 0.7. If the plutonium and americium were removed equally, one would expect a slope of 1.0, therefore, these data indicate plutonium is preferentially removed and more of the americium remains with the solids.

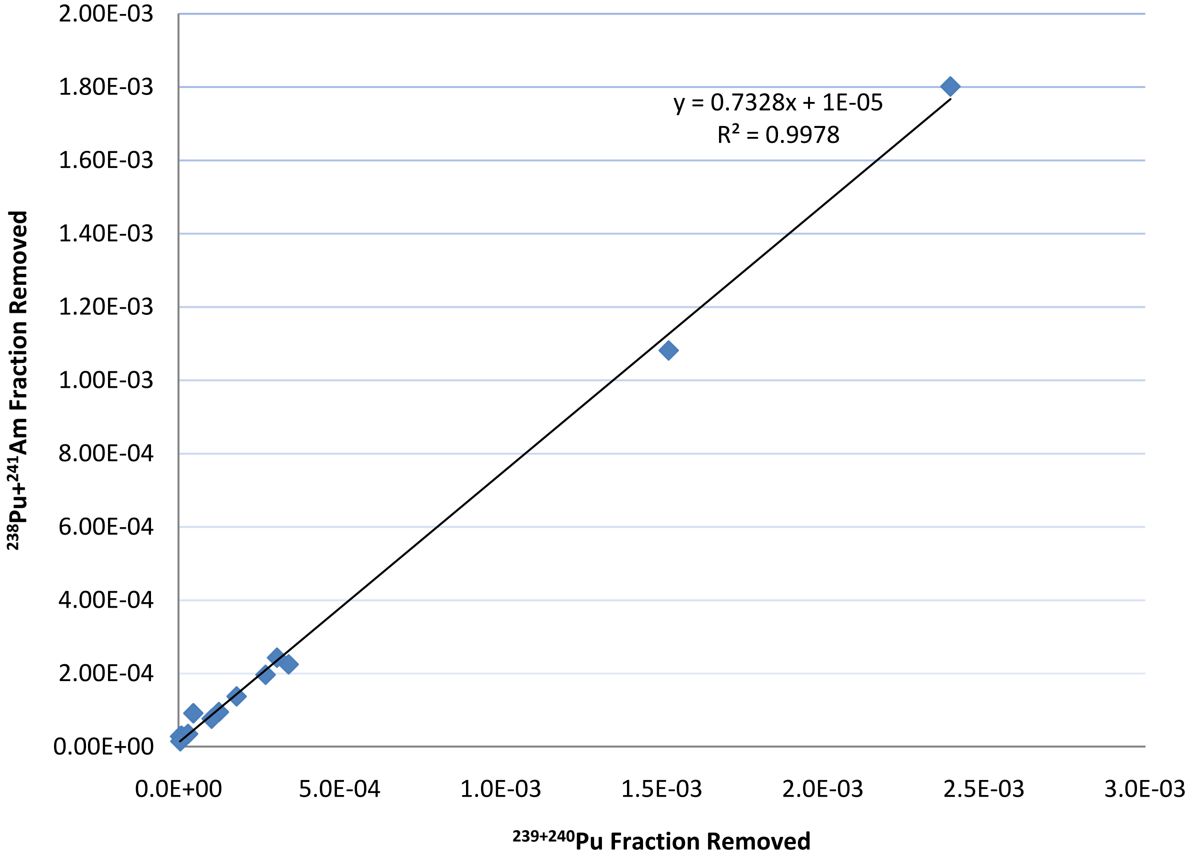

Figure 3.25. Relationship Between $^{239+240}$Pu and ^{238}Pu + ^{241}Am Removal by Oxidative Leaching

3.3.9 Combined Results Comparisons

Comparisons of analytical results for analytical laboratory duplicate samples of the final oxidatively leached solids in Table 3.23 demonstrate that interpreting the results must take into consideration the range of values that may be reported for seemingly homogeneous samples. These duplicates were samples of the final solids after oxidative-leach that were divided in the analytical laboratory and prepared separately. The most extreme example of differences in results for these samples is seen in the uranium results where duplicate samples from Test 5 differ by 44% while there is 0% difference between the Test 10 duplicates.

Table 3.23. Comparison of Analytical Results for Key Components on Duplicate Samples of Final Dried Solids After Oxidative Leaching

Sample	(Analyte)[(a)]	Cr	Fe	Mn	U	Zn
AFA-Test-5-OL-S	(µg/g)	6,300	262,000	203,000	13,900	1,950
	(µg/g)	7,250	255,000	190,000	21,700	2,020
	RPD	14.0%	2.7%	6.6%	43.8%	3.5%
AFA-Test-10-OL-S	(µg/g)	6,740	245,000	192,000	28,400	2,370
	(µg/g)	6,160	247,000	202,000	28,400	2,370
	RPD	9.0%	0.8%	5.1%	0.0%	0.0%

(a) Ni was not reported because Ni crucibles were used in the analytical fusions procedure to dissolve solid samples for analysis.

Analysis results of the washed and dried residual solids following oxidative leaching are shown in Table 3.24.

Table 3.24. Amounts of Key Components in Final Washed and Dried Residual Solids Following Oxidative Leaching with Permanganate

Test	AFA added (mg/L)	Cr (µg)	Fe (µg)	Mn (µg)	Ni[(a)] (µg)	U (µg)	$^{239+240}$Pu (µCi)	^{238}Pu + ^{241}Am (µCi)
1	175	90,000	790,000	560,000	30,000	46,000	60	1.8
2	350	110,000	680,000	500,000	30,000	44,000	62	1.6
3	350	65,000	760,000	570,000	30,000	43,000	65	1.8
4a	700	68,000	830,000	620,000	30,000	59,000	63	1.8
4b	700	81,000	740,000	560,000	30,000	43,000	70	1.9
4c	700	26,000	760,000	590,000	30,000	97,000	73	1.9
5	350	19,000	790,000	610,000	30,000	42,000	75	1.9
6	700	24,000	700,000	600,000	30,000	99,000	73	1.7
7	350	27,000	760,000	600,000	30,000	71,000	77	1.9
8a	0	24,000	750,000	590,000	30,000	72,000	70	1.7
8b	0	27,000	780,000	600,000	30,000	71,000	72	1.8
8c	0	22,000	590,000	650,000	30,000	110,000	68	1.8
9	700	31,000	810,000	610,000	30,000	42,000	74	1.9
10	700	21,000	750,000	590,000	30,000	87,000	73	1.8

(a) Ni crucibles were used in the analytical fusions procedure to dissolve solids samples for analysis. These values are calculated based on simulant makeup and solution analyses.

Two triplicate samples (total six test items) were tested to determine variability in the method. These triplicates were test items 4a-c that contained AFA at the 700 ppm level and items 8a-c that contained no AFA. Results, averages, standard deviations, and relative standard deviations for each of the elements of interest in the test item solids leached with 3 M NaOH and then permanganate are shown in Table 3.25.

Table 3.25. Statistical Analysis of Triplicate Test Items at Two AFA Concentration Levels

Quantities Measured in Final Supernate

AFA Level (mg/L)		Cr (µg)	Fe (µg)	Mn (µg)	U (µg)	Zn (µg)	$^{239+240}$Pu (µCi)	^{238}Pu + ^{241}Am (µCi)
0	Average	580,000	33	1,400	680	<17	8.0E-03	1.7E-04
	Stdev	24,000	4	2,400	350		1.3E-02	2.1E-04
	RSD	4.2%	11.2%	170%	52%		163.6%	120.9%
700	Average	560,000	40	<1.0	930	<18	3.6E-03	1.2E-04
	Stdev	22,000	14		410		2.6E-03	6.1E-05
	RSD	3.9%	34.0%		3.9%		71.3%	51.3%

Quantities Measured in Final Solids

AFA Level (mg/L)		Cr (µg)	Fe (µg)	Mn (µg)	U (µg)	Zn (µg)	$^{239+240}$Pu (µCi)	^{238}Pu + ^{241}Am (µCi)
0	Average	24,000	710,000	610,000	84,381	5,700	70	1.8
	Stdev	2,700	98,000	33,000	22,881	550	2	0.04
	RSD	11%	14%	5.4%	27%	9.7%	2.8%	2.3%
700	Average	58,394	780,000	590,000	66,081	5,700	23	1.9
	Stdev	28,555	44,000	34,000	27,613	540	2	0.08
	RSD	48.9%	5.7%	6%	42%	9.5%	7.8%	4.3%

4.0 Summary and Conclusions

The work described in this report involved testing a Cr(III)-containing simulant for conversion from Cr(III) to Cr(VI) by contact with alkaline solutions containing permanganate in the presence of AFA. The ultimate goal of this work was to determine if the amount of permanganate required to oxidize Cr(III) needs to be adjusted for the presence of AFA because some of the permanganate may be consumed by oxidizing the organic material in the AFA.

A radioactive simulant containing oxides or hydroxides of chromium, manganese, nickel, zinc, uranium, and plutonium was prepared and characterized. Aliquots of the simulant were adjusted to 3 M NaOH for a caustic-leach ratio of 3:1 leachate to settled-solids volume. AFA was added to the test items at dosing rates of 0 to 700 mg AFA per liter of settled solids. The solids were leached first with caustic at 85±5°C, the leachate was removed, and then the solids were washed with dilute caustic solution. The remaining solids underwent oxidative leaching with permanganate at a molar ratio of 1.0 permanganate to Cr(III). The temperatures for oxidative-leach were 25°C or 45°C. Samples of leachate plus washes and residual solids were analyzed at each stage of testing.

The distribution of chromium between simulant waste slurry and the supernate liquids for all caustic and oxidative leaching tests is summarized in Table 4.1. The fraction of chromium remaining in the solids is less than 20% for all tests. These data are presented in Table 4.2. There was some chromium initially soluble. The quantity of soluble chromium did not increase significantly during caustic leaching. The final oxidative-leach shows increased chromium in the liquid with increasing AFA concentration.

For the key elements related to criticality safety, iron, manganese, and nickel, no AFA effects were observed. The radioactive elements in this study were uranium, plutonium, and americium and showed no effects that could be attributed to AFA. The conclusion from these experiments is that there are no AFA effects on caustic-leach or oxidative-leach.

Table 4.1. Chromium Distribution Between Liquid and Solid for Each Phase of Testing

		Weight Fraction Cr					
		Initial		After Caustic-Leach		After Oxidative-Leach	
Test	AFA added (mg/L)	Initial Liquid, 25°C	Initial Solid, 25°C	After CL Liquid, 25°C	After CL Solid, 25°C	After OL Liquid, 25°C	Final Solid, 25°C
8a	0	0.004	0.996	0.009	0.991	0.860	0.140
8b	0	0.004	0.996	0.010	0.990	0.826	0.174
8c	0	0.003	0.997	0.010	0.990	0.895	0.105
1	175	0.003	0.997	0.006	0.994	0.891	0.109
2	350	0.003	0.997	0.007	0.993	0.871	0.129
5	350	0.003	0.997	0.008	0.992	0.957	0.043
4a	700	0.003	0.997	0.009	0.991	0.970	0.030
4b	700	0.003	0.997	0.007	0.993	0.962	0.038
4c	700	0.003	0.997	0.007	0.993	0.957	0.043
10	700	0.003	0.997	0.006	0.994	0.962	0.038
Test	AFA added (mg/L)	Initial Liquid, 45°C	Initial Solid, 45°C	After CL Liquid, 45°C	After CL Solid, 45°C	After OL Liquid, 45°C	Final Solid, 45°C
3	350	0.004	0.996	0.008	0.992	0.957	0.043
7	350	0.004	0.996	0.008	0.992	0.962	0.038
9	700	0.003	0.997	0.008	0.992	0.950	0.050
6	700	0.003	0.997	0.006	0.994	0.966	0.034

Table 4.2. Summary of Cr Removal by Oxidative-Leach Tests

Temperature	AFA Added, mg/L	Fraction Cr Removed	Data Points	Average	Stdev	RSD
25°C	0	0.95				
25°C	0	0.96				
25°C	0	0.96	3@25, 0	0.96	0.0042	0.44%
25°C	175	0.83	1@25,175	0.83	NA	NA
25°C	350	0.79				
25°C	350	0.89	2@25, 350	0.84	0.066	7.9%
25°C	700	0.86				
25°C	700	0.88				
25°C	700	0.95				
25°C	700	0.96	4@25, 700	0.91	0.052	5.7%
45°C	350	0.95				
45°C	350	0.97	2@45, 350	0.96	0.011	1.1%
45°C	700	0.96				
45°C	700	0.97	2@45, 700	0.96	0.0093	0.96%

NA = Not Applicable

5.0 References

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Appendix A

Email Note from F Damerow (BNI) to RA Peterson (PNNL) Directing PNNL to Not Test Actual Waste

Appendix A: Email Note from F Damerow (BNI) to RA Peterson (PNNL) Directing PNNL to Not Test Actual Waste

From: Damerow, Frederick (WGI) [mailto:fwdamero@bechtel.com]
Sent: Friday, May 08, 2009 8:21 AM
To: Peterson, Reid A
Cc: Sundar, Parameshwaran S; Beeman, Gordon H; Jao, Juai C
Subject: Oxidative Leach Actual Waste Testing

Sundar and I discussed the benefit of the actual waste testing this morning. Based on that and our conversation yesterday, I don't believe we there is enough value to justify the work. Let's proceed to develop the letter report and close-out the scope.

Fred Damerow
R&T Systems and Pretreatment Manager
fwdamero@bechtel.com
off 509-371-3613
cell 509-531-5538
MPF B268

Appendix B

Details Related to Radioactive Simulant Leaching Testing

Appendix B: Details Related to Radioactive Simulant Leaching Testing

The methodology of this approach is as follows: the reported analytical results were multiplied by two dilution factors to obtain the reported estimates for the actual component solution concentrations. The first dilution factor was the dilution made from the combined solution and washes aliquot into a known volume of 1 M nitric acid. This was done to prevent any component precipitations from the time of assay until the time of analysis. The second dilution factor represents the dilution of the leachate solutions by wash solution to get the actual assayed solutions. This dilution factor was obtained by comparing the targeted total solution volume to the actual recovered volume of wash solution and leachate. It should be noted that the targeted solution for the caustic leachate also contains some unknown contribution because of the volume of the centrifuged solids themselves. For this reason, these values should be considered as estimates.

Table B.1. Experimental Information for Leach Tests

Test	Solids/ Test (g)	Mass CL & Washes (g)	Density CL & Washes (g/mL)	CL Test Temperature (°C)	Mass OL & Washes (g)	Density OL & Washes (g/mL)	OL Test Temperature (°C)
1	3.280	265.355	1.079	85±5	280.347	1.022	25±1
2	3.198	269.861	1.079	85±5	273.982	1.015	25±1
3	3.240	270.187	1.098	85±5	280.963	1.014	25±1
4a	3.223	293.408	1.077	85±5	279.221	1.015	25±1
4b	3.229	243.719	1.070	85±5	290.138	1.010	25±1
4c	3.240	267.683	1.086	85±5	265.477	1.013	25±1
5	3.186	265.576	1.077	85±5	303.347	1.015	45±1
6	3.222	265.803	1.076	85±5	300.215	1.012	45±1
7	3.190	238.004	1.081	85±5	300.402	1.011	45±1
8a	3.211	267.978	1.075	85±5	265.446	1.010	25±1
8b	3.227	242.607	1.078	85±5	261.274	1.016	25±1
8c	3.189	253.102	1.079	85±5	271.641	1.013	25±1
9	3.230	228.541	1.083	85±5	293.665	1.014	45±1
10	3.189	253.594	1.078	85±5	265.823	1.010	25±1

CL = Caustic Leachate; OL = Oxidative Leachate

Table B.2. Metals Analysis for Initial Equilibrated Supernatant Solution[(a)]

Sample Number	(Analyte)	Al	Cr	Fe	P	Si	Ag	As	B	Ba	Be	Bi	Ca
AFA-Test 1-CLI-M	(µg/mL)	[3.3]	17	[0.12]	<6.6	69	<0.14	<18	34	[0.33]	<0.01	<2.9	[1.5]
AFA-Test 2-CLI-M	(µg/mL)	[3.3]	17	[0.20]	<6.6	72	[0.20]	<18	35	[0.34]	<0.01	<2.9	[1.6]
AFA-Test 3-CLI-M	(µg/mL)	[3.1]	17	[0.21]	<6.6	70	<0.14	<18	34	[0.22]	<0.01	<2.9	[0.97]
AFA-Test 4a-CLI-M	(µg/mL)	[3.0]	16	[0.26]	<6.6	77	<0.14	<18	32	[0.33]	<0.01	<2.9	[0.99]
AFA-Test 4b-CLI-M	(µg/mL)	[2.9]	16	[0.17]	<6.6	79	<0.14	<18	34	[0.35]	<0.01	<2.9	[1.1]
AFA-Test 4c-CLI-M	(µg/mL)	[2.2]	17	[0.16]	<6.6	73	<0.14	<18	28	[0.14]	<0.01	<2.9	[0.98]
AFA-Test 5-CLI-M	(µg/mL)	[3.3]	16	[0.25]	<6.6	83	<0.14	<18	36	[0.25]	<0.01	<2.9	[0.74]
AFA-Test 6-CLI-M	(µg/mL)	[3.1]	16	<0.100	<6.6	83	<0.14	<18	36	[0.26]	<0.01	<2.9	[1.6]
AFA-Test 7-CLI-M	(µg/mL)	[3.1]	17	[0.14]	<6.6	87	<0.14	<18	36	[0.21]	<0.01	<2.9	[1.5]
AFA-Test 8a-CLI-M	(µg/mL)	[3.3]	16	[0.14]	<6.6	74	<0.14	<18	36	[0.22]	<0.01	<2.9	[1.2]
AFA-Test 8b-CLI-M	(µg/mL)	[3.1]	17	[0.15]	<6.6	65	<0.14	<18	36	[0.20]	<0.01	<2.9	[1.0]
AFA-Test 8c-CLI-M	(µg/mL)	[2.7]	17	[0.16]	<6.6	68	[0.19]	<18	35	[0.20]	<0.01	<2.9	[1.3]
AFA-Test 9-CLI-M	(µg/mL)	[2.8]	17	[0.14]	<6.6	66	<0.14	<18	26	[0.20]	<0.01	<2.9	[1.3]
AFA-Test 10-CLI-M	(µg/mL)	[3.2]	17	<0.1	<6.6	86	<0.14	<18	39	[0.26]	<0.01	<2.9	[1.7]

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.2 (contd)$^{(a)}$

	(Analyte)	Cd	Ce	Co	Cu	Dy	Eu	K	La	Li	Mg	Mn	Mo
AFA-Test 1-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[18]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 2-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[29]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 3-CLI-M	(µg/mL)	<0.28	[0.92]	<0.34	<0.21	<0.22	<0.037	[36]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 4a-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	73	<0.21	[0.39]	<0.13	<0.02	<1.0
AFA-Test 4b-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[48]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 4c-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[47]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 5-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	61	<0.21	<0.07	[0.13]	<0.02	<1.0
AFA-Test 6-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[33]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 7-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[49]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 8a-CLI-M	(µg/mL)	<0.28	[1.4]	<0.34	<0.21	<0.22	<0.037	[49]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 8b-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[51]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 8c-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[49]	<0.21	<0.07	<0.13	<0.02	<1.0
AFA-Test 9-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[56]	<0.21	<0.07	[0.23]	<0.02	<1.0
AFA-Test 10-CLI-M	(µg/mL)	<0.28	<0.67	<0.34	<0.21	<0.22	<0.037	[55]	<0.21	<0.07	<0.13	<0.02	<1.0

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.2 (contd)$^{(a)}$

	(Analyte)	Na	Nd	Ni	Pb	Pd	Rh	Ru	S	Sb	Se	Sn
AFA-Test 1-CLI-M	(µg/mL)	[73,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	[4.2]
AFA-Test 2-CLI-M	(µg/mL)	[73,000]	[1.3]	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	<3.0
AFA-Test 3-CLI-M	(µg/mL)	[75,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	[4.2]
AFA-Test 4a-CLI-M	(µg/mL)	[110,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	[3.8]
AFA-Test 4b-CLI-M	(µg/mL)	[73,000]	[1.4]	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	<3.0
AFA-Test 4c-CLI-M	(µg/mL)	[75,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	[14]	<3.2	<18	<3.0
AFA-Test 5-CLI-M	(µg/mL)	[86,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	<3.0
AFA-Test 6-CLI-M	(µg/mL)	[74,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	[4.4]
AFA-Test 7-CLI-M	(µg/mL)	[74,000]	<0.90	<0.510	<3.4	[0.81]	<1.2	<0.97	<12	<3.2	<18	<3.0
AFA-Test 8a-CLI-M	(µg/mL)	[74,000]	[1.5]	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	<3.0
AFA-Test 8b-CLI-M	(µg/mL)	[75,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	[3.4]
AFA-Test 8c-CLI-M	(µg/mL)	[75,000]	[0.98]	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	<3.0
AFA-Test 9-CLI-M	(µg/mL)	[75,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	<3.0
AFA-Test 10-CLI-M	(µg/mL)	[75,000]	<0.90	<0.510	<3.4	<0.77	<1.2	<0.97	<12	<3.2	<18	<3.0

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.2 (contd)[(a)]

	(Analyte)	Sr	Ta	Te	Th	Ti	Tl	U	V	W	Y	Zn	Zr
AFA-Test 1-CLI-M	(µg/mL)	[0.01]	<1.6	<2.6	<0.87	<0.046	<9.0	[22]	[0.11]	<2.5	<0.034	[3.1]	<0.12
AFA-Test 2-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	<0.046	<9.0	[21]	[0.22]	<2.5	<0.034	[3.0]	<0.12
AFA-Test 3-CLI-M	(µg/mL)	[0.01]	<1.6	<2.6	<0.87	<0.046	<9.0	[19]	[0.11]	<2.5	<0.034	[2.5]	<0.12
AFA-Test 4a-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	<0.046	<9.0	[22]	[0.14]	<2.5	<0.034	[2.9]	<0.12
AFA-Test 4b-CLI-M	(µg/mL)	[0.01]	<1.6	<2.6	<0.87	<0.046	<9.0	[21]	[0.19]	<2.5	<0.034	[2.8]	<0.12
AFA-Test 4c-CLI-M	(µg/mL)	[0.01]	<1.6	<2.6	<0.87	<0.046	<9.0	[21]	[0.07]	<2.5	<0.034	[2.0]	<0.12
AFA-Test 5-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	<0.046	<9.0	[21]	[0.18]	<2.5	<0.034	[2.6]	<0.12
AFA-Test 6-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	<0.046	<9.0	[18]	[0.10]	<2.5	[0.04]	[1.9]	<0.12
AFA-Test 7-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	<0.046	<9.0	[19]	[0.15]	<2.5	<0.034	7.70	120
AFA-Test 8a-CLI-M	(µg/mL)	[0.01]	<1.6	<2.6	<0.87	<0.046	<9.0	[21]	[0.16]	<2.5	<0.034	[2.2]	<0.12
AFA-Test 8b-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	<0.046	<9.0	[23]	[0.20]	<2.5	<0.034	[2.6]	<0.12
AFA-Test 8c-CLI-M	(µg/mL)	[0.01]	<1.6	<2.6	<0.87	<0.046	<9.0	[23]	[0.08]	<2.5	<0.034	[2.8]	<0.12
AFA-Test 9-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	<0.046	<9.0	[22]	[0.21]	<2.5	<0.034	[2.7]	<0.12
AFA-Test 10-CLI-M	(µg/mL)	[0.02]	<1.6	<2.6	<0.87	[0.06]	<9.0	[24]	[0.08]	<2.5	<0.034	[2.5]	[0.14]

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.3. Metals Analysis for Initial Equilibrated Simulated Tank Waste Solids[(a)]

Sample Number	(Analyte)	Al	Cr	Fe	P	Si	Ag	As	B	Ba	Be
AFA-Test-1-CLI-S	(µg/g)	<3,900	120,000	160,000	<730	[23,000]	<20	<1,800	1,900	880	<0.65
AFA-Test-2-CLI-S	(µg/g)	<2,600	79,000	110,000	<480	40,000	<13	<1,200	3,700	640	<0.43
AFA-Test-3-CLI-S	(µg/g)	<3,500	110,000	150,000	<660	[25,000]	<18	<1,600	2,200	820	<0.58
AFA-Test-4a-CLI-S	(µg/g)	<3,400	110,000	140,000	<630	29,000	<18	<1,500	2,400	810	<0.56
AFA-Test-4b-CLI-S	(µg/g)	<3,800	120,000	160,000	<710	[26,000]	<20	<1,700	2,200	890	<0.63
AFA-Test-4c-CLI-S	(µg/g)	<3,200	97,000	140,000	<590	[14,000]	<17	<1,400	1,200	710	<0.53
AFA-Test-5-CLI-S	(µg/g)	<3,500	100,000	150,000	<650	[13,000]	<18	<1,600	1,100	780	<0.58
AFA-Test-6-CLI-S	(µg/g)	<3,400	100,000	140,000	<630	[4,900]	<17	<1,500	[420]	800	<0.56
AFA-Test-7-CLI-S	(µg/g)	<3,100	92,000	120,000	<580	[16,000]	<16	<1,400	1,400	730	<0.52
AFA-Test-8a-CLI-S	(µg/g)	<3,100	86,000	110,000	<570	[19,000]	<16	<1,400	1,700	680	<0.51
AFA-Test-8b-CLI-S	(µg/g)	<2,900	88,000	120,000	<540	[15,000]	<15	<1,300	1,300	690	<0.48
AFA-Test-8c-CLI-S	(µg/g)	<2,000	57,000	76,000	<370	17,000	<10	<890	1,600	450	<0.33
AFA-Test-9-CLI-S	(µg/g)	<2,600	110,000	150,000	<480	[6,900]	<13	<1,200	[680]	880	<0.43
AFA-Test-10-CLI-S	(µg/g)	<1,900	99,000	130,000	<350	[13,000]	<10	<850	1,300	770	[0.58]

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.3 (contd)[a]

	(Analyte)	Bi	Ca	Cd	Ce	Co	Cu	Dy	Eu	K	La	Li	Mg
AFA-Test 1-CLI-S	(µg/g)	<320	<15,000	<20	[160]	<36	[77]	<39	<5	na	[62]	[32]	[220]
AFA-Test 2-CLI-S	(µg/g)	<220	<9,900	<13	[160]	[34]	[160]	<26	<3	na	[130]	[21]	[190]
AFA-Test 3-CLI-S	(µg/g)	<290	<13,000	<18	[290]	<33	[92]	<35	<5	na	[64]	[24]	[160]
AFA-Test 4a-CLI-S	(µg/g)	<280	<13,000	[18]	[120]	[36]	270	<34	<4	na	[93]	<22	[170]
AFA-Test 4b-CLI-S	(µg/g)	<310	<14,000	<20	[150]	<35	[80]	<38	<5	na	[65]	[32]	[190]
AFA-Test 4c-CLI-S	(µg/g)	<260	<12,000	<17	[150]	[65]	[66]	<32	<4	na	<30	[21]	[220]
AFA-Test 5-CLI-S	(µg/g)	<290	<13,000	<18	[230]	[71]	2,300	<35	<5	na	[80]	<23	[390]
AFA-Test 6-CLI-S	(µg/g)	<280	<13,000	<17	[260]	<31	[89]	<34	<4	na	[120]	[24]	[110]
AFA-Test 7-CLI-S	(µg/g)	<260	<12,000	<16	<90	<29	[53]	<31	<4	na	[56]	[27]	[140]
AFA-Test 8a-CLI-S	(µg/g)	<250	<12,000	<16	<88	<29	[26]	<31	<4	na	[29]	<20	[140]
AFA-Test 8b-CLI-S	(µg/g)	<240	<11,000	<15	<83	<27	<20.00	<29	<4	na	[33]	<19	[110]
AFA-Test 8c-CLI-S	(µg/g)	<160	<7,500	<10	<57	[25]	[66]	<20	<3	na	<18	<13	[100]
AFA-Test 9-CLI-S	(µg/g)	<210	<9,800	<13	[92]	<24	[52]	<26	<3	na	[32]	<17	[75]
AFA-Test 10-CLI-S	(µg/g)	<160	<7,200	<10	[86]	[27]	[31]	<19	<2	na	[34]	[14]	[95]

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.3 (contd)[a]

	(Analyte)	Mn	Mo	Na	Nd	Ni	Pb	Pd	Rh	Ru	S	Sb
AFA-Test 1-CLI-S	(µg/g)	240	<97	[170,000]	[240]	na	<430	<86	<180	<90	<4,400	<420
AFA-Test 2-CLI-S	(µg/g)	170	<65	240,000	[210]	na	<290	<57	<120	<60	<3,000	<280
AFA-Test 3-CLI-S	(µg/g)	180	<87	[200,000]	[240]	na	<390	<78	<160	<81	<4,000	<380
AFA-Test 4a-CLI-S	(µg/g)	370	<84	[200,000]	[250]	na	<370	<75	<150	<78	<3,900	<360
AFA-Test 4b-CLI-S	(µg/g)	300	<94	[190,000]	[110]	na	<420	<84	<170	<88	<4,300	<410
AFA-Test 4c-CLI-S	(µg/g)	160	<79	[200,000]	[170]	na	<350	<70	<140	<74	<3,600	<340
AFA-Test 5-CLI-S	(µg/g)	640	<87	[180,000]	[240]	na	<390	<77	<160	<81	<4,000	<380
AFA-Test 6-CLI-S	(µg/g)	[76]	<84	[180,000]	[230]	na	<370	<74	<150	<78	<3,800	<360
AFA-Test 7-CLI-S	(µg/g)	[62]	<78	210,000	[120]	na	<350	<69	<140	<73	<3,600	<340
AFA-Test 8a-CLI-S	(µg/g)	[36]	<76	[200,000]	[78]	na	<340	<68	<140	<71	<3,500	<330
AFA-Test 8b-CLI-S	(µg/g)	[11]	<72	210,000	<64	na	<320	<64	<130	<67	<3,300	<310
AFA-Test 8c-CLI-S	(µg/g)	<7.50	<49	270,000	<44	na	<220	<44	<89	<46	<2,300	<210
AFA-Test 9-CLI-S	(µg/g)	<9.80	<64	[160,000]	<57	na	<290	<57	<120	<60	<3,000	<280
AFA-Test 10-CLI-S	(µg/g)	<7.20	<47	180,000	[87]	na	<210	<42	<85	<44	<2,100	<200

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.3 (contd)[(a)]

	(Analyte)	Se	Sn	Sr	Ta	Te	Th	Ti	Tl	U	V	W
AFA-Test 1-CLI-S	(µg/g)	<1,300	<290	<3	<230	<300	<97	[29]	<350	<480	<37	<280
AFA-Test 2-CLI-S	(µg/g)	<980	<220	<2	<180	<230	<75	<5	<270	<370	<29	<210
AFA-Test 3-CLI-S	(µg/g)	<1,500	<340	1,700	<270	<350	<110	[11]	<400	[2,800]	<43	<320
AFA-Test 4a-CLI-S	(µg/g)	<990	<220	1,100	<180	<230	<75	[11]	<270	[2,000]	<29	<220
AFA-Test 4b-CLI-S	(µg/g)	<1,300	<300	1,500	<240	<320	<100	[13]	<360	[1,200]	<39	<290
AFA-Test 4c-CLI-S	(µg/g)	<1,300	<290	1,500	<230	<300	<98	[12]	<350	[2,500]	<37	<280
AFA-Test 5-CLI-S	(µg/g)	<1,400	<330	1,700	<260	<340	<110	<7	<390	[2,900]	<42	<310
AFA-Test 6-CLI-S	(µg/g)	<1,200	<280	1,400	<220	<290	<93	[8.1]	<330	[1,700]	<35	<260
AFA-Test 7-CLI-S	(µg/g)	<1,300	<300	1,500	<240	<310	<100	[16]	[390]	[2,800]	<39	<290
AFA-Test 8a-CLI-S	(µg/g)	<1,300	<290	1,500	<230	<300	<98	<6	<350	[3,500]	<37	<280
AFA-Test 8b-CLI-S	(µg/g)	<1,200	<270	1,300	<220	<280	<91	<6	<320	[1,900]	<35	<260
AFA-Test 8c-CLI-S	(µg/g)	<1,200	<260	1,200	<210	<270	<89	<5	[320]	[2,800]	<34	<250
AFA-Test 9-CLI-S	(µg/g)	<1,100	<250	1,300	<200	<260	<84	<5	<300	[3,400]	<32	<240
AFA-Test 10-CLI-S	(µg/g)	<750	<170	810	<140	<180	<58	[8.7]	<210	[1,900]	<22	<160

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.3 (contd)(a)

	(Analyte)	Y	Zn	Zr
AFA-Test 1-CLI-S	(µg/g)	<3	1,200	<19
AFA-Test 2-CLI-S	(µg/g)	<3	[150]	<14
AFA-Test 3-CLI-S	(µg/g)	<4	1,400	330
AFA-Test 4a-CLI-S	(µg/g)	<3	1,100	420
AFA-Test 4b-CLI-S	(µg/g)	<4	1,400	310
AFA-Test 4c-CLI-S	(µg/g)	<3	1,400	370
AFA-Test 5-CLI-S	(µg/g)	<4	1,500	410
AFA-Test 6-CLI-S	(µg/g)	<3	1,200	220
AFA-Test 7-CLI-S	(µg/g)	<4	2,600	250
AFA-Test 8a-CLI-S	(µg/g)	<3	1,200	840
AFA-Test 8b-CLI-S	(µg/g)	<3	1,100	[150]
AFA-Test 8c-CLI-S	(µg/g)	<3	960	[130]
AFA-Test 9-CLI-S	(µg/g)	<3	1,200	[80]
AFA-Test 10-CLI-S	(µg/g)	<2	700	[39]

(a) Values in brackets [] are ≥ the method detection limit, but are < the estimated quantitation limit, with errors likely to exceed 15%.

Table B.4. Measured Nonradioactive Component Concentrations for Combined Leachate & Wash Solutions

Test	Cr (µg/mL)		Fe (µg/mL)		Mn (µg/mL)		Ni (µg/mL)		U (µg/mL)	
	CL	OL	CL	OL	CL	OL	CL	OL	CL	OL
1	18.4	2,020	[0.66]	0.18	<0.02	<0.04	<0.51	<0.03	[13]	[1.80]
2	19.4	1,960	[0.33]	0.22	<0.02	<0.04	<0.51	<0.03	[14]	6.46
3	20.1	2,010	[0.33]	0.26	<0.02	<0.04	<0.51	<0.03	[18]	6.48
4a	13.5	2,010	[0.18]	0.18	<0.02	<0.04	<0.51	<0.03	[16]	2.65
4b	13.6	1,910	[0.23]	[0.16]	<0.02	<0.04	<0.51	<0.03	[13]	2.35
4c	16.6	2,250	[0.25]	[0.095]	<0.02	<0.04	<0.51	<0.03	[17]	5.44
5	17.6	2,070	[0.31]	0.24	<0.02	4.53	<0.51	<0.03	[14]	3.32
6	17.6	2,030	[0.27]	[0.11]	<0.02	<0.04	<0.51	<0.03	[14]	3.34
7	12.7	2,000	[0.24]	[0.11]	<0.02	<0.04	<0.51	<0.03	[17]	6.59
8a	12.9	2,260	[0.33]	[0.14]	<0.02	<0.04	<0.51	<0.03	[16]	4.17
8b	17.1	2,330	[0.24]	[0.13]	<0.02	<0.04	<0.51	<0.03	[24]	[2.00]
8c	17.1	2,070	<0.100	[0.11]	<0.02	15.50	<0.51	<0.03	[13]	[1.70]
9	20.3	2,010	[0.39]	0.73	<0.02	<0.01	<0.51	<0.03	[17]	[1.90]
10	12.0	2,240	[0.25]	[0.13]	<0.02	<10.10	<0.51	<0.03	[16]	[2.00]

CL = Caustic Leachate; OL = Oxidative Leachate

Table B.5. Measured Radioactive Component Concentrations for Combined Leachate & Wash Solutions

$^{239+240}$Pu (µCi/mL)		^{238}Pu + ^{241}Am (µCi/mL)	
CL	OL	CL	OL
1.47E-05	6.06E-05	4.38E-07	1.33E-06
1.50E-05	4.20E-05	<4.E-07	8.51E-07
2.35E-05	2.96E-05	4.42E-07	6.14E-07
2.21E-05	2.36E-05	5.07E-07	<5.E-07
1.91E-05	1.11E-05	<5.E-07	<6.E-07
2.76E-05	5.01E-06	<4.E-07	<2.E-07
1.43E-05	6.11E-04	<5.E-07	1.18E-05
1.46E-05	7.17E-06	3.27E-07	<2.E-07
2.90E-05	4.01E-04	4.51E-07	7.11E-06
2.42E-05	2.32E-06	4.74E-07	<2.E-07
1.94E-05	1.13E-06	2.81E-07	<2.E-07
1.41E-05	8.72E-05	2.45E-07	1.54E-06
1.64E-05	1.27E-06	2.88E-07	<1.E-07
4.46E-05	8.59E-05	7.39E-07	1.65E-06

CL = Caustic Leachate; OL = Oxidative Leachate

Table B.6. Residual Solids Mass and Component Concentrations

Test	Residual Mass (g)	Cr (μg/g)	Fe (μg/g)	Mn (μg/g)	Ni (μg/g)	U (μg/g)	Zn (μg/g)	$^{239+240}$Pu, from AEA, μCi/g	^{238}Pu+^{241}Am, from AEA, μCi/g
1	3.067	29,400	257,000	184,000	NA	14,900	1,860	19.7	5.93E-01
2	3.005	37,200	226,000	166,000	NA	14,600	1,600	20.7	5.48E-01
3	3.038	21,500	251,000	186,000	NA	14,200	1,830	21.4	5.84E-01
4a	3.001	22,500	275,000	208,000	NA	19,700	2,090	20.8	5.90E-01
4b	3.044	26,700	243,000	183,000	NA	14,000	1,710	22.9	6.14E-01
4c	3.016	8,750	253,000	195,000	NA	32,000	1,860	24.4	6.39E-01
5	3.015	6,300	262,000	203,000	NA	13,900	1,950	24.8	6.38E-01
6	2.979	8,010	236,000	200,000	NA	33,300	2,020	24.4	5.65E-01
7	3.086	8,730	246,000	196,000	NA	23,100	1,990	25.1	6.25E-01
8a	3.002	7,830	250,000	197,000	NA	23,900	1,960	23.2	5.78E-01
8b	3.070	8,810	253,000	195,000	NA	23,000	1,980	23.3	5.84E-01
8c	3.019	7,220	197,000	216,000	NA	36,700	1,670	22.4	6.01E-01
9	3.086	10,000	264,000	197,000	NA	13,500	2,220	24.0	6.27E-01
10	3.063	6,740	245,000	192,000	NA	28,400	2,370	23.9	5.78E-01

NA= Not Available; Ni crucible used for solids fusion

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