

**Preliminary Evaluation of Spent
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Resulting from Gaseous Radioiodine
Control at Hanford's Waste
Treatment Plant**

R. D. Scheele
C. F. Wend
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A. E. Kozelisky
R. L. Sell

December 2002

Prepared for Bechtel National, Inc.
under Contract No. 24590-101-TSA-W0000-0004

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Test specification: 24590-WTP-TSP-RT-01-013
Test plan: TP-RPP-WTP-109
Test exceptions: Three
R&T focus area: HLW Offgas Secondary System
Test Scoping Statement(s): HLW Trend (TN-24590-01-00007m RT-
01)—Risk Register HLW-20

Battelle - Pacific Northwest Division
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by 24590-WTP-TSP-RT-01-013 and TP-RPP-WTP-109. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

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

Date

*G. Todd Wright, Manager
Research and Technology*

Date

Abstract

Battelle's Pacific Northwest Division determined the performance of selected spent-silver mordenite disposal forms in support of Bechtel National, Inc.'s efforts to identify a regulatory-compliant waste form for spent-silver mordenite used to control radioiodine releases from Hanford's Waste Treatment Plant. The objective of this work did not include optimization of the disposal form. In our qualification testing of silver mordenite, reduced silver mordenite, fluoride-treated silver mordenite, iodine-loaded reduced silver mordenite, and their grouted forms, we found that grouted silver mordenite with added calcium iodide and grouted iodine-loaded reduced silver mordenite released the U.S. Environmental Protection Agency and Washington State regulated metals at levels below the EPA's and Washington State's Universal Treatment Standards for land disposal.

Summary

Objectives

Battelle's Pacific Northwest Division (PNWD) performed an initial scoping study to identify potential waste forms for disposal of spent-silver mordenite. This was done to support Bechtel National, Inc.'s (BNI) effort to identify and develop a regulatory-compliant waste form for disposal of the silver mordenite^(a) to be used to control radioiodine emissions from Hanford's Waste Treatment Plant (WTP). The WTP is being designed to vitrify some of Hanford's stored high level waste (HLW). The objective of this scoping study was to identify a potential disposal form for spent hydrogen-reduced silver mordenite (Ag^oZ) and did not include optimization of the disposal form. This work successfully accomplished the goals of the test specification 24590 WTP-TSP-RT-01-013^(b) and test plan TP-RPP-WTP-109^(c) by determining that a regulatory-compliant waste form is available.

For this testing, BNI chose grout for immobilizing spent-silver mordenite. For land disposal, any silver mordenite disposal form must meet both Federal (40 CFR 268; 40 CFR 261) and Washington State (WAC 2000a; WAC 2000b) regulatory limits; WAC 173-303-140 (WAC 2000b) limits treated waste TCLP releases by reference to Federal standards.

Conduct of Testing

In this study, PNWD purchased C*Chem[®]'s IONEX Type Ag-900 silver mordenite (AgZ) and prepared Ag^oZ, hydrogen fluoride-treated AgZ (AgZF), elemental iodine loaded Ag^oZ (Ag^oZI), grout without additives, grouted AgZ, grouted AgZ with added calcium iodide, grouted Ag^oZ, grouted AgZF, and grouted Ag^oZI. The halogen-loaded silver mordenites were chosen to provide materials at least partially representative of spent-silver mordenite after exposure to the mixed halogen-containing melter off-gas (MOG). The effects of NO_x and SO_x were not studied. We prepared triplicate 100-g grout samples at a 25-wt% waste loading.

To assess the AgZ waste-disposal form's performance, PNWD contracted a Washington State accredited laboratory (Severn-Trent STL) to measure the Resource Conservation and Recovery Act (RCRA) hazardous and Washington State Dangerous Metal Toxicity Characteristics as determined using the U.S. Environmental Protection Agency's (EPA's) Toxicity Characteristic Leach Procedure (TCLP) Method 1311 (EPA 2001). The TCLP testing laboratory measured the metals, excluding mercury, using EPA's procedures 6010B (EPA 2001). The laboratory used the EPA's method 7470A to measure mercury (EPA 2001).

(a) The text "silver mordenite" is used in this document to represent the family of silver mordenite forms tested where the acronym AgZ is used for discussions regarding unreduced or as-received silver mordenite.

(b) Test Specification: *Stabilization of Spent Silver Mordenite Disposal*, 24590-WTP-TSP-RT-01-013, S Kelly, Jr. September 20, 2001, Bechtel National Incorporated, Richland, Washington.

(c) Test Plan: *Disposal Treatment Requirements for Spent Silver Mordenite*, TP-RPP-WTP-109, Rev 0, RD Scheele, October 11, 2001, Battelle Pacific Northwest Division, Richland, Washington.

We submitted 100-g triplicate samples to the TCLP-testing laboratory to determine the concentrations of metals regulated as underlying hazardous constituents (UHCs) in the EPA’s Universal Non-Wastewater Treatment Standard (UTS) (40 CFR 268). The metals regulated as UHCs include all the metals regulated based on their toxicity characteristic (TC). The UTS metals are silver, arsenic, barium, beryllium, cadmium, chromium, nickel, lead, antimony, selenium, thallium, and mercury. Vanadium and zinc are not considered UHCs even though they appear in the UTS (40 CFR 268); we provide vanadium and zinc for information only. Washington State as an authorized state uses the same waste codes as the EPA for TC metals.

Results and Performance against Objectives

All of the UHCs, excluding silver, released during TCLP testing were below Washington State and Federal regulatory levels for untreated and treated wastes. Silver was the only Washington State or EPA UHC found in TCLP leachates above regulatory levels. As Table S.1 and Figure S.1 show, some of the silver mordenite disposal forms released silver levels below all regulatory limits. Of the ungrouted silver mordenites, only Ag°Z had silver TCLP releases below the Washington State and the EPA toxicity characteristic designation level of 5.0 mg Ag/L leachate; however, Ag°Z did exceed the EPA’s UTS of 0.14 mg Ag/L. Grouting reduced the silver TCLP release concentrations of AgZ and Ag°ZI to below 5 mg Ag/L and successfully lowered the silver release from Ag°ZI to less than 0.14 mg Ag/L. Adding CaI₂ to the grouted AgZ yielded similar success, reducing the grouted AgZ’s release level to below the UTS TC level. This latter result indicates that adding a grout-compatible soluble iodide can effectively aid in reducing the silver release to below regulatory designation levels.

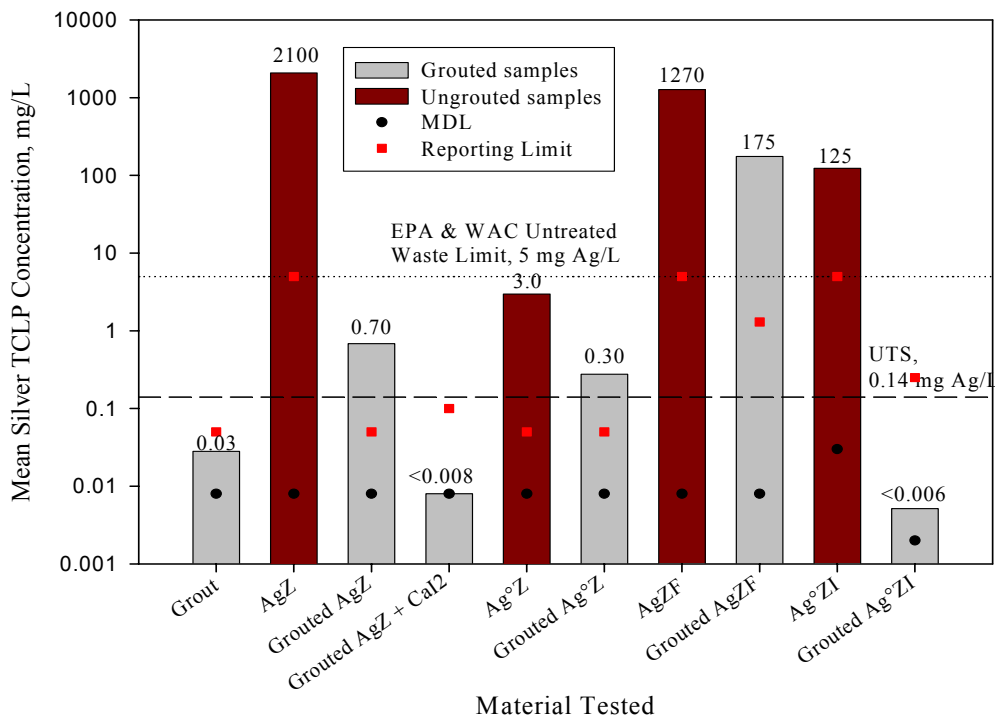


Figure S.1. Mean Silver TCLP Releases for Tested Silver Mordenite Disposal Test Forms

We recommend additional studies to fill in missing knowledge gaps on the behavior of potential spent Ag[°]Z forms and to optimize the spent Ag[°]Z disposal form. The recommended studies include testing untreated and grouted HF-treated Ag[°]Z and chlorine-loaded Ag[°]Z, possibly investigating the effects of exposure to NO_x and/or SO_x on Ag[°]Z, investigating the effects of exposure to a fully representative simulated MOG, and optimizing grout composition and performance related to regulatory compliance.

In summary, PNWD's studies on identifying a regulatory-compliant disposal form for spent silver mordenite from the WTP indicates that a disposal form can be developed that will limit Washington State dangerous and EPA hazardous constituents to below regulatory limits. These studies found that silver was the only UHC released above regulatory levels and that by adding a grout-compatible soluble iodide, silver TCLP releases can be reduced to below regulatory designation levels.

Quality Requirements

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

The TCLP analyses for silver were performed in accordance with EPA Test Methods for Evaluating Solid Waste - Physical Chemical Methods, SW-846, Third Edition and applicable elements of Bechtel's Quality Assurance Project Plan for Testing Programs Generating Environmental Regulatory Data, PL-24590-QA00001, as delineated in the subcontract to the performing laboratory.

PNWD addressed verification activities by conducting an Independent Technical Review of the final data report in accordance with procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and the reported work satisfied the Test Plan objectives. The TCLP data were validated in accordance with the Data Validation Procedure for Chemical Analysis of Tank Waste and Related Samples, ADMIN-RPP-WTP-02-006, Rev 0.

Table S.1. Mean TCLP Releases for Tested Silver Mordenite Disposal Test Forms. See 40 CFR 261 and 40 CFR 268 for Federal hazardous waste limits and WAC 173-303-090 (WAC 2000a) and WAC 173-303-140 (WAC 2000b) for Washington State dangerous waste limits.

Underlying Hazardous Constituents	Mean TCLP Release Concentration, mg/L ^(b)										Regulatory TCLP Designation Limits, mg/L	
	AgZ	Grout	Grouted AgZ	Grouted AgZ + CaI2	Ag°Z	Grouted Ag°Z	Grouted AgZF	AgZF	Ag°ZI	Grouted Ag°ZI	EPA Hazardous & Washington State Dangerous Waste	EPA Universal Treatment Standard
Silver	2090	<0.028 ^{J, UJ}	0.69 ^J	<0.008 ^{UJ}	3.0 ^J	0.28	175 ^J	1280	124 ^J	<0.006 ^{J, UJ}	5.0	0.14
Arsenic	<0.004	<0.004	<0.0054	<0.004	0.013 ^B	<0.004	<0.0064	<0.008	<0.03 ^{UJ}	0.005 ^J	5.0	5.0
Barium	0.028 ^B	0.71	0.35 ^B	0.56	0.052 ^B	0.52	0.81	0.036 ^B	<0.2 ^{UJ}	0.71 ^J	100.0	21
Beryllium	0.0019 ^B	<0.0009	<0.0012	<0.0005	0.0032 ^B	<0.0005	<0.00084	0.029 ^B	<0.006 ^{UJ}	<0.0006 ^{J, UJ}	None	1.22
Cadmium	<0.0005	<0.0005	<0.0013	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.008 ^{J, UJ}	0.0023 ^J	1.0	0.11
Chromium	<0.005	0.013 ^B	0.017 ^B	0.0082 ^B	<0.0053	0.0081 ^B	0.047 ^{J, B}	0.062 ^B	<0.02 ^{J, UJ}	0.017 ^J	5.0	0.60
Nickel	<0.03	0.084 ^B	<0.03	<0.03	<0.03	0.049 ^B	<0.07	<0.04	0.046 ^{J, B}	0.027 ^J	None	11
Lead	0.01 ^B	<0.004	<0.003	0.0043 ^B	0.057 ^{J, B}	<0.0031 ^{UJ}	<0.012 ^J	0.011 ^{UJ, B}	0.057 ^{UJ, B}	<0.006 ^{UJ}	5.0	0.75
Antimony	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.07 ^{UJ}	<0.0075 ^{J, UJ}	None	1.15
Selenium	<0.011	0.010 ^B	0.013 ^B	0.015 ^B	0.0085 ^B	0.0094 ^B	0.035 ^B	<0.011	<0.03 ^{UJ}	0.0081 ^{UJ}	1.0	5.7
Thallium	<0.01	<0.011	<0.005	<0.005	<0.005	<0.005	<0.005 ^{UJ}	<0.005	<0.08 ^{J, UJ}	<0.008 ^{UJ}	None	0.20
Vanadium ^(a)	<0.02	<0.02 ^{UJ}	<0.02	<0.02	<0.02	<0.02	<0.02 ^{UJ}	<0.04	<0.04 ^{UJ, J}	<0.006 ^{J, UJ}	None	1.6
Zinc ^(a)	0.06 ^{UJ, B}	<0.02 ^{UJ}	<0.02 ^{UJ}	<0.02 ^{UJ}	0.054 ^{UJ, B}	<0.02	<0.02 ^J	0.01 ^B	0.16 ^{UJ, B}	0.011 ^{UJ}	None	4.3
Mercury	<0.0003 ^{UR}	<0.0003 ^{UJ}	<0.0001 ^{UJ}	<0.0001 ^{UJ}	<0.0002 ^{UJ}	<0.0002	<0.001	<0.0001 ^{UJ}	<0.0002 ^{UJ}	<0.00002	0.2	0.025

(a) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.

(b) < values indicate at least one result was less than the MDL and the mean is estimated using the MDL.

U – At least one result in the mean was analyzed for but was not detected. The data should be considered usable for decision-making purposes.

UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.

J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

UR - At least one result in the mean was analyzed for and not detected; however, due to an identified QC deficiency, the data should be considered unusable for decision-making purposes.

B – At least one result in the mean was an estimated value less than the reporting limit and greater than the method detection limit.

Acronyms

AgZ	Silver Ion Exchanged Mordenite
AgZF	Fluoride-loaded Silver Ion Exchanged Mordenite
Ag°Z	Hydrogen Reduced Silver Mordenite (Metallic Silver Present)
Ag°ZI	Iodine-loaded Hydrogen Reduced Silver Mordenite
BNI	Bechtel National, Inc.
CFR	Code of Federal Regulations
DOE	U.S. Department of Energy
DOG	Dissolver Off-gas
EPA	U.S. Environmental Protection Agency
EQL	Estimated Quality Level
HLW	High Level Waste
HWVP	Hanford Waste Vitrification Plant
MDL	Analytical Method Detection Limit
MOG	Melter Off-Gas
PNL	Pacific Northwest Laboratory
PNWD	Pacific Northwest Division
QAPjp	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
TC	Toxicity Characteristic
TCLP	Toxicity Characteristic Leaching Procedure (SW-846 Method 1311)
UHC	Underlying Hazardous Constituent
UTS	Universal Non-Wastewater Treatment Standard (40 CFR 268.48)
WTPSP	Waste Treatment Plant Support Project
WAC	Washington Administrative Code
WTP	Waste Treatment Plant

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Severn Trent Laboratories – St Louis, Inc. performed the Toxicity Characteristic Leach Procedure and performed the analyses and was responsible for some quick turnaround times in the analysis. Special thanks to Marti Ward for her attention to our needs as we all worked toward meeting the early deadline.

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1.0 Introduction

Bechtel National, Inc. (BNI) in support of the U.S. Department of Energy's (DOE) planned disposal of the radioactive wastes stored on the Hanford site located in southeast Washington State, is designing a high level waste (HLW) treatment plant (WTP) to vitrify some of Hanford's HLW. BNI requested that Battelle's Pacific Northwest Division (PNWD) investigate the disposal path for silver mordenite, which is to be used to control radioiodine emissions from the WTP. The need to develop a Resource Conservation and Recovery Act (RCRA) (40 CFR 268; 40 CFR 261) compliant disposal route for spent silver mordenite was identified by HLW trend, TN-24590-01-00007mRT-01. The work by PNWD was authorized by Requisition# 24590-101-TSA-W000-004 Task 4. The focus area is HLW Offgas Secondary System.

Gaseous radioactive iodine, ^{129}I with a half-life of 17 million years, will be released from Hanford's high-level waste during vitrification. Based on information provided by L. Bostic of BNI, radioiodine releases to the environment from the WTP are regulated by Washington State (WAC 1998) and the U.S. Environmental Protection Agency (EPA) (40 CFR 61). The designers of the WTP have chosen reduced silver mordenite (Ag°Z) technology to control gaseous radioiodine releases.

Silver mordenite is a zeolite that has been found in laboratory testing to efficiently remove iodine and resist the acidic gases in simulated dissolver off-gas (DOG) streams for a nuclear fuel reprocessing plant (Burger and Scheele 1983; Scheele, Burger, and Matsuzaki 1983; Scheele, Burger, and Soldat 1984; Scheele, Burger, and Halko 1988). Because of AgZ 's and Ag°Z 's chemical resistances to the constituent gases in a vitrification plant's melter off-gas (MOG), which are similar to a reprocessing plant's DOG, and its high retention factors (RFs) ($> 10^4$ for iodine in laboratory-scale systems) Burger and Scheele (1991) recommended AgZ or Ag°Z for use in treating the Hanford Waste Vitrification Plant's (HWVP) MOG.

The MOG is a complex mixture of highly oxidizing acidic gases such as NO_x , substantial amounts of gaseous water, and various other inorganics, including chlorine, sulfur, and fluorine. The halogens will compete with iodine for the silver sites or could affect the stability of the AgZ . Burger and Scheele (1991) provide the predicted MOG composition for the HWVP MOG with halogen concentrations of 6×10^{-6} mol Cl/L, 4.4×10^{-5} mol F/L, and 2.4×10^{-11} mol I/L.

Because silver is regulated with respect to land disposal by Washington State (WAC 2000a; WAC 2000c), and by the RCRA additional information is required to determine the appropriate disposal-treatment requirements for spent Ag°Z . Based on earlier AgZ disposal testing (Burger, Scheele, and Weimers 1981), BNI selected grout to treat and immobilize the spent AgZ .

To develop the needed disposal information, through Test Specification 24590 WTP-TSP-RT-01-013^(a) BNI requested that Battelle's PNWD prepare several reference, candidate, and worst-case silver-mordenite waste forms and determine their toxicity characteristic (TC) using Washington State and EPA testing and analytical procedures. The test disposal forms selected for evaluation were the untreated

(a) Test Specification: *Stabilization of Spent Silver Mordenite Disposal*, 24590-WTP-TSP-RT-01-013, S Kelly, Jr. September 20, 2001, Bechtel National Incorporated, Richland, Washington.

disposal forms of AgZ, Ag^oZ, HF treated AgZ (AgZF), and elemental iodine-loaded Ag^oZ (Ag^oZI) and the treated disposal forms of grout, grouted AgZ, grouted AgZ with added CaI₂, grouted Ag^oZ, grouted AgZF, and grouted Ag^oZI. Following test plan TP-RPP-WTP-109,^(a) we purchased or prepared these materials and used the EPA's Toxicity Characteristic Leach Procedure (TCLP) (SW-846 Method 1311) coupled with the EPA's analytical methods SW-846 Methods 6010B and 7470A (EPA 2001) to measure the releases of RCRA and Washington State underlying hazardous constituents (UHCs).

This report provides the results of the TCLP testing performed on the selected materials. The objective of this work did not include optimization of the disposal form.

(b) Test Plan: *Disposal Treatment Requirements for Spent Silver Mordenite*, TP-RPP-WTP-109, Rev 0, RD Scheele, October 11, 2001, Battelle Pacific Northwest Division, Richland, Washington.

2.0 Regulatory Land-Disposal Limits

Because both Washington State and EPA regulate the land disposal of silver as a dangerous (WAC 2000a; WAC 2000c) and a hazardous constituent (40 CFR 261; 40 CFR 268), the land disposal of the spent silver mordenite must satisfy existing disposal requirements. The applicable regulations require that wastes having the silver-containing wastes be tested to determine if a disposal form exhibits a TC. If the process waste exhibits a TC (WAC 2000a; 40 CFR 261), then for that waste to be land disposed, it must be treated, and the treated waste must satisfy the requirements provided in the Washington State's WAC 173-303-140 (WAC 2000c) and EPA's 40 CFR 268. To determine if a waste exhibits a TC or once treated can be land disposed, the waste form must be tested using the EPA's TCLP with the extract characterized for the Washington State (WAC 2000c) or RCRA (40 CFR 268) UHCs provided in Table 2.1 using EPA procedures (EPA 2001); Washington State invokes this procedure for their testing requirements (WAC 2000c).

Washington State's WAC 173-303-090 (WAC 2000a) and the EPA's 40 CFR 261 provide the Washington State and EPA Toxicity Characteristic criteria for land disposal of untreated hazardous waste, respectively. The EPA's Universal Non-Wastewater Treatment Standard (UTS) (40 CFR 268; WAC 2000c) provides the Washington State and Federal performance requirements for a treated hazardous waste, such as grouted silver mordenite; WAC 173-303-140 (WAC 2000c) incorporates the UTS by reference. Table 2.1 summarizes the Federal and Washington Toxicity Characteristic designation criteria and land-disposal criteria.

Table 2.1. Regulatory TCLP Release Limits for Untreated and Treated Hazardous Waste (40 CFR 261; WAC 2000a; 40 CFR 268; WAC 2000c)

Permissible TCLP Extract Concentrations, mg/L		
Underlying Hazardous Constituents	EPA & Washington State Untreated Waste Toxicity Characteristic	EPA & Washington State Universal Non-Wastewater Treatment Standard
Silver	5.0	0.14
Arsenic	5.0	5.0
Barium	100.0	21
Beryllium	None	1.22
Cadmium	1.0	0.11
Chromium	5.0	0.60
Nickel	None	11
Lead	5.0	0.75
Antimony	None	1.15
Selenium	1.0	5.7
Thallium	None	0.20
Vanadium ^(a)	None	1.6
Zinc ^(a)	None	4.3
Mercury	0.2	0.025

(a) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.

The first column in Table 2.1 provides the UTS hazardous constituents for all treated wastes. The second column provides the Washington State and Federal Toxicity Characteristic criteria for untreated waste; if a waste is hazardous or dangerous based on the silver-toxicity-characteristic criteria in Column 2 of Table 2.1, zinc and vanadium are not defined as underlying hazardous constituents and therefore are not subject to the UTS and are provided for information only. The third column in Table 2.1 provides both the Washington State and Federal allowable TCLP release limits for any UTS hazardous constituents for both untreated and treated waste forms. For this report, we placed silver at the top of the list in the table since it is the predominant hazardous/dangerous constituent in silver mordenite at 15 wt%.

3.0 Description of Testing

In this scoping study to determine whether a Washington State- and RCRA-compliant waste form could be produced from the spent silver mordenite, we tested reference, suspected worst-case, and candidate silver mordenite disposal forms as required by WAC 173-303-110 (WAC 2000b) and 40 CFR 268. This was done following the EPA’s SW-846 TCLP Method 1311 and the EPA’s acid digestion Methods 7760 for analysis of silver and 3010A for the other metals; it was also performed with analysis per EPA Methods 6010B for metals other than mercury and 7470A for mercury (EPA 2001). The original test plan did not identify method 7470 so it was later added by Test Exception. Table 3.1 lists the materials tested. The objective of this work was not to optimize the waste form but to determine whether a regulatory-compliant waste disposal form existed or if one offered sufficient promise to merit further development.

Table 3.1. Silver Mordenite and Reference Materials Tested

Material	Number of Replicates	Replicate Size, g	Material Size
Grout sans mordenite (control)	3	100	< 1 cm
AgZ	3	100	0.16 cm dia extrudate
Grouted AgZ	3	100	< 1 cm
AgZF	3	100	0.16 cm dia extrudate
Grouted AgZF	3	100	< 1 cm
Grouted AgZ + 10 wt% CaI ₂	3	100	< 1 cm
Ag°Z	3	100	0.16 cm dia extrudate
Grouted Ag°Z	3	100	< 1 cm
Ag°ZI	3	100	0.16 cm dia extrudate
Grouted Ag°ZI	3	100	< 1 cm

PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the quality assurance project plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization. This work was conducted to the quality requirements of NQA-1-1989 and NQA-2a-1990, Part 2.7 as instituted through PNWD’s Waste Treatment Plant Support Project Quality Assurance Requirements and Description (WTPSP) Manual. The measurement and test equipment were compliant with the QA program requirements.

The TCLP analyses for silver were performed in accordance with EPA Test Methods for Evaluating Solid Waste - Physical Chemical Methods, SW-846, Third Edition and applicable elements of Bechtel’s Quality Assurance Project Plan for Testing Programs Generating Environmental Regulatory Data, PL-24590-QA00001, as delineated in the subcontract to the performing laboratory.

The testing of AgZ and grouted AgZ provides reference cases as untreated and treated given the ionic nature of silver in AgZ. Thomas et al. (1977) describe the preparation of AgZ as 1) ion exchange of sodium or hydrogen mordenite by exposure at 60°C to an excess of a soluble silver salt, such as silver nitrate, followed by 2) washing the prepared AgZ with deionized water to remove any residual silver ions, and then 3) baking at 150°C to remove free water. The silver will be in AgZ as ionic silver with limited solubility in pure water.

During TCLP testing, significant silver could be removed from the AgZ because of silver acetate's solubility. In TCLP testing, the 100 g AgZ sample containing 15 g Ag was extracted with 2 L of either 0.064 M sodium acetate, 0.035 M acetic acid, or 0.1 M glacial acetic acid (EPA 2001). The solubility of silver acetate is 0.062 M or 6.7 g Ag/L (Dean 1973) or 13.4 g silver in 2 L. Thus, it is possible to extract nearly all the silver (88%) during TCLP testing of AgZ.

HF-treated AgZ and I₂-loaded Ag^oZ should represent the two bounding cases for spent Ag^oZ exposed to halogens. AgZF should represent the worst case for spent silver mordenite because AgF, the assumed sorption product, is very soluble at 14 M (Weast 1984), although the silver concentration could be controlled by silver acetate's solubility, which is less than silver fluoride's. AgZI should provide the best performance of all the possible spent Ag^oZ forms because of the very low solubility of AgI, $k_{sp} = 8.3 \times 10^{-17}$, again assuming AgI formation (Dean 1973). Using Burger's predicted MOG composition for the HWVP (Burger and Scheele 1991), chlorine is the other significant halogen in the MOG, and although AgCl is quite insoluble, k_{sp} of 1.8×10^{-10} (Dean 1973), it is more soluble than silver iodide. At the predicted ratio of Cl:I of 10⁵, chlorine should be the predominant halogen in the spent Ag^oZ (Burger and Scheele 1991).

Originally we planned to test HF-treated Ag^oZ to represent the worst-case halogen-treated spent Ag^oZ; however, when we calculated the Gibbs free energy for the reaction of HF with silver metal, we found that the reaction with HF was not thermodynamically favored. The calculated free energy at 500 K for the reaction provided in Equation 1 was endothermic ($\Delta G = +100$ kJ/mol F) using Barin's (1989) free-energy data. To improve the likelihood that the HF-treated silver mordenite contained fluoride, in concert with the BNI cognizant engineer, we changed to AgZ because HF reacts exothermically via Equation 2 with Ag₂O ($\Delta G = -1.1$ kJ/mol F). We used silver oxide as the surrogate representative for the ionic silver in AgZ. We know of no available thermodynamic data for silver, neither in AgZ nor for silver silicate or aluminate. Although oxygen and oxides of nitrogen are present in the MOG, we did not consider their effects on the reaction of HF with Ag^oZ because these would likely have to proceed via a two-step mechanism, and we do not know how this will affect the capability of Ag^oZ to sorb HF.



The grout without any added silver mordenite provides the background levels and serves as the control for the grouted samples. To assess the performance of a waste form, it is important to know the behavior of each individual waste form component.

We submitted the samples for TCLP testing and analysis per SW-846 methods 1311, 6010B, and 7470A to a National Environmental Laboratory Accreditation Conference and Washington State Department of Ecology accredited laboratory; Appendix A provides a copy of the Washington State accreditation certificate. Before submittal, the grout without AgZ and the grouted samples were sized to pass through a 9.5-mm sieve as required by SW-846 1311 for TCLP testing. SW-846 Method 1311 required no additional sizing for the AgZ, Ag^oZ, AgZF, and Ag^oZI since they were 0.16-cm dia extrudate and would pass through the 9.5-mm sieve. Although suggested by Method 1311, we did not refrigerate the submitted samples because the high birth temperatures and conditions of AgZ, Ag^oZ, AgZF, and Ag^oZI provide sufficient chemical and thermal stability to prevent degradation of tested materials by reaction with air or by thermal decomposition during their short transport by next-day delivery and before

their analysis within 3 days. Being inorganic and highly stable materials resistant to oxidation, the grouts were not refrigerated.

To assure control of aliquot and sample identities, subsamples were stored or contained in uniquely labeled cans that shielded the material from light. We did not refrigerate nor use any other special storage or shipping protections because the materials are stable at ambient conditions since they were prepared at ambient conditions or at elevated temperatures.

PNWD addressed TCLP test results verification activities by conducting an Independent Technical Review of the final data report in accordance with procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the Test Plan objectives. The TCLP data were validated in accordance with the Data Validation Procedure for Chemical Analysis of Tank Waste and Related Samples, ADMIN-RPP-WTP-02-006, Rev 0.

3.1 Preparation of TCLP Tested Materials

Of the materials TCLP tested, AgZ is the only material commercially available. PNWD prepared the remaining materials as described in this section.

3.1.1 AgZ

Silver-exchanged mordenite was purchased from C*Chem[®]. Based on the vendor-supplied analysis, the as-received IONEX Type Ag 900 AgZ contained 15.2-wt% silver (16.4-wt% silver on a dry basis). The AgZ was purchased without any special sizing as a 0.16-cm-dia extrudate. The as-received AgZ was a light gray as shown in Figure 3.1.

The AgZ was stored at ambient conditions in the C*Chem[®] supplied container until used to prepare the various silver mordenite samples (see Figure 3.2) or subdivided into three 100-g aliquots for TC determination. The three aliquots that were submitted for TC determination were partitioned into three individual uniquely labeled metal containers that prevented light from impinging on the AgZ, and they were stored and shipped at ambient conditions.

3.1.2 Ag[°]Z Preparation

The Ag[°]Z was prepared using methods developed by Thomas and coworkers (1977) and successfully used by Scheele, Burger, and Matsuzaki (1983). First, we treated the purchased AgZ with Ar or N₂ for 3 to 4 h at 300°C to remove residual air and moisture. After drying, flowing H₂ bathed the AgZ for 24 h at 500°C to convert ionic silver within the zeolite to metallic silver. Third, we purged the AgZ with flowing Ar or N₂ to remove residual H₂. The resulting Ag[°]Z was brown as shown in Figure 3.2.



Figure 3.1. Silver Mordenite



Figure 3.2. Reduced Silver Mordenite

The apparatus shown schematically in Figure 3.3 consisted of an inert gas (N_2 or Ar) supply for removing residual oxygen from the AgZ, a supply of hydrogen, a thermometer/thermocouple combination, valves, gas flowmeters, a preheater, a column of AgZ, and pressure gauges at the preheater inlet and AgZ column outlet. Tube furnaces were used for the preheater and to heat the AgZ column.

The first batch of prepared Ag^oZ was stored in a labeled individual metal container that limited exposure of the Ag^oZ to light until submitted for TCLP testing or grouted. The 100-g aliquots submitted for TC determination were removed from the common container and placed into individual labeled metal cans and stored at ambient temperature.

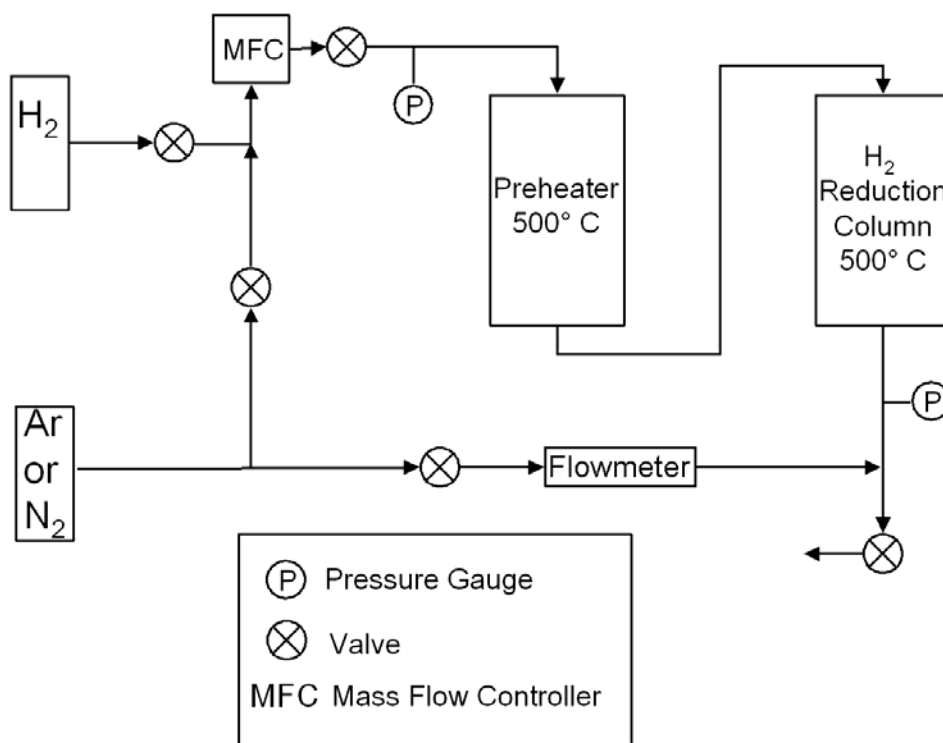


Figure 3.3. Schematic of Hydrogen Reduction System

3.1.3 AgZF Preparation

We prepared the AgZF by treating AgZ with HF at the operational parameters and concentrations provided in Table 3.2 (Scheele and Burger 1987). The operational parameters are based on past operational parameter-optimization studies at the Pacific Northwest Laboratory (PNL) and the Idaho National Engineering Laboratory. We used the HF concentration predicted by Burger and Scheele (1991) for the HWVP MOG. The face velocity and HF concentration are for the gas at 20°C.

Table 3.2. Conditions for Preparation of AgZF and Ag^oZI
(Scheele and Burger 1987; Burger and Scheele 1991)

Operating Parameter	Level
Bed Temperature	150°C
Packing Size	0.16 cm dia extrudate
Bed Diameter	5 cm
Bed length	15 to 25 cm
Face Velocity (Non-Critical)	5 to 7.5 m/min
Halogen (F as HF, I as I ₂) concentration	$4\text{-}5 \times 10^{-5}$ mole/L

The bulk of the gas was slightly humidified to 2 to 20 torr H₂O by bubbling a portion of the supplied air through ambient-temperature water. The stream was slightly humidified to assure that sufficient water was present for any reactions that may involve water and to assure that water is present to provide at least a minimum amount of mobility to the silver within the pores of the zeolite. Because zeolites are good desiccants, the pores should be well saturated with water, and increasing the water concentration in the gas stream should add very little more water to the zeolites. The stream was not fully humidified to the plant level per guidance of the BNI Cognizant Engineer.

The AgZ was treated with a 10% molar excess of the stoichiometric amount of HF (assuming AgF forms) to assure that the material was fully loaded with the HF. In anticipation of AgF formation with its slight yellow color, we visually monitored the column during HF loading; but found no evidence of a color front. A portion of the effluent stream was passed through a laboratory-scale fritted bubbler containing a sodium hydroxide solution to remove the HF and analyzed using a fluoride ion specific electrode. The resulting AgZF was a brownish gray as shown in Figure 3.4.

We used the apparatus shown schematically in Figure 3.5 to prepare the HF-treated AgZ. The system was composed of

- valves to regulate gas flow
- pressure gauges to leak test the apparatus and to monitor pressure drop across the bed
- a 150°C oven
- an air supply (bottled air)
- a water bubbler system to humidify the gases

- a bottle of diluted HF
- calibrated mass-flow controllers to control the HF flow through the AgZ
- mass-flow controllers and gas flowmeters for the bulk gases
- a preheater column
- an AgZ column
- a sodium hydroxide aqueous scrubber for the effluent gases.



Figure 3.4. Fluoride Loaded Silver Mordenite

Both the column and preheater were placed in a 150°C temperature-controlled oven. Teflon[®] and plastic tubing were used to deliver HF and air to the oven. The preheater was a nominal 90-cm stainless steel coil of 6.35-mm OD tubing. The tubing between the preheater and the column was stainless. The AgZ was contained in an acrylic column.

The amount of fluoride loaded onto the AgZ is not well known. Based on mass difference and assuming the 7.1-wt% water content of the AgZ on its day of receipt, 45% of the fluoride loaded onto the AgZ. Using our analysis of the 1 M NaOH scrubber and assuming efficient scrubbing of HF by 1 M NaOH indicates that the AgZ column sorbed nearly all of the HF, including the 10% molar excess (assuming AgF formation) that passed into the column. A small amount of fluoride was found in the hydroxide scrubber.

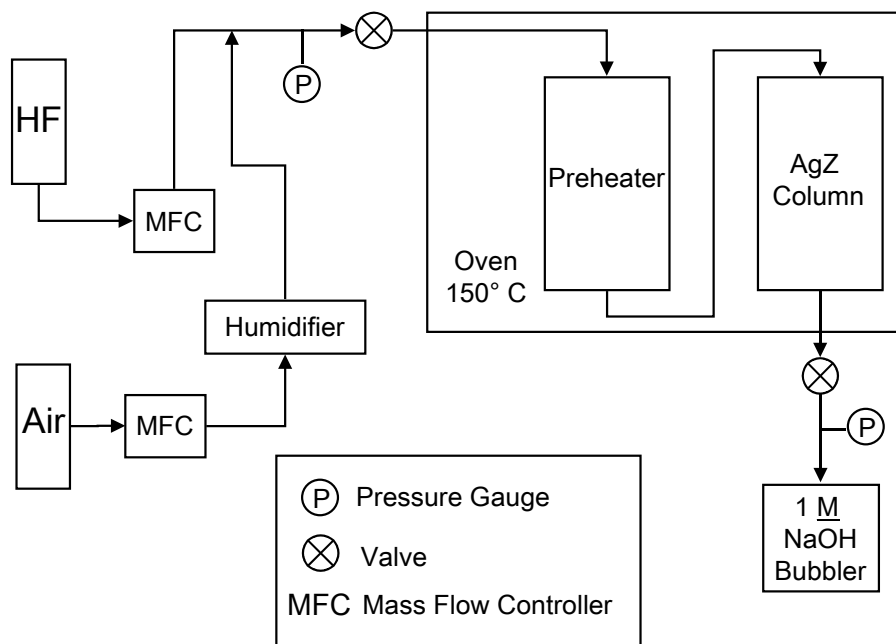


Figure 3.5. HF System Schematic

Before submitting for TCLP testing, the prepared HF-treated mordenite was mixed using a spatula in an attempt to provide a homogenous mix and then stored in a labeled individual metal container until submitted for TC determination or grouted. The 100-g aliquots that were submitted for TC determination were removed from the common container and placed into individual labeled metal jars that eliminated light exposure and stored at ambient conditions.

Ag°ZI Preparation

The Ag°ZI was prepared by treating Ag°Z with a 10% molar excess, assuming AgI formation, of elemental iodine gas at the operational parameters and concentrations provided in Table 3.2 (Scheele and Burger 1987). The operational parameters are based on past operational parameter optimization studies at PNL and the Idaho National Engineering Laboratory. To shorten the length of time required to load the Ag°Z with I₂, we input the iodine at Burger and Scheele's (1991) predicted HWVP MOG fluoride concentration of 4.5×10^{-05} mol F/L rather than the predicted 2.4×10^{-11} mol I/L. The face velocity and I₂ concentration are for the gas at 20°C.

To prepare the Ag°ZI, we passed an I₂-containing humidified gas stream through a preheater and then through a 150°C column of Ag°Z using the apparatus presented schematically in Figure 3.6. A pump provided the bulk, or 92%, of the air. We delivered the required amount of gaseous I₂ by passing air through a 65°C solution of KI saturated with I₂, which provided near equilibrium I₂ concentration of 6.2×10^{-04} mole I/L and humidified the air. Both the column and preheater were placed inside a 150°C temperature-controlled oven. We partitioned the column effluent into two streams, with one portion bubbled through a solution of sodium hydroxide using a laboratory-scale fritted bubbler to remove the I₂ and the second portion passed through a small silver faujasite column to indicate iodine in the effluent. We weighed the bed before and after I loading and analyzed the sodium hydroxide trap solution using an iodide-specific ion electrode and found 96% and 99.5%, respectively, of the theoretical iodine loading, assuming the formation of AgI.

We used chemically compatible materials to transport the gases. Plastic tubing delivered the bulk of the air to the stainless steel preheater. The I₂-laden gas, delivered to the oven in glass tubing, and the preheated air were blended and transported to the glass column in glass tubing.

We visually monitored the I₂ loading on the column by following the bright yellow front as the iodine loaded onto the Ag°Z. Figure 3.8 shows the bright yellow color of the fully loaded Ag°Z at temperature after we concluded the preparation while Figure 3.7 shows how Ag°ZI appears at room temperature.

The Ag°ZI was well mixed to provide a nominally homogenous mix before being packaged into three 100-g aliquots for TCLP testing and analysis and before being grouted. The grouted Ag°ZI monoliths were prepared immediately.

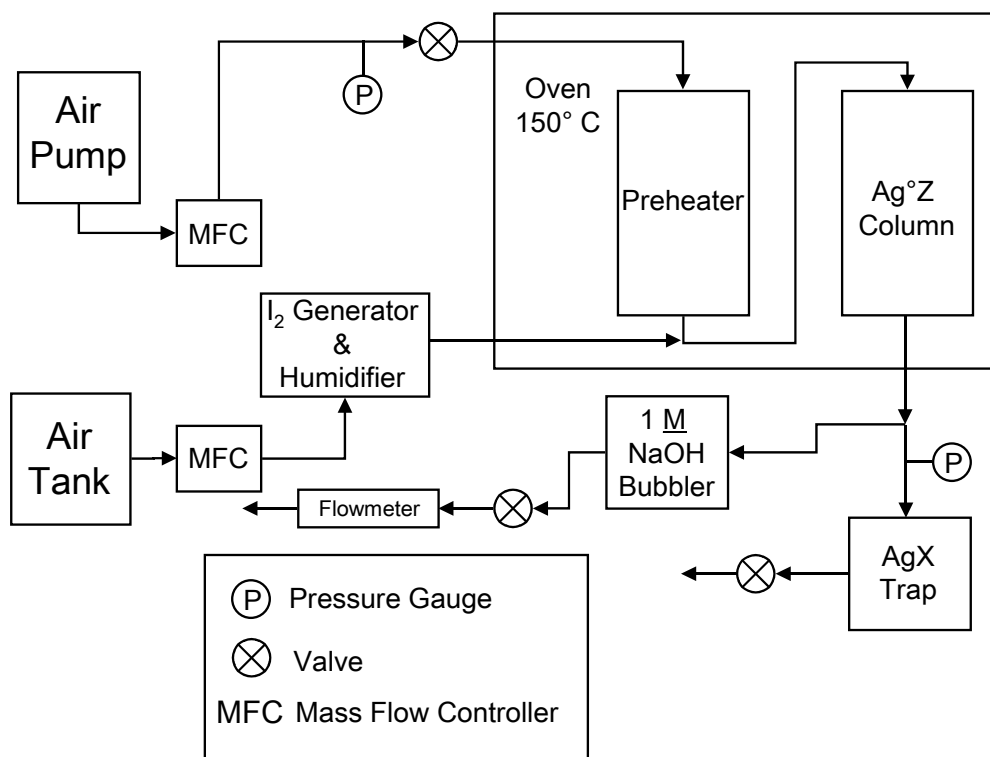


Figure 3.6. Iodine System Schematic

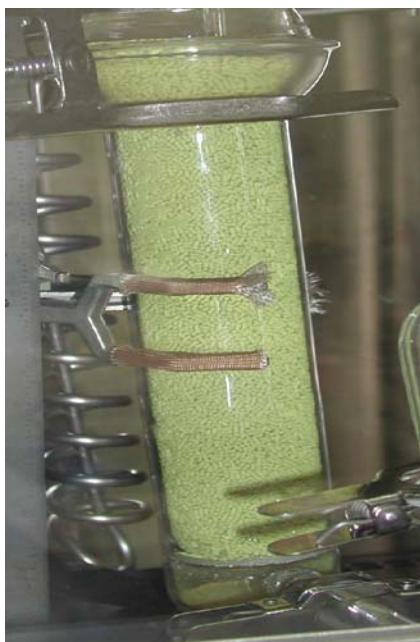


Figure 3.7. I₂-Loaded Ag°Z at 150°C



Figure 3.8. I₂-Loaded Ag°Z at 20°C

3.1.4 Grout Preparation

We prepared six separate grouts for testing per Table 3.1. For each grouted material, we planned to prepare three nominal 110-g replicate grout monoliths using the target composition provided in Table 3.3. Using the target composition proved troublesome in some instances because the treated silver mordenites were dried by reduction or exposure to the halide gases. In consultation with the Cognizant BNI Engineer, the water:cement ratio was allowed to increase until a workable mix was achieved.

Table 3.3. Target Grout Composition

Grout	Portland Type III cement
Water: cement ratio (dry mass basis)	0.30
Waste Loading, mass %	25
Curing time, h	72

The procedure for preparing the grouts was simple. The ingredients were mixed using a spatula to prepare a uniform mixture, transferred to labeled plastic containers, and cured 72 h per direction of the BNI Cognizant Engineer in a 100% humidity atmosphere. We found that it is best if the dry ingredients are mixed together before adding the water. A monolith of grout without any added silver mordenite, which is presented in Figure 3.9 and Figure 3.10, presents grouted AgZ with added CaI₂ sized for TCLP testing.

As shown in Table 3.1, in addition to the AgZ-, Ag^oZ-, AgZF-, and Ag^oZI-containing grouts, we prepared a reference set of grouts without any silver mordenite loading and a second set of grouted AgZ with 10 wt% CaI₂. We added CaI₂ to test a potential approach to reduce silver's solubility. The unloaded grout was prepared to provide a baseline behavior for the grout itself.



Figure 3.9. Portland Type III Grout Monolith



Figure 3.10. Grouted AgZ + CaI₂ Sized for TCLP Testing

Table 3.4 provides the final make-up compositions of the prepared grouts. We were able to prepare the grout without added silver mordenite using the recipe provided in Table 3.3 without difficulty. As shown in Table 3.4, we had to adjust the water-to-cement ratio for the remaining grouts to accommodate the mordenites' desire for water.

Table 3.4. Grout Make-Up Preparations

Mordenite Added	Water, wt%	Dry Cement, wt%	Mordenite, wt%	CaI₂, wt%	Water:Cement ratio
None	23	77	0	0	0.3
AgZ	21	54	25	0	0.4
AgZ, CaI ₂	19	44	26	11	0.44
Ag°Z	21	54	25	0	0.39
AgZF	21	54	25	0	0.39
Ag°ZI	24	51	25	0	0.47

After curing, the cement monoliths were size reduced to less than 1 cm for the TCLP testing and leachate analysis. The three grout monoliths were individually and independently size reduced using a hammer and chisel inside a container to prevent the loss of material. We sieved the size-reduced grout to yield three 100-g replicates required by SW-846, Method 1311. Each recovered individual replicate was assigned and labeled with a unique identity to control, store, and maintain the identity of the samples. The sized grouts were stored in plastic bags inside metal containers at room temperature.

4.0 Results of Toxicity Characterization Determination

All of the grouted and ungrouted samples were submitted, in triplicate, to a National Environmental Laboratory Accreditation Conference and Washington State Department of Ecology accredited laboratory for TCLP testing. The laboratory reported the method detection limit (MDL) and the analyte reporting limit or estimated quality limit (EQL) for each sample run. If a measurement was below the MDL, then it is reported here as less than the MDL.

The MDL is determined according to standard EPA guidelines, and the reporting limit is determined by the analyst to be a value that is statistically different from the MDL (typically 10 times the MDL). The MDL may vary from sample to sample because the analyst may change the dilution of a sample to bring the concentration to within the instrument's calibration range. In practice, the reporting limit or EQL is the level at and above which the analyst believes the method provides an accurate measure of the analyte. Below the reporting limit and above the MDL, the analyst considers the reported value to be an estimate.

In several instances, the matrix spike recoveries for silver and/or mercury in grout-derived solutions were outside the target control range of 75 to 125%, while the laboratory control-sample recoveries were always within the target control range. Based on the laboratory-control-sample recoveries being within control ranges, the instrumentation was functioning properly. When the matrix spike recovery is out of the control range, this suggests that the matrix is releasing an element or combination of elements that interferes with the measurement of an analyte. To eliminate the matrix interference, options include reanalysis using alternative analytical approaches, such as calibrating the instrument with calibration standards in a representative sample matrix or by adding known amounts of analyte to the sample solution; this latter method is known as "known addition" (Kolthoff et al. 1969). It is interesting that the matrix spike recovery outside of normal control levels is not universal for all grout-derived TCLP solutions. We have no explanation for this difference in behavior.

The implications of an out-of-control-range spike recovery depend on the measured concentration in the TCLP extract. Method 1311 recommends that the matrix spike level be at or near the regulatory level (EPA 2001). At this spike level, a poor spike recovery on a sample with a much higher than regulatory level has no impact on the interpretation. If the sample has an analyte concentration near the spike level, the user of the data must evaluate the results and spike recovery. Adjusting the measured result using the matrix recovery is not a valid approach to provide an accurate measure of the true concentration but should be useful to provide a very rough estimate.

The TCLP testing results were validated by PNWD Quality Assurance Staff using a PNWD-authored and BNI-accepted procedure. The data qualifiers applied as a result of this data validation are:

- U - The constituent was analyzed for but was not detected. The data should be considered usable for decision-making purposes.
- UJ - The constituent was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.
- J - Indicated that the constituent was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

- UR - Indicate that the constituent was analyzed for and not detected; however, due to an identified QC deficiency, the data should be considered unusable for decision-making purposes.
- R - Indicates that the constituent was analyzed for and detected; however, due to an identified QC deficiency, the data should be considered unusable for decision-making purposes.

Throughout the discussion in this section, the results are compared to either the untreated waste Toxicity Characteristic limits for the ungrouted samples or the UTS for the grouted samples; the Washington State and EPA limits are the same for each waste disposal form, treated or untreated. See Table 2.1 for these limits. The results of these tests follow.

4.1 Silver Mordenite (AgZ)

Three replicate samples of as-received AgZ were submitted for TCLP testing and leachate analysis. As shown in Table 4.1, all of the UHCs except silver are below both the Washington State and Federal regulatory limits (Table 2.1). Ranging from 1920 to 2250 mg Ag/L, the silver release is several orders of magnitude above the regulatory limits. Because of the poor matrix-spike recovery for mercury of 20%, the mercury result for AgZ must be rejected, even though no mercury was observed in the samples.

Table 4.1. Results of AgZ TCLP Testing

Underlying Hazardous Constituents	Concentration, mg/L					
	Universal Treatment Standard	AgZ ^(c) #1	AgZ ^(c) #2	AgZ ^(c) #3	Reporting Limit	Method Detection Limit
		TCLP Extract Concentration	TCLP Extract Concentration	TCLP Extract Concentration		
Silver ^(a)	0.14	2250	2090	1920	5	0.008
Arsenic	5.0	<0.004	<0.004	<0.004	0.75	0.004
Barium	21	0.019	0.031	0.032	0.5	0.003
Beryllium	1.22	0.0016	0.0016	0.0023	0.13	0.0005
Cadmium	0.11	<0.0005	<0.0005	<0.0005	0.13	0.0005
Chromium	0.60	<0.005	<0.005	<0.005	0.25	0.005
Nickel	11	<0.03	<0.03	<0.03	1	0.03
Lead	0.75	0.0079	0.011	0.011	0.25	0.003
Antimony	1.15	<0.009	<0.009	<0.009	0.25	0.009
Selenium	5.7	<0.007	0.012	0.014	0.13	0.007
Thallium	0.20	<0.005	0.014	0.0092	0.5	0.005
Vanadium ^(b)	1.6	<0.02	<0.02	<0.02	1.3	0.02
Zinc ^(b)	4.3	0.045 UJ	0.068 UJ	0.067 UJ	0.5	0.02
Mercury ^(a)	0.025	<0.0003 UR	<0.0003 UR	<0.0003 UR	0.001	0.0003

(a) Silver spike level <100× matrix silver concentration. Matrix spike recovery was 20% for Mercury.

(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.

(c) Data reported between the reporting limit and the method detection limit are estimated.

UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.

UR - At least one result in the mean was analyzed for and not detected; however, due to an identified QC deficiency, the data should be considered unusable for decision-making purposes.

4.2 Grout Without Silver Mordenite

We submitted triplicate grout samples of Portland Type III Cement with no added silver mordenite for TCLP testing and leachate analysis. This testing was to determine the TCs of the UTS hazardous constituents of the cement used to prepare the grouted silver mordenites tested.

Table 4.2 provides the results of the TCLP tests on the grout. Note that several of the UHCs were found in the TCLP leachates above reporting levels and the MDLs but below the UTS. The reporting limit and MDL in columns 4 and 5 are for the first two samples with the reporting limit, and MDL for the 3rd sample are in columns 7 and 8.

We are surprised by measured silver in the pure Portland cement grout extract. Possible explanations for its presence include silver in the grout or silver from a previous sample with a substantial silver concentration in the transfer line. To explain the observation of silver, further testing is required. To help explain the presence of silver in two of the three replicates, we recommend chemically analyzing the Portland cement used to prepare the grout and/or prepare and TCLP test fresh pure grout samples.

When silver was observed, silver was still below the UTS. The matrix spike recoveries for the batch containing grout, AgZ in grout, and AgZ in grout with CaI₂ amendment for these samples were 54% for silver and 74% for mercury; the sample used for the matrix spike was Grout Without Additives #2. The analyst believes that the grout releases constituents that interfere with the extract analysis since the liquid control spikes were within existing control levels. If one were to use the matrix spike recovery to adjust the measured silver concentration, which is not a valid practice, the mean release is 0.05 mg Ag/L, which remains below the 0.14 mg Ag/L UTS regulatory level. Applying the same approach to mercury does not change the conclusions with respect to mercury concentrations with respect to regulatory levels.

Table 4.2. Grout TCLP Results

Underlying Hazardous Constituents	Concentration, mg/L							
	Universal Treatment Standard	Grout ^(c) #1	Grout ^(c) #2	Reporting Limit	Method Detection Limit	Grout ^(c) #3	Reporting Limit	Method Detection Limit
		TCLP Extract Concentration	TCLP Extract Concentration			TCLP Extract Concentration		
Silver ^(a)	0.14	0.06 J	<0.008 UJ	0.05	0.008	0.016	0.05	0.008
Arsenic	5.0	<0.004	<0.004	0.75	0.004	<0.004	0.75	0.004
Barium	21	0.66	0.78	0.5	0.003	0.69	0.5	0.003
Beryllium	1.22	<0.0005	0.0015	0.13	0.0005	<0.0005	0.13	0.0005
Cadmium	0.11	<0.0005	<0.0005	0.13	0.0005	<0.0005	0.13	0.0005
Chromium	0.60	0.01	0.016	0.25	0.005	0.012	0.25	0.005
Nickel	11	0.083	0.083	1	0.03	0.086	1	0.03
Lead	0.75	0.0045	<0.003	0.25	0.003	0.004	0.25	0.003
Antimony	1.15	<0.009	<0.009	0.25	0.009	<0.009	0.25	0.009
Selenium	5.7	0.012	0.007	0.13	0.007	0.012	0.13	0.007
Thallium	0.20	0.014	<0.005	0.5	0.005	0.009	0.5	0.005
Vanadium ^(b)	1.6	<0.02 UJ	<0.02 UJ	1.25	0.02	<0.02	1.25	0.02
Zinc ^(b)	4.3	<0.02 UJ	<0.02 UJ	0.5	0.02	<0.02 UJ	0.5	0.02
Mercury ^(a)	0.025	<0.0003 UJ	<0.0003 UJ	0.001	0.00003	<0.00006 UJ	0.001	0.00006

(a) Matrix spike recoveries were 54% and 74% for silver and mercury, respectively.
(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.
(c) Data reported between the reporting limit and the method detection limit are estimated.
UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.
J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

4.3 Grouted Silver Mordenite

As shown in Table 4.3, except for silver, the amount of UHCs released during TCLP testing for each of the triplicate grouted AgZ samples are below Washington State and Federal regulatory limits. The performance of the grouted AgZ with respect to these metals is consistent with the performances of AgZ and grout by themselves.

Grouted AgZ is an improved disposal form compared to ungrouted AgZ. The silver released by the grouted AgZ ranged from 0.62 to 0.75 mg Ag/L, all below the EPA’s untreated waste toxicity characteristic levels but exceeding the UTS’s 0.14 mg Ag/L. These TCLP concentrations are well below the 2000-mg Ag/L measured for the AgZ by itself. This reduction indicates significant protection of the AgZ by the grout or a substantially lower release of silver as a result of treatment.

The matrix spike recoveries for the batch containing grout, AgZ in grout, and AgZ in grout with CaI₂ amendment for these samples were 54% for silver and 74% for mercury; the sample used for the matrix spike was Grout without additives #2. For the grouted AgZ, this poor recovery will not affect the

interpretation of the TCLP results with respect to regulatory requirements because all the reported results are above the regulatory levels.

Table 4.3. Grouted Silver Mordenite TCLP Results

Underlying Hazardous Constituents	Concentration, mg/L					
	Universal Treatment Standard	Grouted AgZ ^(c) #1	Grouted AgZ ^(c) #2	Grouted AgZ ^(c) #3	Reporting Limit	Method Detection Limit
		TCLP Extract Concentration	TCLP Extract Concentration	TCLP Extract Concentration		
Silver ^(a)	0.14	0.62 J	0.75 J	0.68 J	0.05	0.008
Arsenic	5.0	<0.004	<0.004	0.0082	0.75	0.004
Barium	21	0.33	0.35	0.35	0.5	0.003
Beryllium	1.22	<0.0005	<0.0005	0.0026	0.13	0.0005
Cadmium	0.11	<0.0005	<0.0005	0.0029	0.13	0.0005
Chromium	0.60	0.016	0.016	0.019	0.25	0.005
Nickel	11	<0.03	<0.03	<0.03	1	0.03
Lead	0.75	<0.003	<0.003	0.003	0.25	0.003
Antimony	1.15	<0.009	<0.009	<0.009	0.25	0.009
Selenium	5.7	0.013	0.013	0.013	0.13	0.007
Thallium	0.20	<0.005	<0.005	<0.005	0.5	0.005
Vanadium ^(b)	1.6	<0.02	<0.02	<0.02	1.3	0.02
Zinc ^(b)	4.3	<0.02 UJ	<0.02 UJ	<0.02 UJ	0.5	0.02
Mercury ^(a)	0.025	<0.00006 UJ	<0.00006 UJ	<0.00006 UJ	0.001	0.00006

(a) Matrix spike recoveries were 54% and 74% for silver and mercury, respectively.
(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.
Data reported between the reporting limit and the method detection limit are estimated.
UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.
J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

Using the poor matrix spike recovery of 54% for silver to adjust the silver concentration, which is not a valid practice, would only increase the suspected result, which is already above the UTS regulatory level and would not affect the conclusion regarding treatment performance with respect to regulatory criteria. Using the 74% matrix spike recovery for mercury to adjust the measured amount would not raise the mercury concentration near the UTS regulatory level of 0.025 mg Hg/L. These rough estimates suggest that no further testing is required to improve matrix spike recoveries.

4.4 Grouted Silver Mordenite with Calcium Iodide Amendment

The addition of 10-wt% CaI₂ to grouted 25 wt% AgZ improves the performance of the grouted AgZ disposal form. Adding CaI₂ reduced the silver concentration in the TCLP leachate to <0.008 mg Ag/L as shown in Table 4.4, which is less than the UTS limits. As with grouted AgZ, all of the other UHCs fall below the Washington State and Federal UTS TCLP concentration limits; see Table 2.1.

The matrix spike recoveries for the batch containing grout, AgZ in grout, and AgZ in grout with CaI₂ amendment for these samples were 54% for silver and 74% for mercury; the sample used for the matrix spike was Grout without additives #2. Using the matrix spike recoveries to estimate silver and mercury concentrations would not affect conclusions regarding compliance with regulatory levels. These rough estimates suggest that no further testing is required to improve matrix spike recoveries.

The reduction in silver release by adding CaI₂ is accomplished due to the very low solubility product of AgI, $k_{sp}=8.3 \times 10^{-17}$ (Dean 1973). CaI₂ is soluble in pure room temperature water at 7 M (Weast 1984); the calcium in the cement will reduce this solubility by an unknown amount because of its own limited solubility. With iodide in solution, any silver that is dissolved will react with the available iodide within the grout, thus effectively preventing the release of silver to the TCLP extract.

Table 4.4. Grouted Silver Mordenite with Calcium Iodide Amendment TCLP Results

Underlying Hazardous Constituents	Universal Treatment Standard	Concentration, mg/L				
		Grouted AgZ with CaI ₂ ^(c) #1	Grouted AgZ with CaI ₂ ^(c) #2	Grouted AgZ with CaI ₂ ^(c) #3	Reporting Limit	Method Detection Limit
		TCLP Extract Concentration	TCLP Extract Concentration	TCLP Extract Concentration		
Silver ^(a)	0.14	<0.008 UJ	<0.008 UJ	<0.008 UJ	0.1	0.008
Arsenic	5.0	<0.004	<0.004	<0.004	0.75	0.004
Barium	21	0.57	0.59	0.52	0.5	0.003
Beryllium	1.22	<0.0005	<0.0005	<0.0005	0.13	0.0005
Cadmium	0.11	<0.0005	<0.0005	<0.0005	0.13	0.0005
Chromium	0.60	0.008	0.0073	0.0092	0.25	0.005
Nickel	11	<0.03	<0.03	<0.03	1	0.03
Lead	0.75	0.0036	0.0045	0.0048	0.25	0.003
Antimony	1.15	<0.009	<0.009	<0.009	0.25	0.009
Selenium	5.7	0.014	0.014	0.015	0.13	0.007
Thallium	0.20	<0.005	<0.005	<0.005	0.5	0.005
Vanadium ^(b)	1.6	<0.02	<0.02	<0.02	1.3	0.02
Zinc ^(b)	4.3	<0.02 UJ	<0.02 UJ	<0.02 UJ	0.5	0.02
Mercury ^(a)	0.025	<0.00006 UJ	<0.00006 UJ	<0.00006 UJ	0.001	0.00006

(a) Matrix spike recoveries were 54% and 74% for silver and mercury, respectively.
(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.
(c) Data reported between the reporting limit and the method detection limit are estimated.
UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.

4.5 Reduced Silver Mordenite

As Table 4.5 shows, Ag^oZ performed well in TCLP testing with respect to all UHCs except for silver. The amounts of all UHCs except silver were below Washington State and Federal UTS concentration limits.

The silver concentrations in the TCLP leachates for the three Ag°Z samples ranged from 2.9 to 3.0 mg Ag/L TCLP leachate or below the Washington State and EPA toxicity characteristic criteria of 5.0 mg Ag/L. The silver concentrations exceeded the EPA’s 0.14 mg Ag/L UTS limit for treated wastes. Ag°Z with its 3 mg Ag/L release level performed far better than AgZ with its 2000 mg Ag/L release concentration.

Table 4.5. Reduced Silver Mordenite TCLP Results

Underlying Hazardous Constituents	Concentration, mg/L							
	Universal Treatment Standard	Ag°Z ^(c) #1		Ag°Z ^(c) #2		Ag°Z ^(c) #3		
		TCLP Extract Concentration	TCLP Extract Concentration	Reporting Limit	Method Detection Limit	TCLP Extract Concentration	Reporting Limit	Method Detection Limit
Silver ^(a)	0.14	2.9 J	3.0 J	0.05	0.008	3.0 J	0.05	0.008
Arsenic	5.0	0.017	0.012	0.75	0.004	0.0072	0.75	0.004
Barium	21	0.065	0.038	0.5	0.003	0.051	0.5	0.003
Beryllium	1.22	0.0035	0.0031	0.125	0.0005	0.003	0.125	0.0005
Cadmium	0.11	<0.0005	<0.0005	0.125	0.0005	<0.0005	0.125	0.0005
Chromium	0.60	0.0058	<0.005	0.25	0.005	<0.005	0.25	0.005
Nickel	11	<0.03	<0.03	1	0.03	<0.03	1	0.03
Lead	0.75	0.055 J	0.057 J	0.25	0.003	0.059 J	0.25	0.003
Antimony	1.15	<0.009	<0.009	0.25	0.009	<0.009	0.25	0.009
Selenium	5.7	0.0082	0.01	0.125	0.007	0.0072	0.125	0.007
Thallium	0.20	<0.005	<0.005	0.5	0.005	<0.005	0.5	0.005
Vanadium ^(b)	1.6	<0.02	<0.02	1.25	0.02	<0.02	1.25	0.02
Zinc ^(a)	4.3	0.057 UJ	0.051 UJ	0.5	0.02	0.054 UJ	0.5	0.02
Mercury ^(a)	0.025	<0.00006 UJ	<0.00006 UJ	0.001	0.00006	<0.0003 UJ	0.01	0.0003

(a) Matrix spike recoveries were 136% for one silver spike and 74% for mercury.
(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.
(c) Data reported between the reporting limit and the method detection limit are estimated.
UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.
J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

4.6 Grouted Reduced Silver Mordenite

Grouting Ag°Z improved the performance of the waste disposal form with respect to silver release compared to Ag°Z by itself. Comparing the silver-release from Ag°Z found in Table 4.5 with that from grouted Ag°Z in Table 4.6 shows a reduction in silver concentration from 3 to 0.3 mg Ag/L. Table 4.6 also shows that all UHCs except silver were below the UTS limits.

The silver release level of 0.3 mg Ag/L was below the Toxicity Characteristic designation level of 5.0 mg Ag/L. The silver concentration for each replicate sample was a factor of 2 over the UTS limit for treated wastes.

Table 4.6. Grouted Reduced Silver Mordenite TCLP Results

Underlying Hazardous Constituents	Concentration, mg/L					
	Universal Treatment Standard	Grouted Ag ^o Z ^(b) #1	Grouted Ag ^o Z ^(b) #2	Grouted Ag ^o Z ^(b) #3	Reporting Limit	Method Detection Limit
		TCLP Extract Concentration	TCLP Extract Concentration	TCLP Extract Concentration		
Silver	0.14	0.28	0.38	0.17	0.05	0.008
Arsenic	5.0	<0.004	<0.004	<0.004	0.75	0.004
Barium	21	0.45	0.56	0.56	0.5	0.003
Beryllium	1.22	<0.0005	<0.0005	<0.0005	0.125	0.0005
Cadmium	0.11	<0.0005	<0.0005	<0.0005	0.125	0.0005
Chromium	0.60	0.0085	0.0086	0.007	0.25	0.005
Nickel	11	0.045	0.05	0.051	1	0.03
Lead	0.75	0.0033 UJ	0.003 UJ	<0.003 UJ	0.25	0.003
Antimony	1.15	<0.009	<0.009	<0.009	0.25	0.009
Selenium	5.7	0.0079	0.0091	0.011	0.125	0.007
Thallium	0.20	<0.005	<0.005	<0.005	0.5	0.005
Vanadium ^(a)	1.6	<0.02	<0.02	<0.02	1.25	0.02
Zinc ^(a)	4.3	<0.02	<0.02	<0.02	0.5	0.02
Mercury	0.025	<0.00006	<0.00006	<0.00006	0.001	0.00006

(a) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.

(b) Data reported between the reporting limit and the method detection limit are estimated.

UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.

4.7 Silver Mordenite Loaded with Fluoride

The HF-treated AgZ was expected to perform the worst of any halogen-loaded silver mordenite with respect to silver release during TCLP testing because AgF is the most soluble halide, 14.3 M at 15.5°C (Weast 1984). As expected, the silver concentration was quite high with releases ranging from 250 to 1830 mg Ag/L; see

Table 4.7. Two of the three replicates released near the 2200 mg Ag/L concentration that was released by AgZ.

The matrix spike recoveries for silver and mercury for AgZF were outside of the control limits. The extract from AgZF#1 was used as the matrix. Spike levels were 2.5 mg Ag/L and 0.025 mg/L for mercury. The silver spike level was <1% of the measured silver concentration and so is within the analytical error for the method. Because the silver level is far above the regulatory criteria, the recovery has no impact on conclusions regarding the regulatory status of the material. Measured mercury levels are well below regulatory criteria, and thus the matrix recovery should have no impact on assessments regarding disposal.

All the UHCs except silver are below the Washington State and Federal regulatory limits. Silver is well above the 5.0 mg Ag/L limit for the untreated waste Toxicity Characteristic limit and above the UTS limit of 0.14 mg Ag/L.

Table 4.7. Silver Mordenite Loaded with Fluoride TCLP Results

Underlying Hazardous Constituents	Concentration, mg/L					
	Universal Treatment Standard	AgZF ^(c) #1	AgZF ^(c) #2	AgZF ^(c) #3	Reporting Limit	Method Detection Limit
		TCLP Extract Concentration	TCLP Extract Concentration	TCLP Extract Concentration		
Silver ^(a)	0.14	248	1740	1830	5	0.008
Arsenic	5.0	0.016	<0.004	<0.004	0.75	0.004
Barium	21	0.043	0.046	0.018	0.5	0.003
Beryllium	1.22	0.033	0.027	0.026	0.125	0.0005
Cadmium	0.11	<0.0005	<0.0005	<0.0005	0.125	0.0005
Chromium	0.60	0.091	0.049	0.046	0.25	0.005
Nickel	11	0.046	<0.03	<0.03	1	0.03
Lead	0.75	0.019 UJ	0.0067 UJ	0.0056 UJ	0.25	0.003
Antimony	1.15	<0.009	<0.009	<0.009	0.25	0.009
Selenium	5.7	0.015	<0.007	0.0082	0.125	0.007
Thallium	0.20	<0.005	<0.005	<0.005	0.5	0.005
Vanadium ^(b)	1.6	0.074	<0.02	<0.02	1.25	0.02
Zinc ^(b)	4.3	0.23	0.043	0.025	0.5	0.02
Mercury ^(a)	0.025	<0.00006 UJ	<0.00006 UJ	<0.00006 UJ	0.001	0.00006

(a) Matrix spike recoveries for silver at 2.5 mg/L and mercury at 0.025 mg/L less than 75%. Silver spike was <1% of silver content in matrix.

(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.

(c) Data reported between the reporting limit and the method detection limit are estimated.

UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.

4.8 Grouted Silver Mordenite Loaded with Fluoride

As with grouted AgZ, measured TCLP releases of the UHCs with the exception of silver fell below the Washington State and Federal regulatory limits; compare Table 4.8 with Table 2.1. In Table 4.8, we grouped Samples 1 and 3 together with their reporting and minimum detection limits and Sample 2 grouped with its reporting limit and MDL. TCLP silver release concentrations ranged from 0.51 to 523 mg Ag/L with two of the samples at 0.51 and 0.76 mg Ag/L. Thus in two cases, the silver release was less than the Toxicity Characteristic limit for untreated waste. All three silver concentrations exceeded the UTS limit of 0.14 mg Ag/L.

Although the 523-mg Ag/L result is inconsistent with the other results, we have no basis to discard any individual analytical result. For the grouted AgZF, the assessment of the material’s performance with respect to regulatory requirements will be unaffected by any of the three results since all exceed the treated waste limit of 0.14 mg Ag/L extract. The two lower results do suggest that it may be possible to reduce the silver release by optimizing the waste form.

The matrix silver spike recovery at 48% was outside control limits; the sample used for the matrix spike was grouted AgZF #1. Because the measured silver levels are above the 0.14-mg Ag/L UTS UHC criteria, the poor silver spike recovery should have no impact on evaluating the results with respect to disposal criteria.

It is interesting that the constituents released from Grouted AgZF#2 were typically higher than from its two replicate siblings. In fact, Grouted AgZF#2 is also the only sample out of all samples tested that shows a mercury concentration at or above the reportable limit. We have no explanation for the typically higher measured levels in Grouted AgZF #2. The analysts were confident in the measurements, and we could not identify any differences between grout-sample preparations.

4.9 Iodine-Loaded Ag^oZ

The TCLP releases of the UHCs for all but silver from the three Ag^oZI replicates fell below the Toxicity Characteristic limits as shown in

Table 4.9. Silver releases were nominally 120 mg Ag/L and significantly above the Toxicity Characteristic limit. As would be expected based on the relative solubilities of AgI and AgF, the silver release from Ag°ZI was significantly less than that from AgZF, 120 vs. 1270 mg Ag/L.

4.10 Grouted Reduced Silver Mordenite Loaded with Iodine

Grouting Ag°ZI reduced the TCLP silver release to less than both Washington State and EPA regulatory levels. The silver concentrations for two of the replicates were below the MDL of 0.0012 mg Ag/L, and the third was 0.013 mg Ag/L. All of the results were below the UTS TCLP release concentration of 0.14 mg Ag/L that is the most restrictive of the three regulatory standards provided in

Table 4.10. As with the other tested materials, the concentrations of the other UHCs were below all regulatory designation limits.

Table 4.8. Grouted Silver Mordenite Loaded with Fluoride

Underlying Hazardous Constituents	Concentration, mg/L							
	Universal Treatment Standard	Grouted AgZF ^(c) #1	Grouted AgZF ^(c) #3			Grouted AgZF ^(c) #2		
		TCLP Extract Concentration	TCLP Extract Concentration	Reporting Limit	Method Detection Limit	TCLP Extract Concentration	Reporting Limit	Method Detection Limit
Silver ^(a)	0.14	0.51 J	0.76 J	0.05	0.008	523	1.3	0.008
Arsenic	5.0	<0.004	<0.004	0.75	0.004	0.011	0.75	0.004
Barium	21	0.4	0.33	0.5	0.002	1.7	0.5	0.003
Beryllium	1.22	<0.0005	<0.0005	0.13	0.0005	0.0015	0.13	0.0005
Cadmium	0.11	<0.0005	<0.0005	0.13	0.0005	<0.0005	0.13	0.0005
Chromium	0.60	0.0088	0.01	0.25	0.005	0.12 J	0.25	0.005
Nickel	11	<0.03	<0.03	1	0.025	0.13	1	0.03
Lead	0.75	<0.003	<0.003	0.25	0.002	0.029 J	0.25	0.003
Antimony	1.15	<0.009	<0.009	0.25	0.009	<0.009	0.25	0.009
Selenium	5.7	0.0073	0.012	0.13	0.007	0.083	0.13	0.007
Thallium	0.20	<0.005	<0.005	0.5	0.005	<0.005 UJ	0.5	0.005
Vanadium ^(b)	1.6	<0.02	<0.02	1.3	0.01	<0.02 UJ	1.3	0.02
Zinc ^(b)	4.3	<0.02	<0.02	0.5	0.011	<0.02 J	0.5	0.02
Mercury	0.025	<0.00006	<0.00006	0.001	0.00005	0.001	0.001	0.00006

(a) Matrix silver spike recovery was 48%.

(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.

(c) Data reported between the reporting limit and the method detection limit are estimated.

UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.

J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

Table 4.9. Iodine Loaded Reduced Silver Mordenite TCLP Results

Underlying Hazardous Constituents	Concentration, mg/L					Reporting Limit	Method Detection Limit
	Universal Treatment Standard	Ag°ZI ^(c) #1 TCLP Extract Concentration	Ag°ZI ^(c) #2 TCLP Extract Concentration	Ag°ZI ^(c) #3 TCLP Extract Concentration			
Silver ^(a)	0.14	119 J	126 J	126 J		5	0.03
Arsenic	5.0	<0.03 UJ	<0.03 UJ	<0.03 UJ		7.5	0.03
Barium	21	<0.2 UJ	<0.2 UJ	<0.2 UJ		5	0.2
Beryllium	1.22	<0.006 UJ	<0.006 UJ	<0.006 UJ		1.3	0.006
Cadmium	0.11	<0.005 UJ	0.009 J	0.0088 J		1.3	0.005
Chromium	0.60	<0.009 UJ	<0.009 UJ	0.028 J		2.5	0.009
Nickel	11	0.046 J	0.044 J	0.047 J		10	0.04
Lead	0.75	0.07 UJ	0.045 UJ	0.054 UJ		2.5	0.04
Antimony	1.15	<0.07 UJ	<0.07 UJ	<0.07 UJ		2.5	0.07
Selenium	5.7	<0.03 UJ	<0.03 UJ	<0.03 UJ		1.3	0.03
Thallium	0.20	<0.08 UJ	<0.08 J	<0.08 UJ		5	0.08
Vanadium ^(b)	1.6	<0.04 UJ	<0.04 UJ	<0.04 UJ		12.5	0.04
Zinc ^(b)	4.3	0.23 UJ	0.12 UJ	0.11 UJ		5	0.06
Mercury	0.025	0.0005	<0.00002	<0.00002		0.001	0.00002

(a) Matrix silver concentration >4× spike level.
(b) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.
(c) Data reported between the reporting limit and the method detection limit are estimated.
UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.
J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

Table 4.10. Grouted Reduced Silver Mordenite Loaded with Iodine TCLP Results

Underlying Hazardous Constituents	Concentration, mg/L			Reporting Limit	Method Detection Limit	
	Universal Treatment Standard	Grouted Ag°ZI ^(a) #1 TCLP Extract Concentration	Grouted Ag°ZI ^(a) #2 TCLP Extract Concentration			Grouted Ag°ZI ^(a) #3 TCLP Extract Concentration
Silver	0.14	0.013 J	<0.002 UJ	<0.002 UJ	0.25	0.002
Arsenic	5.0	0.0034 J	0.0058 J	0.0041 J	0.75	0.003
Barium	21	0.7 J	0.68 J	0.73 J	0.5	0.02
Beryllium	1.22	<0.0005 UJ	<0.0005 UJ	0.0008 J	0.13	0.0005
Cadmium	0.11	0.002 J	0.0015 J	0.0032 J	0.13	0.0005
Chromium	0.60	0.017 J	0.016 J	0.018 J	0.25	0.0009
Nickel	11	0.025 J	0.026 J	0.029 J	1	0.004
Lead	0.75	0.0089 UJ	<0.004 UJ	<0.004 UJ	0.25	0.004
Antimony	1.15	0.0084 J	<0.007 UJ	<0.007 UJ	0.25	0.007
Selenium	5.7	0.0094 UJ	0.0053 UJ	0.0094 UJ	0.13	0.003
Thallium	0.20	<0.008 UJ	<0.008 UJ	<0.008 UJ	0.5	0.008
Vanadium	1.6	0.0054 J	<0.004 UJ	0.0063 J	1.3	0.004
Zinc	4.3	0.012 UJ	0.01 UJ	0.0094 UJ	0.5	0.006
Mercury	0.025	<0.00002	<0.00002	<0.00002	0.001	0.00002

(a) Data reported between the reporting limit and the method detection limit are estimated.
 UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.
 J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

5.0 Conclusions and Recommendations

In support of BNI's efforts to identify a disposal form for spent silver mordenite arising from radioiodine control operations in the planned Hanford WTP, PNWD evaluated several reference and potential disposal forms. The tested materials were as-purchased silver mordenite, hydrogen-reduced silver mordenite, iodine and fluoride-loaded silver mordenites, and their grouted forms at a 25 wt% loading. This testing found that the tested forms will not release Washington State and EPA hazardous or dangerous constituents above UTS limits, with the exception of silver. Silver releases varied from material to material with all but two silver mordenite-containing materials exceeding the UTS silver release limits.

As illustrated in Table 5.1 and Figure 5.1, grouting with the addition of a grout-compatible soluble iodide such as CaI_2 will reduce the TCLP silver release from the worst-case AgZ to below EPA UTS and Washington State Dangerous waste designation levels. The TCLP silver release from AgZ was 2200 mg Ag/L. Simple grouting reduced the release of the disposal form to 0.7 mg Ag/L. Adding 10 wt% CaI_2 to grouted AgZ further reduced the silver release to <0.008 mg Ag/L.

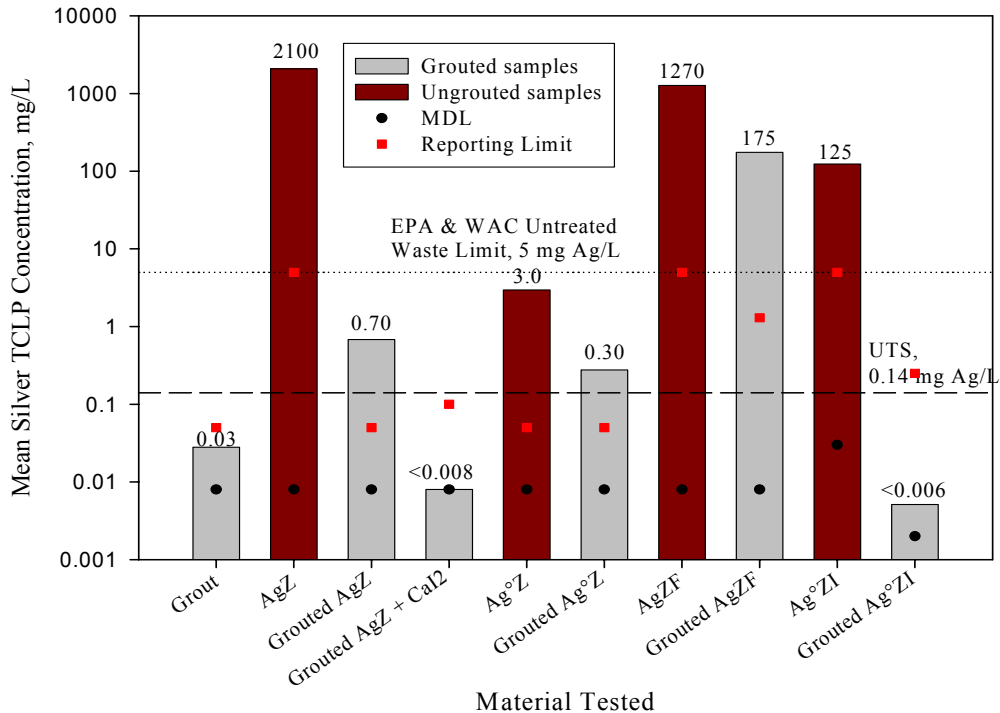


Figure 5.1. Mean Silver TCLP Releases from Tested Reference and Candidate Spent Silver Mordenite Disposal Forms

The TCLP results for the grouted Ag^oZI offer promise with its silver release of <0.005 mg Ag/L, which is less than UTS. This suggests that when grouted Ag^oZ is exposed to halogens that react with silver to form insoluble halides such as AgCl and AgI, it will produce a spent-silver-mordenite disposal form that will release silver at levels below UTS levels. Testing chlorine treated Ag^oZ is needed to validate this hypothesis. It would also be valuable to validate the thermodynamic calculations that indicate that HF will not react with the metallic silver in Ag^oZ with TCLP testing of HF-treated Ag^oZ; the HF-loading behavior indicates that HF is sorbed by AgZ as predicted by thermodynamics.

Table 5.1. Average TCLP releases of Underlying Hazardous Constituents. See 40 CFR 261 (2002) and 40 CFR 268 (2002) for Federal hazardous waste limits and WAC 173-303-090 (WAC 2000a) and WAC 173-303-140 (WAC 2000c) for Washington State dangerous waste limits.

Underlying Hazardous Constituents	Mean TCLP Release Concentration, mg/L ^(b)										Regulatory TCLP Designation Limits, mg/L	
	AgZ	Grout	Grouted AgZ	Grouted AgZ + CaI2	Ag°Z	Grouted Ag°Z	Grouted AgZF	AgZF	Ag°ZI	Grouted Ag°ZI	EPA Hazardous & Washington State Dangerous Waste	EPA Universal Treatment Standard
Silver	2090	<0.028 ^{J,UJ}	0.69 ^J	<0.008 ^{UJ}	3.0 ^J	0.28	175 ^J	1280	124 ^J	<0.006 ^{J,UJ}	5.0	0.14
Arsenic	<0.004	<0.004	<0.0054	<0.004	0.013 ^B	<0.004	<0.0064	<0.008	<0.03 ^{UJ}	0.005 ^J	5.0	5.0
Barium	0.028 ^B	0.71	0.35 ^B	0.56	0.052 ^B	0.52	0.81	0.036 ^B	<0.2 ^{UJ}	0.71 ^J	100.0	21
Beryllium	0.0019 ^B	<0.0009	<0.0012	<0.0005	0.0032 ^B	<0.0005	<0.00084	0.029 ^B	<0.006 ^{UJ}	<0.0006 ^{J,UJ}	None	1.22
Cadmium	<0.0005	<0.0005	<0.0013	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.008 ^{J,UJ}	0.0023 ^J	1.0	0.11
Chromium	<0.005	0.013 ^B	0.017 ^B	0.0082 ^B	<0.0053	0.0081 ^B	0.047 ^{J,B}	0.062 ^B	<0.02 ^{J,UJ}	0.017 ^J	5.0	0.60
Nickel	<0.03	0.084 ^B	<0.03	<0.03	<0.03	0.049 ^B	<0.07	<0.04	0.046 ^{J,B}	0.027 ^J	None	11
Lead	0.01 ^B	<0.004	<0.003	0.0043 ^B	0.057 ^{J,B}	<0.0031 ^{UJ}	<0.012 ^J	0.011 ^{UJ,B}	0.057 ^{UJ,B}	<0.006 ^{UJ}	5.0	0.75
Antimony	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.07 ^{UJ}	<0.0075 ^{J,UJ}	None	1.15
Selenium	<0.011	0.010 ^B	0.013 ^B	0.015 ^B	0.0085 ^B	0.0094 ^B	0.035 ^B	<0.011	<0.03 ^{UJ}	0.0081 ^{UJ}	1.0	5.7
Thallium	<0.01	<0.011	<0.005	<0.005	<0.005	<0.005	<0.005 ^{UJ}	<0.005	<0.08 ^{J,UJ}	<0.008 ^{UJ}	None	0.20
Vanadium ^(a)	<0.02	<0.02 ^{UJ}	<0.02	<0.02	<0.02	<0.02	<0.02 ^{UJ}	<0.04	<0.04 ^{UJ,J}	<0.006 ^{J,UJ}	None	1.6
Zinc ^(a)	0.06 ^{UJ,B}	<0.02 ^{UJ}	<0.02 ^{UJ}	<0.02 ^{UJ}	0.054 ^{UJ,B}	<0.02	<0.02 ^J	0.01 ^B	0.16 ^{UJ,B}	0.011 ^{UJ}	None	4.3
Mercury	<0.0003 ^{UR}	<0.0003 ^{UJ}	<0.0001 ^{UJ}	<0.0001 ^{UJ}	<0.0002 ^{UJ}	<0.0002	<0.001	<0.0001 ^{UJ}	<0.0002 ^{UJ}	<0.00002	0.2	0.025

(c) Vanadium and zinc are not UHCs for wastes exhibiting the silver toxicity characteristic. The results are presented for information only.

(d) < values indicate at least one result was less than the MDL and the mean is estimated using the MDL.

U – At least one result in the mean was analyzed for but was not detected. The data should be considered usable for decision-making purposes.

UJ - At least one result in the mean was analyzed for and was not detected. Due to a QC deficiency identified during validation, the value reported may not accurately reflect the minimum detectable activity. The data should be considered usable for decision-making purposes.

J - At least one result in the mean was analyzed for and detected. The associated value is estimated due to a QC deficiency identified during data validation. The data should be considered usable for decision-making purposes.

UR - At least one result in the mean was analyzed for and not detected; however, due to an identified QC deficiency, the data should be considered unusable for decision-making purposes.

B – At least one result in the mean was an estimated value less than the reporting limit and greater than the method detection limit.

The best performing of the untreated wastes was unexposed Ag°Z. The silver release during TCLP testing was 0.28 mg Ag/L, which is below the EPA's and Washington State's untreated waste standard of 5.0 mg Ag/L TCLP extract but above the UTS level of 0.14 mg Ag/L TCLP extract. Although the Ag°Z is below the untreated waste standard, it is not fully representative of spent Ag°Z exposed to halogens, and based on our studies, it represents a best-case model for spent halogen-exposed Ag°Z. Its performance likely can be explained by the low solubility of metallic silver.

The observed behavior of AgZF and grouted AgZF is consistent with our expectations that the HF-treated AgZ should represent a worst case for the halogen-treated silver mordenites because of the high solubility of AgF (14 M). AgZF performed better than AgZ but when grouted released more silver (175 mg Ag/L) than grouted AgZ (0.7 mg Ag/L). This material could be used as representative of the worst-case basis for spent halogen-treated silver mordenite in studies to optimize the disposal form for spent Ag°Z.

As expected, Ag°ZI released less silver than AgZF; however, the 125 mg Ag/L TCLP extract was much higher than the untreated waste Toxicity Characteristic of 5.0 mg Ag/L. The AgZF released an average 1300 mg Ag/L during TCLP. The Ag°ZI could, in waste-form optimization studies, be used to represent the best-case basis for waste-form optimization.

Testing HF- and chlorine-treated Ag°Z disposal forms would complete disposal studies with respect to all the potential behaviors of halogen effects on spent Ag°Z and should provide a basis for choosing a disposal form without any added CaI₂. Assuming Burger's predicted HWVP MOG composition (Burger and Scheele 1991), chlorine should be the predominant halogen in the MOG (Cl:I = 10⁵) that will react with the metallic silver in Ag°Z. Thus, the chlorine-loaded Ag°Z should be most representative of the spent Ag°Z. The HF-treated Ag°Z would complete the study of halogen effects.

The testing of Ag°Z exposed to a fully representative simulated MOG would provide the most representative material for waste-form performance and optimization studies and confirmation of other bounding materials testing. The current and possible future testing of HF treated Ag°Z, chlorine-loaded Ag°Z, and Ag°ZI would provide bounding behaviors. The one unknown complication is the effect of NO_x.

Adding CaI₂ to grouted silver mordenite to limit silver release provides another attractive strategy for assuring a regulatory-compliant spent-silver mordenite disposal form. Further studies of CaI₂ concentration levels may be valuable and are suggested for waste-form optimization studies.

Most studies of NO_x effects on AgZ and Ag°Z have been focused on their effects on iodine trapping and have been fairly short term because iodine concentrations were higher than expected in a DOG. This may not be important because the chlorine concentrations in the MOG will consume available silver and could reduce exposure time to NO_x times already studied. We recommend comparing expected use times to those already studied.

Because this study was not designed to develop the optimum spent-silver-mordenite disposal form, we recommend further studies to optimize disposal-form compositions and thus performance with respect to regulatory levels. These studies should investigate the effects of CaI₂ concentration, waste loading, water content, and preparation procedure, e.g., order of mixing, including when to add water.

6.0 References

- 40 CFR 61. 2002. "National Emission Standards for Hazardous Air Pollutants." *U.S. Code of Federal Regulations*. U.S. Environmental Protection Agency,
- 40 CFR 261. 2002. "Identification and Listing of Hazardous Waste." *U.S. Code of Federal Regulations*. U.S. Environmental Protection Agency.
- 40 CFR 268. 2002 "Land Disposal Restrictions." *U.S. Code of Federal Regulations*. U.S. Environmental Protection Agency.
- Barin I. 1989. *Thermochemical Data of Pure Substances*, VCH Publishers, New York.
- Bechtel National, Inc. (BNI). 2001. *Stabilization of Spent Silver Mordenite Disposal* 24590-WTP-TSP-RT-01-013, Richland, Washington.
- Burger LL, RD Scheele, and KD Wiemers. 1981. *Selection of a Form for Fixation of Iodine 129*, PNL-4045, Pacific Northwest Laboratory, Richland, Washington.
- Burger LL, and RD Scheele. 1983. *The Status of Radioiodine Control for Nuclear Fuel Reprocessing Plants*, PNL-4689, Pacific Northwest Laboratory, Richland, Washington.
- Burger LL and RD Scheele. 1991. *HWVP Iodine-Trap Evaluation*, PNL 7581, Pacific Northwest Laboratory, Richland, Washington.
- Dean JA. 1973. *Lange's Handbook of Chemistry*, McGraw Hill, New York
- U.S. Department of Energy (DOE). 1993. *Radiation Protection of the Public and the Environment*, DOE 5400.5, Washington, D.C.
- Kolthoff IM, EB Sandell, EJ Meehan, and S Bruckenstein. 1969. *Quantitative Chemical Analysis*, The Macmillan Company, New York.
- Scheele RD, LL Burger, and CL Matsuzaki. 1983. *Methyl Iodide Sorption by Reduced Silver Mordenite*. PNL-4489, Pacific Northwest Laboratory, Richland, Washington.
- Scheele RD, LL Burger, and JK Soldat. 1984. *The Adequacy of Radioiodine Control and Monitoring at Nuclear Fuels Reprocessing Plants*, PNL-5108, Pacific Northwest Laboratory, Richland, Washington.
- Scheele RD, LL Burger, and BT Halko. 1988. *Comparison of Silver Sorbents for Application to Radioiodine Control at the PUREX Process Facility Modification*, PNL-6607, Pacific Northwest Laboratory, Richland, Washington.
- Scheele RD and LL Burger. 1987. *Evaluation of Silver Mordenite for Radioiodine Retention at the PUREX Process Facility Modification*, PNL-6261, Pacific Northwest Laboratory, Richland, Washington.

Thomas TR, LP Murphy, BA Staples, and JT Nichols. 1977. *Airborne Elemental Iodine-Loading Capacities of Metal Zeolites and a Method for Recycling Silver Zeolite*, ICP-1119, Idaho National Engineering Laboratory, Idaho Falls, Idaho.

U.S. Environmental Protection Agency (EPA). 2001. *Test Methods for Evaluating Solid Waste – Physical/Chemical Methods, SW-846, Third Edition*. Available URL: <http://www.epa.gov/epaoswer/hazwaste/test/main.htm>

Washington State Administrative Code (WAC). 2000a. WAC 173-303-090, Dangerous Waste Characteristics. Available URL: <http://www.leg.wa.gov/wac/>

Washington State Administrative Code (WAC). 2000b. WAC 173-303-110, Sampling and Testing Methods. Available URL: <http://www.leg.wa.gov/wac/>

Washington State Administrative Code (WAC). 2000c. WAC 173-303-140, Land Disposal Restrictions. Available URL: <http://www.leg.wa.gov/wac/>

Washington State Administrative Code (WAC). 1998. WAC 246-247, Radiation Protection – Air Emissions. Available URL: <http://www.leg.wa.gov/wac/>

Weast RC. 1984. *Handbook of Chemistry and Physics*. Chemical Rubber Company, Cleveland, Ohio.

Appendix A

Toxic Characterization Leach Procedure Laboratory Accreditation

The State of
Department



Washington
of Ecology

This is to certify that

**STL St. Louis
Earth City, MO**

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective August 31, 2001, and shall expire August 30, 2002.

Witnessed under my hand on October 9, 2001.

Perry F. Brake, Chemist
Lab Accreditation Unit Supervisor

Lab Accreditation Number
C116

Scope of Accreditation

STL St. Louis

Earth City, MO

is accredited by the State of Washington Department of Ecology to perform analyses for the parameters listed below using the analytical methods indicated. This Scope of Accreditation applies to non-potable water analyses only. Accreditation for all parameters is final unless indicated otherwise in a note. Accreditation is for the latest version of a method unless otherwise specified in a note. EPA refers to the U.S. Environmental Protection Agency. SM refers to American Public Health Association's publication, Standard Methods for the Examination of Water and Wastewater, 19th edition, unless otherwise noted. ASTM stands for the American Society of Testing and Materials. PSEP stands for Puget Sound Estuary Program. Other references are detailed in the notes section.

Parameter Name	Reference	Method Number	Notes
Alkalinity, Total	EPA	310.1	
Ammonia	EPA	350.1	2
Biochemical Oxygen Demand, BOD/CBOD	EPA	405.1	2
Bromide	EPA	300.0	
Calcium	EPA	200.7/6010	
Chemical Oxygen Demand (COD)	EPA	410.4(7.3)	
Chloride	EPA	300.0	
Cyanide, Total	EPA	335.2(8.10)	
Fluoride	EPA	300.0	
Hardness, Total (as CaCO ₃)	EPA	130.2	2
Magnesium	EPA	200.7/6010	
Nitrate	EPA	353.1	
Nitrate + Nitrite	EPA	353.1	
Nitrite	EPA	353.1	
Oil & Grease	EPA	413.1	
Orthophosphate	EPA	365.1	
Phenolics, Total Recoverable	EPA	420.2	
Phosphorus, Total	EPA	365.1	
Potassium	EPA	200.7/6010	
Silica	EPA	200.7	
Silica	EPA	6010	
Sodium	EPA	200.7/6010	
Solids, Total Dissolved	EPA	160.1	
Solids, Total Suspended	EPA	160.2	
Specific Conductance	EPA	120.1	2
Sulfide	EPA	376.1	

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Parameter Name	Reference	Method Number	Notes
Total Organic Carbon	EPA	415.1	2
Total Organic Halides	EPA	450.1	
Turbidity	EPA	180.1	
Aluminum	EPA	200.7/6010	
Antimony	EPA	200.7/6010	
Arsenic	EPA	206.2/7060	
Barium	EPA	200.7/6010	
Beryllium	EPA	200.7/6010	
Cadmium	EPA	200.7/6010	
Chromium	EPA	200.7/6010	
Cobalt	EPA	200.7/6010	
Copper	EPA	200.7/6010	
Iron	EPA	200.7/6010	
Lead	EPA	200.7/6010	
Manganese	EPA	200.7/6010	
Mercury	EPA	245.1/7470	
Molybdenum	EPA	200.7/6010	
Nickel	EPA	200.7/6010	
Selenium	EPA	270.2/7740	
Silver	EPA	200.7/6010	
Thallium	EPA	279.2/7841	
Tin	EPA	200.7	
Tin	EPA	6010	1
Vanadium	EPA	200.7/6010	
Zinc	EPA	200.7/6010	
Chlorinated Herbicides	EPA	8151	
Organochlorine Pesticides	EPA	8081	
Polychlorinated Biphenyls	EPA	608/8082	
Polycyclic Aromatic HC (HPLC)	EPA	610/8310	
BNA Extr (Semivolatile) Organics	EPA	625/8270	
Volatile Organic Compounds	EPA	624/8260	
Alpha, Gross	EPA	900.0	
Beta, Gross	EPA	900.0	
Cesium-134/Cesium-137	EPA	901.0	
Gamma	HASL	300	
Radium-226	EPA	903.1	
Radium-228	EPA	904.0	

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Parameter Name	Reference	Method Number	Notes
Strontium-89/90	EPA	905.0	
Tritium	EPA	906.0	
Uranium	ASTM	D5174-91	

Accredited Parameter Note Detail

(1) Method modified to ensure digestion and quantification of metal which is not included in EPA method. (2) Provisional pending submission of acceptable performance evaluation sample analysis results (WAC 173-50-110).

 11/1/01

Authentication Signature

Perry Brake -- Unit Supervisor, Washington State Department of Ecology -- Lab Accreditation Unit

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