**Removal of Contaminants from Equipment and Debris
and Waste Minimization Using the TECHXTRACT® Technology**

Final Report - Base Phase

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ABSTRACT

From September, 1996 through July, 1997, EET, Inc. conducted a series of experiments under a U.S. Department of Energy (DOE) Program Research and Development Agreement (PRDA). This project, entitled "Removal of Contaminants from Equipment and Debris and Waste Minimization Using the TECHXTRACT[®] Technology" was conducted under DOE Contract DE-AC21-96MC33138, administered by the Federal Energy Technology Center.

The contract is divided into two phases - a base phase during which bench scale testing was conducted; and an optional phase for a field demonstration of a full-scale system. This report documents the results from the base phase of the contract.

The base phase included the following major elements:

- Evaluation of the effectiveness of various decontamination options, using both surrogate and radioactively contaminated samples.
- Evaluation of various methods for the treatment of the secondary waste streams from the preferred decontamination system(s).
- Evaluation of decontamination effectiveness for concrete rubble.
- Preliminary engineering design and cost estimation for a full-scale system.
- Preliminary economic analysis of the proposed system versus other currently available options for disposition of the materials.

Results from the base phase, which are described in the following report, are very positive. Testing has shown that free release requirements and extremely high decontamination factors can be achieved for a variety of materials and radionuclides. Results for concrete rubble decontamination were less conclusive. The bench scale testing has led to the design of two different systems, both based on the TECHXTRACT[®] chemistry, for potential full-scale demonstration. Based on the preliminary economic analysis, this system compares favorably with currently available commercial options, including disposal.

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EXECUTIVE SUMMARY

The U.S. Department of Energy (DOE) has identified a need for “new separation methods ... for leaching material from surfaces [and] recovering and concentrating radioactive and toxic materials removed in decontamination.” This report documents the results of work performed by EET, Inc., under DOE Contract DE-AC21-96MC33138, entitled “Removal of Contaminants from Equipment and Debris and Waste Minimization Using the TECHXTRACT® Technology.” The objective of this Program Research and Development Agreement (PRDA) contract was to evaluate certain systems for the decontamination of scrap metal and debris and for the treatment and minimization of the resultant secondary waste. The evaluations were to include performance measurements, optimization recommendations, and economic analyses.

Evaluation and optimization of the decontamination system was the primary focus of the contract. EET’s research included work with surrogate and radioactively contaminated samples. SIMCON 2 coupons obtained from Idaho National Engineering Laboratory (INEL) served as surrogates for contaminated metal surfaces. These samples use non-radioactive cesium (Cs) and zirconium (Zr) adhered to 1” stainless steel coupons as surrogate contaminants. Concrete rubble samples were prepared by EET using several different metals as surrogates. Decontamination trains developed for SIMCON coupons and simulated concrete rubble under laboratory conditions were tested in field demonstrations at Alaron Corp., Wampum, PA. using radioactive samples. The samples used for testing at Alaron were taken from actual materials which were awaiting decontamination or disposal, and represented many different substrates and radionuclides.

Decontamination experiments included the use of three proprietary chemical blends from EET - TECHXTRACT® 0100, 0200, and 0300. In previous decontamination projects performed by EET (not involving chemical baths), these three blends have normally been used in a sequential application to maximize their synergistic effects and to address a wide range of contaminants. In addition to the TECHXTRACT® blends, other non-proprietary, common chemistries and water were evaluated in the base phase. Experiments also evaluated the effect of chemical concentration, chemical additives, temperature, and batch time on decontamination performance.

These experiments identified two different treatment trains as having the best performance in the removal of Cs and Zr surface contamination from SIMCON 2 coupons. Removal efficiencies of up to ca. 99%, corresponding to decontamination factor (DF) values approaching 100, were achieved. The first decontamination train (Train I) is based on a diluted and enhanced TECHXTRACT® 0300 solution in a heated ultrasonic bath. The second train (Train II) incorporates sequential treatment in diluted and enhanced solutions of all three TECHXTRACT® blends. Train II is more complicated and costly, and is only expected to be applicable in cases where surface contamination is much more tenacious than that deposited on SIMCON 2 coupons.

In bench scale testing using radioactively contaminated samples, Train I processing successfully decontaminated a wide variety of substrates containing 'fixed' surface contamination to free release ($<5,000$ dpm/100cm² β - γ) and even to 'background' levels. Train I processing was applied to copper tubing, titanium tubing, aluminum, carbon steel, and stainless steel tubing. Batch times as short as 7.5 min. and multiple uses of the same solution were capable of achieving free release standards. Figure I shows sequential treatment results for stainless steel tubing sections. The same solution (300 ml) cleaned over 100 cm² of tubing without any decrease in its effectiveness. Lead and stainless steel fuel rack substrates were successfully decontaminated using modified TECHXTRACT® formulations and sequences. DF values of several hundred were measured in some experiments. Many of the radioactively contaminated samples in this portion of the project were considered impossible to decontaminate using traditional methods. Despite this, every type of substrate - many representing high value scrap metals - was decontaminated to meet free release standards.

Laboratory experiments using concrete rubble impregnated with zirconium, cobalt, lanthanum, and nickel achieved DF values of over 20 for cobalt, lanthanum, and nickel, and 12 for Zr. Tests on radioactively-contaminated rubble at Alaron achieved DF values of about 2. These were much lower than the values achieved on metal samples, and were inconclusive given the limited number of available samples.

Several waste treatment strategies for removing heavy metals from the combined effluent streams were identified. Heavy metals forming insoluble metal hydroxides can be recovered from Train I effluents by co-precipitation with iron (Fe^{3+}). Co-precipitation of heavy metals with Fe^{3+} allows effective heavy metal removal in one vessel at one pH. A simulated Train I effluent stream containing 250 ppm each of non-radioactive cobalt, lanthanum, nickel, uranium, and zirconium was treated using Fe^{3+} co-precipitation at pH 9.5. The results are summarized in Table I. Residual heavy metals after filtration of the heavy metal sludge were all below 0.15 ppm. After a second Fe^{3+} co-precipitation and filtration step, the residual heavy metal concentration was below the detection level of 0.01 ppm (DF > 25,000). The metal sludge generated from the second precipitation step can be recycled and used as the Fe^{3+} source in the first precipitation. In this fashion high metal removal efficiency is obtained while minimizing the solid waste production.

Waste generated from TECHXTRACT® 0200 and 0100 effluent contains chelates and organic complexing agents. Such compounds must first be destroyed before heavy metal recovery by co-precipitation with Fe^{3+} can be successful. Testing by EET showed that successful chelant destruction can be achieved with an advanced oxidation process (AOP) photoreactor, which uses a combination of ultraviolet (UV) radiation and chemical oxidation via hydrogen peroxide (H_2O_2).

Cesium does not form an insoluble metal hydroxide and therefore must be removed by another technique. A cesium selective ion exchange resin (IONSIV®IE-911) was obtained from UOP. Cesium isotherm studies on the IONSIV resin indicated that a maximum loading of ca. 150 ppm Cs per g resin is obtained in Train I and Train II matrices.

Overall, the effluent treatment portion of the project showed several viable options. Selection of a treatment method is highly dependent on the radionuclides, the effluent composition (which depends on the decontamination train), and discharge limits. All of these are a function of site and project-specific conditions.

Simplified engineering design and capital cost estimates were developed for systems based on Train I and Train II configurations. Train II is much more expensive, due to the extra equipment for the additional chemical baths and the more complicated effluent treatment process. The estimated cost for each system is:

- Train I \$176,250
- Train II \$450,225

These costs do not include shipping or assembly into a modular system, which will add approximately 25% to the system cost.

The overall economics for the proposed system were also evaluated. The mid-point case for a Train I system, including capital and operating costs, is \$0.65 per pound of scrap metal that is decontaminated. Sensitivity cases resulted in a low value of \$0.14/lb, and a high of \$1.84/lb. The Train II system had a total estimated mid-point cost of \$1.02/lb. Overall, the economics compare favorably with existing options for radioactively-contaminated scrap metal. The cost of performing radiological surveys is included in these costs and is a major cost factor. Other key economic variables are scrap metal density, batch times, chemical consumption rates, and labor.

The results from the base phase contract show the efficacy of the TECHXTRACT® based decontamination system. Given the magnitude of DOE's Decontamination and Decommissioning (D&D) program and the value associated with recycling scrap metal, considerable benefits could be realized from the deployment of mobile, on-site units based on this technology. A field demonstration at a DOE site under the contract's option phase would allow for completion of this evaluation on a full-scale basis.

Figure I Decontamination of stainless steel heat exchanger tubing sections using TECHXTRACT® 0300 blend and sonication as a function of total surface area processed. Legend: (○) initial contamination; (●) final contamination. The free release standard for the samples was 5000 dpm/100 cm².

Table I: Residual heavy metal concentrations in treated Train I waste effluent.

Waste Train	Treatment	Metal	C _o (ppm) before treatment	C _f (ppm) after treatment	DF C _o /C _f
Train I (0300-DI)	Fe co-ppt	Co	250	0.120	2100
		Fe	2,000	0.015	133,000
		La	250	0.038	6600
		Ni	250	0.092	2700
		U	250	<0.010	>25000
		Zr	250	<0.010	>25000
Train I (0300-DI)	two times Fe co-ppt	Co	250	<0.010	>25000
		Fe	2,000	<0.010	>200,000
		La	250	<0.010	>25000
		Ni	250	<0.010	>25000
		U	250	<0.010	>25000
		Zr	250	<0.010	>25000

1.0 INTRODUCTION

This report documents the results of the base phase of Department of Energy (DOE) Contract DE-AC21-96MC33138, "Removal of Contaminants from Equipment and Debris and Waste Minimization Using the TECHXTRACT[®] Technology." This contract was awarded in September 1996 by DOE's Office of Science and Technology through the Efficient Separations and Processing (ESP) Crosscutting Area and the Federal Energy Technology Center (FETC).

The objectives of the base phase of the contract, as defined in the Management Plan, were to:

- Evaluate the effectiveness of the TECHXTRACT[®] decontamination system in meeting "free release" standards for radioactively contaminated metal.
- Evaluate the effectiveness of the same system for decontaminating concrete rubble.
- Evaluate and select a secondary waste treatment system for the spent chemicals and water from the decontamination process.
- Design an optimized system for this decontamination based on the TECHXTRACT[®] process.
- Assess the economics of the full-scale system versus disposal of the materials without decontamination.

EET's project effort was divided into eight tasks in the base phase. As shown in the work breakdown structure, these tasks were:

- 1.1 Project Kickoff
- 1.2 Decontamination Line Optimization
- 1.3 Site Selection/Decontamination Line Engineering
- 1.4 Waste Treatment Testing
- 1.5 Waste Treatment Design
- 1.6 Rubble Decontamination Testing
- 1.7 Preliminary Economics
- 1.8 Optional Field Test Plan and Baseline Report

Whenever possible, work was done concurrently on multiple tasks. This allowed for more efficient personnel utilization and a shorter project schedule.

The primary location at which the research was performed was EET's laboratory facility in Austin, TX. All testing involving non-radioactive materials was performed there, as well as the primary engineering design work. Since EET does not hold a license to handle radioactive materials, a subcontractor was used for the portions of the project involving radioactive samples. Alaron Corporation, in Wampum, PA, was selected as the subcontractor for this portion of the project. Alaron operates a full-scale, fixed base decontamination business in Wampum. A major advantage of the subcontract with Alaron was their ability to provide a wide variety of coupons from actual contaminated materials for EET's tests.

This report includes a discussion of all of the experimental procedures and results for the base phase of the contract. It also presents the recommended system design(s) and the economic analyses. This report does not include any conclusions regarding site selection for a full-scale field demonstration. EET made several inquiries regarding potential DOE sites for the optional phase of the contract. Delays in the award of the second round of Large Scale D&D Demonstration sites prevented these discussions from reaching a conclusion. The lack of a defined site also impacted the waste treatment design activities and field test plan, since both of these are site dependent. After the review of this report is complete, EET can proceed with site selection efforts if funding for the option phase is available.

The TECHXTRACT[®] technology consists of chemical composition and processes which are the property of EET, Inc. The technology is covered under U.S. patents 5,421,906 and 5,512,202, European patent application 0693977, and other pending U.S. and international patents. TECHXTRACT[®] is a registered trademark of EET, Inc.

2.0 Surface Decontamination Experiments Using Surrogates

In this section, the ability of the TECHXTRACT® blends and other solutions to decontaminate surrogate radionuclide contaminants from metal surfaces is investigated. Stainless steel SIMCON 2 coupons contaminated with a surface cesium (Cs) and zirconium (Zr) oxide layer were obtained from INEL. INEL analyzed each coupon for initial (C_o) and final (C_f) Cs and Zr contamination.

The chemical formulations developed by EET, Inc., namely TECHXTRACT® 0300, 0200, and 0100, have been successfully used in manual applications to decontaminate metal and concrete surfaces. Sequential applications of the blends are often used in heavily contaminated areas. The decontamination sequence is typically TECHXTRACT® 0300, TECHXTRACT® 0200, rinse and vacuum to remove the chemicals, TECHXTRACT® 0100, and a final rinse and vacuum. In manual applications, each chemical is scrubbed onto the contaminated surface. This process is repeated until the required decontamination levels are achieved.

The experimental program investigated the influence of temperature, chemical concentration, the number of treatment steps, mechanical stirring, and several chemical formulations including the base and enhanced versions of the TECHXTRACT® blends. The development of optimal conditions was an iterative process. In this section, only selected results are presented. Experimental results and conditions of all experiments performed are presented in Appendix A.

2.1 TECHXTRACT® Solutions

The TECHXTRACT® technology includes proprietary chemical compositions and methods owned by EET. The technology is protected under U.S. Patents 5,421,906 and 5,512,202. Other U.S. and international patents are pending.

The technology encompasses three different base chemical blends. TECHXTRACT® 0300 is a blend of inorganic acids and buffering agents and exhibits a low pH. TECHXTRACT® 0300 is typically the first blend applied in surface decontamination efforts. The chemical formulation of 0300 does not contain complexing agents that interfere with standard heavy metal removal techniques.

TECHXTRACT® 0200 is a mixture of mild organic and inorganic acids and includes small concentrations of chelates, mainly ethylene diamine tetraacetic acid (EDTA). TECHXTRACT® 0200 is typically the second step in the sequential treatment process. The chelates in the 0200 blend retain and complex solubilized metals. They therefore do interfere with standard heavy metal recovery techniques and must be destroyed before metal recovery can be successful.

TECHXTRACT® 0100 is a multi-phased, water based alkaline mixture of proprietary chemicals. Major chemical constituents include nonionic and/or cationic surfactants, emulsifiers and chelating agents. Additional constituents include hydrotropes for retaining organic

ingredients in solution and wetting agents used to assist in the transport of the chemicals into pores and microscopic voids of porous materials. This blend is especially useful in emulsifying hydrocarbons from contaminated surfaces and for holding solubilized metals in solution. Additionally, TECHXTRACT® 0100 may be used as a pretreatment of surfaces heavily contaminated with oils and greases. Chelating agents such as EDTA and nitrilotriacetic acid (NTA) in the 0100 blend are present in moderate to high concentrations and also must be destroyed before heavy metal recovery can be successful. A treatment process recovering the heavy metals in spent TECHXTRACT® solutions is discussed in Section 3.

2.2 INEL's SIMCON 2 Coupons

SIMCON 2 coupons contaminated with a Cs and Zr oxide layer were obtained from INEL and served as surrogates for initial optimization testing. By using non-radioactive surrogates, EET was able to conduct its optimization studies much more quickly and economically. The cylindrical stainless steel SIMCON 2 coupons are approximately 1" in diameter, 1/4" thick, weigh about 25g and contain a 'cold' (non-radioactive) surface oxide layer of the metals.

The numbered coupons were prepared by placing a small amount of an aqueous Cs and Zr containing solution on the circular surface of a coupon. The coupons were then heat treated by air calcination at ca. 700°C forming a surface oxide layer. The SIMCON 2 coupons are of a convenient size to fit into an X-ray fluorescence (XRF) analyzer. INEL performed XRF analysis of the SIMCON 2 coupons to determine the concentrations (mass) of Cs and Zr before (C_o) and after (C_f) treatment. Contamination on SIMCON 2 coupons is representative of a type found at INEL's facility and the coupons have been successfully used to screen prospective decontamination technologies. A more detailed description of SIMCON 2 coupons can be found in a report entitled "Development of Simulated Contamination (SIMCON) and Miscellaneous Decontamination Scoping Tests." The report, prepared by Rick Demmer, was published by Westinghouse Idaho Nuclear Company, Inc., INEL's prime contractor at the time of the report (DE-AC07-8410-12435).

After treatment at EET, the coupons were returned to INEL for analysis of the final Cs and Zr contents. INEL analyzed the coupons and calculated the percentage Cs and Zr removal ($\frac{C_o - C_f}{C_o} \times 100$) and a decontamination factor ($DF = C_o/C_f$) for each coupon. In these calculations, C_o and C_f are measured in micrograms (μg). INEL's detection limit using XRF is 3 μg for both Cs and Zr. Typical initial surface contamination was in the 100 - 200 μg range for each metal. The upper confidence limit of the percentage contaminant removal varies with the initial surface concentration and is therefore limited to about 97 - 98.5%, corresponding to DF values of about 33 - 67. The treated SIMCON 2 coupons were photographed prior to their return to INEL for analysis. They were returned in batches of fourteen and identified only by coupon number.

2.3 Crest Ultrasonic Baths

Based on EET's previous experience, some type of agitation was known to be a critical factor for successful decontamination. Research had shown that the use of ultrasonics is one of the

most efficient ways to achieve this agitation. For this project, sonication was applied by laboratory scale ultrasonic baths from Crest Ultrasonics (model No. 275HT). The baths operate at 120V, 3 amperes, and have approximately a 3/4 gallon liquid capacity. The sonication frequency was 38.5 kHz. The energy output can not be varied. Each unit can regulate the bath temperature from ambient, ca. 25°C, to about 80°C and has a mechanical timer for selection of the sonication duration (0 - 30 min.).

Although the units are constructed from stainless steel, the manufacturer recommended that the baths not come in direct contact with strong acids or other corrosive liquids. Therefore, the decontamination solutions were placed in 500 ml Pyrex beakers. The SIMCON 2 coupons were placed on the bottom of Pyrex beakers containing 300 ml of a test solution. The beakers served as a barrier to protect the Crest units from possible corrosive attack of the test solutions. Two beakers were sonicated in each Crest Unit. The units were filled with tap water containing a small amount of a surfactant to assist in transferring the ultrasonics to the reagents in the Pyrex beakers. The Pyrex beakers somewhat weaken the ultrasonic intensity that reach the coupons, however, any potential damage to the steel lining of the baths is avoided.

2.4 SIMCON 2 Decontamination - Experimental Results and Conditions

TECHXTRACT® 0300, 0200, and 0100 solutions were individually tested and a range of operational parameters were investigated including the effect of TECHXTRACT® concentration, bath temperature, and sonication time. Chemical reagents were added to some blends to evaluate their effect on the performance of the solutions. Temperatures between ambient (25°C) and 70°C were investigated. Treatment temperatures between ca. 40 and 50°C were optimal: further increases in bath temperature did not significantly enhance the decontamination results. Most experiments were therefore performed at ca. 43°C.

The effect of operating parameters was followed in-house by measurement of the weight loss of the coupons and by visual inspection. In their untreated state, the coupon surface was covered with a black oxide layer. Upon progressive treatment, the metallic surface of the stainless steel coupon became visible. These in-house assays served to judge the effectiveness of a particular treatment process to bridge time delays while waiting for the quantitative analysis from INEL. The desired decontamination efficiency for a successful treatment process was at least 95% ($DF \geq 20$).

Optimization of the experimental parameters was an iterative process. A summary of all the experiments performed, coupon weight loss, qualitative surface oxide removal determined by visual inspection, and the quantitative results as measured by INEL using XRF are given in Appendix A.

In the following sections, only pertinent experiments characterizing the effectiveness of TECHXTRACT® formulations are discussed. Section 2.4.1 shows the effectiveness of reagent

chemicals without the TECHXTRACT® blends. Sections 2.4.2 to 2.4.4 present experiments investigating the effectiveness of individual TECHXTRACT® blends with and without additives. Section 2.4.5 gives the effectiveness of sequential TECHXTRACT® chemistry using all three TECHXTRACT® formulations. Section 2.5 summarizes SIMCON 2 decontamination results.

2.4.1 Effectiveness of Reagent Chemicals

Pertinent experiments investigating the effectiveness of the standard reagent chemicals sulfuric acid (H_2SO_4), hydrochloric acid (HCl), and potassium hydroxide (KOH) are presented in this section. These chemicals have been used to enhance the performance of TECHXTRACT® formulations in field experiments and have been used by others for stand-alone decontamination. This section presents the performance of these reagents without the addition of TECHXTRACT® formulations. The experimental conditions and results are presented in Table 2.4.1.1. Unless otherwise noted, these experiments were performed with sonication at 43°C.

Base line experiments were performed using 60 min. sonication in deionized water (DI). The results are shown in Exp. #61, and #74 - #77. The weight loss per coupon in each experiment is shown in Figure 2.4.1.1. The average weight loss was 18.5 mg and the standard deviation was 2.4 mg. Figure 2.4.1.2 presents the corresponding DF values. The average DF values for Cs and Zr were 5.1 and 6.4, respectively. The standard deviations were 1.4 μg (Cs) and 1.1 μg (Zr).

Table 2.4.1.1: *Experiments investigating the effectiveness of enhancement chemicals (no TECHXTRACT® blends).*

Exp. coupon	Decontamination Train Conditions	Time (min.)	Wt. Loss	Visual Oxide Removal	Decon Factors Cs Zr	
#12 (316)	50% HCl	60	53.2 mg	95%	161*	108*
#26 (339)	50% HNO ₃	60	18.3 mg	20%	8.4	6.7
#43 (361)	10% H ₂ SO ₄	90	65.9 mg	65%	73.0*	47.0*
#49 (373)	1.) 10% H ₂ SO ₄	30	66.0 mg	40%	46.7*	25.0*
	2.) 10% HCl	30		45%		
	3.) DI	30		45%		
	4.) KOH and DI @ pH 13 ^(a)	30		60%		
	5.) DI	30		60%		
#58 (392)	1.) KOH and DI @ pH 13 ^(a)	45	19.9 mg	10%	5.4	7.8
	2.) DI	15				
#60 (394)	1.) 10% HCl	45	30.2 mg	65%	46.7	64.0*
	2.) DI	15				
#61 (395)	1.) DI	60	19.6 mg	20%	4.1	6.3
#74 (411)	1.) DI	60	14.4 mg	20%	3.5	5.3
#75 (412)	1.) DI	60	19.2 mg	10%	7.0	8.2
#76 (413)	1.) DI	60	18.5 mg	30%	5.2	5.8
#77 (416)	1.) DI	60	20.6 mg	20%	5.8	6.7
#79 (422)	1.) DI (stirring only, no ultrasonics)	60	1.0 mg	5%	2.8	1.1
#80 (423)	1.) 10% H ₂ SO ₄	45	84.6 mg	20%	30.6	26.3*
	2.) DI	15				
#115 (507)	1.) DI	150	28.1 mg	20%	6	16.4

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

^(a) pH adjusted by KOH

Figure 2.4.1.1: *Weight loss per coupon using a decontamination train consisting of 60 min. sonication in DI water at 43°C. The average weight loss in these experiments was 18.5 mg. The standard deviation was 2.4 mg.*

Figure 2.4.1.2: *DF values for Cs and Zr during SIMCON 2 decontamination using a DI based train. Average DF values were 5.1 (Cs) and 6.4 (Zr). Standard deviations were 1.4 (Cs) and 1.1 (Zr).*

Sonication is essential for effective decontamination. No significant weight loss was observed using only mechanical stirring in DI water for 60 min. (Exp. #79). Sonication in DI water for extended time periods slightly improve contaminant removal from SIMCON 2 coupons (Exp. #115). After 150 min. sonication in DI (Exp. #115), the DF values increased to 6 (Cs) and 16.4 (Zr). Sonication magnifies dramatically the decontamination rate.

Chemicals enhancing the effectiveness of TECHXTRACT® solutions were next investigated using a two train process (45 min. sonication in the solution of interest followed by a 15 min. sonication rinse in DI). Chemical additives encompassed solutions of 10% H₂SO₄, 10% HCl, and KOH pH adjusted DI water (Exp. #80, #60, and #58, respectively). The highest weight loss in these experiments was observed when using a 10% H₂SO₄ solution (Exp. #80), however, this process only removed an estimated 20% of the initially visible oxide layer.

Experiments 58 and 60 investigated sonication in an alkaline DI solution and a 10% HCl solution, respectively. Sonication in pH 13 DI water did not perform superior to sonication in DI. When using a 10% HCl solution, DF values of over 20 were achieved, however, this treatment process only removed an estimated 65% of the visible oxide layer. A five step decontamination train using only reagent chemicals and 30 min. sonication per bath was performed in Exp. #49. The final contaminant levels were sufficient to obtain DF values of over the target value of 20, however, the process only visually cleaned an estimated 60% of the surface.

Single train decontamination processes using common acids were performed using 50% HCl (Exp. #12, 60 min. sonication), 50% HNO₃ (Exp. #26, 60 min. sonication), and 10% H₂SO₄ (Exp. #43, 90 min. sonication). The coupon cleaned by 50% HCl had a weight loss of 53.2 mg and DF values of 161* and 108* for Cs and Zr, respectively. These values were among the highest achieved, however, the excessive chloride content in this solution may complicate heavy metal removal from the composite waste effluent in subsequent ion exchange and incineration processing. The highest weight loss for one step decontamination train using an acid solution was 65.9 mg after 90 min. sonication using a 10% H₂SO₄ bath. The corresponding DF values were 73.0* for Cs and 47.0* for Zr. As will be shown in the following sections of this chapter, the removal of contamination from SIMCON 2 coupons can be dramatically increased using TECHXTRACT® formulations in combination with enhancement chemicals.

Conclusions:

- 1.) Optimum processing temperature is about 40-50°C. Further temperature increases do not dramatically enhance the treatment effectiveness.
- 2.) Sonication, in comparison to mechanical stirring, dramatically increases the cleaning effectiveness of processing solutions.

- 3.) Non-oxidizing mineral acids like H_2SO_4 and HCl are superior to oxidizing additives like HNO_3 .

2.4.2 Effectiveness of TECHXTRACT® 0300 Solutions

This section presents data leading to the selection of TECHXTRACT® 0300 solution to decontaminate SIMCON 2 coupons. TECHXTRACT® 0300 was tested in dilutions up to 1:10 and in conjunction with enhancement reagents, e.g. H_2SO_4 , hydrogen peroxide (H_2O_2), sodium chloride (NaCl), and nickel chloride (NiCl_2). Pertinent experimental findings are summarized in Table 2.4.2.1.

The effect of TECHXTRACT® 0300 solution concentration was investigated in experiments #61 (Table 2.4.1.1), #62, #82, and #84 (Table 2.4.2.1). The DF values, shown in Figure 2.4.2.1, ranged from 6 to 9 for Cs and 5 to 6 for Zr. Using only sonication in DI water for 60 min. (Exp. #61, 0% 0300), the DF values for Cs and Zr were 4.1 and 6.3, respectively. Using full strength 0300 (Exp. #82), the DF values were 5.9 and 4.7 for Cs and Zr, respectively. The 1:10 diluted 0300 solution (Exp. #62) performed slightly better than either the DI or the full strength blend. Neither the full strength 0300 blend or the diluted 0300 blends, without enhancement reached the target DF value of 20.

The addition of H_2SO_4 dramatically increased the effectiveness of the 0300 blends. The effect of 0300 concentration in the presence of 10% (by volume) H_2SO_4 was investigated in experiments #80 (Table 2.4.1.1), #63, #83, and #85 (Table 2.4.2.1) using two step decontamination trains. The DF values are shown in Figure 2.4.2.2. Using full strength 0300 (100% Exp. #83) enhanced with 10% H_2SO_4 , the DF values were 6.3 (Cs) and 10.1 (Zr). The best overall results were achieved using TECHXTRACT® 0300 diluted 1:10 enhanced with 10% H_2SO_4 (Exp. #63). The DF values were 23.9 and 24.7* for Cs and Zr, respectively using H_2SO_4 enhanced 10% 0300. While the DF values were similar for Exp. 80 with 10% sulfuric acid solution and Exp. 63 with 10% TECHXTRACT® 0300 plus 10% sulfuric acid, the oxide layer was 90% removed in Exp. 80 versus only 10% removed in Exp. 63. The superior cleaning efficiency of 10% 0300 + 10% H_2SO_4 was substantiated in Exp. #73 in which DF values of 77* for Cs and 59* for Zr were achieved. These DF values are well above the desired threshold of 20.

Table 2.4.2.1: *Experiments investigating the effectiveness of TECHXTRACT® 0300 based solutions. All experiments were sonicated at ca. 43°C unless otherwise noted.*

Exp. coupon	Decontamination Train Conditions	Time (min.)	Wt. Loss	Visual Oxide Removal	Decon- DF Cs	Factors DF Zr
#16 (321)	100% 0300 (steel pan)	30(b)	16.2 mg	15%	10.3	5.1
#17 (322)	100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ (steel pan)	30(b)	14.9 mg	15%	12.8	14.0
#18 (323)	100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂	30(b)	12.6 mg	10%	11.2	15.9
#59 (393)	1.) 10% 0300 & 20% H ₂ SO ₄	45				
	2.) DI	15	57.0 mg	95%	78.3	61.0*
#62 (396)	1.) 10% 0300	45				
	2.) DI	15	30.6 mg	35%	10.1	5.6
#63 (397)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	71.3 mg	90%	23.9	24.7*
#64 (398)	1.) 10% 0300 (60°C)	45				
	2.) DI (60°C)	15	36.6 mg	45%	8.6	7.4
#65 (399)	1.) 10% 0300 & 10% H ₂ SO ₄ (60°C)	45				
	2.) DI (60°C)	15	71.8 mg	98%	51.0*	25.7*
#66 (400)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	60.4 mg	98%	29.3*	39.0*
#67 (401)	1.) 10% 0300 & 10% H ₂ SO ₄ + 5% NaCl	45				
	2.) DI	15	90.5 mg	95%	91.7*	65.7*
#68 (405)	1.) 10% 0300 & 10% H ₂ SO ₄ + 1% NiCl ₂	45				
	2.) DI	15	232 mg	80%	46.0	21.8
#69 (406)	1.) 10% 0300 & 10% H ₂ SO ₄ + 3% NiCl ₂	45				
	2.) DI	15	191.8 mg	60%	26.8	11.7
#70 (407)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	70.4 mg	96%	69.0*	44.3*
#71 (408)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	90.6 mg	96%	24.5	27.7*
#72 (409)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	72.6 mg	85%	21.0	33.7*

Table 2.4.2.1: (Continued)

#73 (410)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	90.6 mg	96%	77.0*	58.7*
#78 (421)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI mechanical stirring only	45 15	40.0 mg	10%	---	---
#81 (424)	1.) 10% 0300 & 10% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) DI	45 15	37.2 mg	65%	8.4	4.6
#82 (425)	1.) 100% 0300 2.) DI	45 15	14.7 mg	20%	5.9	4.7
#83 (435)	1.) 100% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	28.6 mg	10%	6.3	10.1
#84 (437)	1.) 50% 0300 2.) DI	45 15	48.3 mg	35%	9.2	5.4
#85 (438)	1.) 50% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	56.7 mg	70%	7.3	8.1

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

(a) pH adjusted by KOH

(b) 10 min. presoak in solution at temperature without sonication

Figure 2.4.2.1: Experiments investigating the effectiveness of TECHXTRACT® 0300 concentration (0%, 10%, 50%, and 100%) in the removal of Cs and Zr from SIMCON 2 coupons. The two step decontamination train consisted of 45 min. sonication in an 0300 solution followed by 15 min. sonication in DI. Both baths were at ca. 43°C.

Figure 2.4.2.2: *Experiments investigating the effectiveness of H₂SO₄ enhanced TECHXTRACT® 0300 concentration (0%, 10%, 50%, and 100%) in the removal of Cs and Zr from SIMCON 2 coupons. Each solution contained 10% by volume H₂SO₄. The two step decontamination train consisted of 45 min. sonication in an 0300 solution followed by 15 min. sonication in DI. Both baths were at ca. 43°C.*

Other pertinent experiments investigating the addition of reagent chemicals to the 0300 formulation were performed. Experiments adding NiCl₂ to a 10% 0300 solution enhanced with 10% H₂SO₄ (Exp. # 68 and 69) dramatically increased the weight loss. The coupons had a bright metallic surface and were lightly pitted. However, the addition NiCl₂ (or NaCl, Exp. #67) to enhanced 0300 solutions is not required to achieve very high DF values. The addition of H₂O₂ to the 0300 bath solutions (Exp. #17, #18, and #81) decreased their effectiveness in removing Cs and Zr contamination from SIMCON 2 surfaces.

Two step decontamination processing is shown schematically in Figure 2.4.2.3. SIMCON 2 coupons were sonicated at 43°C for 45 min. in the TECHXTRACT® 0300 bath (1:10 DI diluted enhanced with 10% by volume H₂SO₄) followed by 15 min. sonication in the DI rinse bath. The final contaminant remaining on SIMCON 2 coupons can be reduced to within INEL's detection limits by decontamination in this two step decontamination train, which is referred to as "Train I" elsewhere in this report.

Sequential tests (Exp. #95 - #104) were performed using 45 min. sonication in 1:10 diluted TECHXTRACT® 0300 enhanced with 10% by volume H₂SO₄ bath followed by 15 min. sonication in the DI rinse bath. Both baths were maintained at ca. 43°C and all experiments were performed in the same solutions. By visual observation, treated coupons were 95% to 98% clean. Experimental results and conditions are presented in Table 2.4.2.2.

Figure 2.4.2.3: Schematic of a two step decontamination train (Train I processing) using TECHXTRACT® 0300 and DI. This process (at ca. 43°C) successfully removed Cs and Zr contamination to INEL's detection limit after 45 min. sonication in a 1:10 diluted 0300 formulation enhanced by the addition of 10% H₂SO₄ followed by 15 min. sonication in DI. DF values of 78.5* and 56.7* were measured for Cs and Zr, respectively.

Table 2.4.2.2: Sequential experiments investigating the effectiveness of TECHXTRACT® 0300 based solutions. All experiments were performed at ca. 43°C and under sonication and in the same solutions.

Exp. coupon	Decontamination Train Conditions	Time (min.)	Wt. Loss	Visual Oxide Removal	Decon- DF Cs	Factors DF Zr
#95 (450)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	63.0 mg	97%	78.5	52.0
#96 (451)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	76.7 mg	96%	43.7*	45.0*
#97 (455)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	73.4 mg	97%	23.7*	54.0*
#98 (456)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	68.9 mg	98%	42.0*	26.3*
#99 (458)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	71.6 mg	96%	29.7*	31.3*
#100 (460)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	69.7 mg	98%	43.0*	53.7*
#101 (461)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	73.7 mg	94%	56.0*	41.3*
#102 (471)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	80.8 mg	97%	22.3*	39.0*
#103 (472)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	59.8 mg	95%	18.3*	56.7*
#104 (464)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	76.8 mg	97%	25.7*	31.7*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

Individual Cs and Zr DF values are shown in Figure 2.4.2.4. DF values of up to ca. 80 were achieved using this two step decontamination train. The average DF value for Cs removal in these experiments was 38. For Zr removal, the average DF value was 43. More importantly, the final contaminant levels found in all experiments except #95 were at or below INEL's detection limit. This experimental series demonstrates that treatment of multiple coupons in the same solution is possible.

Figure 2.4.2.4: *DF for Cs and Zr removal from SIMCON 2 coupons using a two step 0300 based decontamination train. The process used 45 min. sonication in a 0300 solution (1:10 +10% H₂SO₄) followed by 15 min. sonication in DI. The baths were at ca. 43°C and all experiments were performed using the same solutions.*

Conclusions:

- 1.) A mixture of 10% TECHXTRACT® 0300 enhanced with 10% H₂SO₄ was found to be the best overall 0300 based treatment solution. Cs and Zr contamination on SIMCON 2 coupons can be reduced to below INEL's detection limit. A 10% H₂SO₄ enhanced 10% 0300 solution performed superior to treatment using 100% 0300, 100% 0300 plus 10% H₂SO₄, 50% 0300, 50% 0300 plus 10% H₂SO₄, 10% 0300, 10% H₂SO₄, and DI treatment .
- 2.) The addition of H₂O₂ to a 10% TECHXTRACT® 0300 plus 10% H₂SO₄ solution decreased the effectiveness of the combined solution.

- 3.) The addition of NiCl_2 and NaCl is not required to achieve high DF values.
- 4.) Sequential treatment of 10 SIMCON 2 coupons in the same 10% 0300 plus 10% H_2SO_4 solution showed no deterioration of the treatment results.
- 5.) In most experiments, treatment in 10% 0300 plus 10% H_2SO_4 removed the Cs and Zr contamination to or below INEL's XRF detection limit.
- 6.) The enhanced TECHXTRACT® 0300 decontamination process (Train I) is preferred over any of the stand-alone reagent chemistries due to the much greater removal of the visible oxide layer.

2.4.3 Effectiveness of TECHXTRACT® 0200 Solutions

This section presents pertinent data for the selection of a TECHXTRACT® 0200 solution to decontaminate SIMCON 2 coupons. TECHXTRACT® 0200 was tested in DI dilutions up to 1:10 and in conjunction with the addition of HCl and H₂SO₄. Experiments using a TECHXTRACT® 0200 based two step decontamination train similar to process shown in Figure 2.4.2.3 were performed. A summary of pertinent experimental results is given in Table 2.4.3.1.

Table 2.4.3.1: *Experiments investigating the effectiveness of TECHXTRACT® 0200 based solutions. All experiments were sonicated at ca. 43°C unless otherwise noted.*

Exp. coupon	Decontamination Train Conditions	Time (min.)	Wt. Loss	Visual Oxide Removal	Decon- DF Cs	Factors DF Zr
#53 (379)	1.) 10% 0200 2.) DI	45 15	21.3 mg	40%	4.2	12.8
#54 (380)	1.) 10% 0200 & 10% HCl 2.) DI	45 15	28.0 mg	70%	31.2	26.4
#86 (439)	1.) 100% 0200 2.) DI	45 15	28.5 mg	10%	46.0	13.0
#87 (440)	1.) 100% 0200 & 10% HCl 2.) DI	45 15	40.3 mg	30%	53.9	53.0*
#88 (442)	1.) 50% 0200 2.) DI	45 15	49.5 mg	40%	42.5	57.7*
#89 (443)	1.) 50% 0200 & 10% HCl 2.) DI	45 15	45.5 mg	30%	41.4	70.7*
#90 (444)	1.) 100% 0200 & 10% H ₂ SO ₄ 2.) DI	45 15	48.0 mg	40%	55.5	51.3*
#91 (445)	1.) 50% 0200 & 10% H ₂ SO ₄ 2.) DI	45 15	40.5 mg	40%	73	85.3*
#92 (447)	1.) 10% 0200 & 10% H ₂ SO ₄ 2.) DI	45 15	41.7 mg	40%	96.9	97.2*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

The effect of chemical concentration (without acid enhancement) was investigated in experiments #53, #61 (Table 2.4.1.1), #86, and #88. The DF values in these experiments are presented in Figure 2.4.3.1. Using a 10% 0200 based train, the DF values were 4.2 and 12.8 for Cs and Zr, respectively. The best DF values in this set of experiment were observed using a 50% 0200 based train. The corresponding DF values were 42.5 for Cs and 57.7* for Zr. By

enhancing the 0200 solution with HCl, or H₂SO₄, higher DF values can be obtained at lower 0200 concentrations.

Experiments investigating the effect of TECHXTRACT® 0200 concentration (0%, 10%, 50%, and 100%) in the presence of 10% by volume HCl were are shown in Figure 2.4.3.2. The HCl enhanced solutions cleaned the SIMCON 2 coupons better than the respective base (non-enhanced) experiments. In this series of experiments, best visual results observed were observed by using a 10% 0200 solution enhanced by 10% HCl (Exp. #54). The visual observation is not reflected in the XRF results shown in Figure 2.4.3.2.

Experiments investigating the effect of TECHXTRACT® 0200 concentration in the presence of 10% by volume H₂SO₄ are shown in Figure 2.4.3.3. In all experiments, coupons cleaned by the H₂SO₄ enhanced solutions performed superior to the respective non-enhanced base experiments. The best results in this series was observed using a 10% 0200 solution enhanced by 10% H₂SO₄. The DF values of a 10% H₂SO₄ enhanced 1:10 diluted 0200 based train were 96.9 and 97.2* for Cs and Zr, respectively.

Figure 2.4.3.1: *Experiments investigating the effectiveness of TECHXTRACT® 0200 concentration (0%, 10%, 50%, and 100%) in the removal of Cs and Zr from SIMCON 2 coupons. The two step decontamination train consisted of 45 min. sonication in an 0200 solution followed by 15 min. sonication in DI. Both baths were at ca. 43°C.*

Figure 2.4.3.2: *Experiments investigating the effectiveness of HCl enhanced TECHXTRACT® %0200 concentration (0%, 10%, 50%, and 100%) in the removal of Cs and Zr from SIMCON 2 coupons. Each solution contained 10% by volume HCl. The two step decontamination train consisted of 45 min. sonication in an 0200 solution followed by 15 min. sonication in DI. Both baths were at ca. 43°C.*

Figure 2.4.3.3: *Experiments investigating the effectiveness of H₂SO₄ enhanced TECHXTRACT® %0200 concentration (0%, 10%, 50%, and 100%) in the removal of Cs and Zr from SIMCON 2 coupons. Each solution contained 10% by volume H₂SO₄. The two step decontamination train consisted of 45 min. sonication in an 0200 solution followed by 15 min. sonication in DI. Both baths were at ca. 43°C.*

Conclusions:

- 1.) The best cleaning results using base (non-enhanced) 0200 blend was observed using a 50% (1:1 DI diluted) solution.
- 2.) The addition of 10% HCl to 0200 solutions increased the cleaning effectiveness in the entire 0200 concentration range.
- 3.) The addition of 10% H₂SO₄ to 0200 solutions increased the cleaning effectiveness in the entire 0200 concentration range. The best overall results were observed using 10% (1:10 DI diluted) 0200 plus 10% H₂SO₄.

2.4.4 Effectiveness of TECHXTRACT® 0100 Solutions

This section presents pertinent data for the selection of a TECHXTRACT® 0100 solution to decontaminate SIMCON 2 coupons. TECHXTRACT® 0100 was tested in dilutions up to 1:10. The experiments were performed in TECHXTRACT® 0100 based baths using a two step decontamination train at 43°C similar to process shown in Figure 2.4.2.1. A summary of the experiments is given in Table 2.4.4.1. Individual Cs and Zr DF values are shown in Figure 2.4.4.3.

Treatment in a train based on TECHXTRACT® 0100 solutions at full strength (100%) and 50% (Exp. 93 and 94) were not superior to treatment using only DI (Exp. #61). The removal of Cs and Zr contamination from SIMCON 2 coupons in a train based on a 10% 0100 solution (Exp. #55) and a 10% 0100 pH adjusted to ca. 13.5 produces a DF value of 5.5 (Cs) and 4.2 (Zr). The best visual results were obtained using 10% 0100 solutions (Exp. #55 and #56).

Table 2.4.4.1: *Experiments investigating the effectiveness of TECHXTRACT® 0100 based solutions. All experiments were sonicated at ca. 43°C unless otherwise noted.*

Exp. coupon	Decontamination Train Conditions	Time (min.)	Wt. Loss	Visual Oxide Removal	Decon- Factors	
					DF Cs	DF Zr
#55 (387)	1.) 10% 0100	45	19.3 mg	45%	3.9	16.0
	2.) DI	15				
#56 (390)	1.) 10% 0100 @ pH 13.5 ^(a)	45	16.2 mg	40%	5.5	4.2
	2.) DI	15				
#93 (448)	1.) 100% 0100	45	15.5 mg	5%	2.8	9.8
	2.) DI	15				
#94 (449)	1.) 50% 0100	45	17.1 mg	5%	3.8	20.0
	2.) DI	15				

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

(a) pH adjusted by KOH

Figure 2.4.4.1: *Experiments investigating the effectiveness of TECHXTRACT® 0100 concentration in the removal of Cs and Zr from SIMCON 2 coupons. The two step decontamination train consisted of 45 min. sonication in an 0100 solution followed by 15 min. sonication in DI. Both baths were at ca. 43°C.*

Conclusions:

- 1.) Enhancement of 0100 blend by raising the pH to ca. 13.5 by KOH had no major effect on the removal efficiency of Cs and Zr contamination from SIMCON 2 coupons.
- 2.) The effectiveness of the 0100 formulation is much smaller than the effects observed with enhanced 0300 and 0200 formulations.
- 3.) The best visual oxide removal results were observed using 10% (1:10 DI diluted) 0100 blends.
- 4.) These results confirm EET's expectations. An acid, such as TECHXTRACT® 0300 or 0200, is needed to solubilize metals and metal oxides before effective decontamination is achieved.

2.4.5 Effectiveness of Sequential Treatment in TECHXTRACT® Based Solutions

The experiments presented in the preceding sections of this chapter lead to the selection of individual TECHXTRACT® solutions to decontaminate SIMCON 2 coupons. This section presents the results of using all three TECHXTRACT® blends in a five step decontamination train shown schematically in Figure 2.4.5.1. Unless otherwise noted, all experiments were performed at ca. 43°C and under sonication. This five step process can reduce Cs and Zr

contamination on SIMCON 2 coupons to below the detection limits of the XRF analysis used by INEL.

Figure 2.4.5.1 *Schematic of a five step decontamination train (Train II processing) using TECHXTRACT® based solutions. The enhanced TECHXTRACT® solutions are 1:10 dilutions. Train II processing successfully removed initial Cs and Zr contamination to below INEL's background measurements in most experiments. DF values of up to 25* for Cs and 79* for Zr were measured.*

Sequential tests were performed using a five step sequence, 30 min. sonication per bath and “enhanced” 1:10 TECHXTRACT® solutions in experiments #105 - #109. Enhancement involves the addition of the most effective reagents from the individual tests (Sections 2.4.2 to 2.4.4). The total treatment time was 150 min. Experimental results and conditions are presented in Table 2.4.5.1. Each coupon in this sequence was treated without changing the treatment solutions. The final Cs and Zr contamination on treated SIMCON 2 coupons were reduced to or below INEL's background measurements in all experiment. Decontamination factors of up to 25* for Cs removal (Exp. #106) and 79* for Zr removal (Exp. #105) were observed. Individual Cs and Zr DF values are shown graphically in Figure 2.4.5.2.

A similar sequence using 15 min. sonication per bath was performed in experiments #110 - #114. Total treatment time in these experiments was 75 min. The experimental results and conditions are presented in Table 2.4.5.2. Individual DF values are presented graphically in Figure 2.4.5.3. Again, all coupons were treated without changing the solutions. All experiments showed final contaminant levels at or below INEL's detection limit. The last coupon treated in this sequence (Exp. #114) showed a DF of 26* for Cs removal and a DF of 37* for Zr removal. Treating multiple coupons sequentially in the same solutions at reduced sonication times is possible depending on the desired level of decontamination.

Table 2.4.5.1: *Sequential experiments investigating the effectiveness of TECHXTRACT® chemistry using Train II processing and 30 min. sonication per bath. All experiments were performed using the five step train 0300-0200-DI-0100-DI (all baths were sonicated at ca. 43°C). The same solutions were used in all five experiments.*

Exp. coupon	Decontamination Train Conditions	Time (min.)	Wt. Loss	Visual Oxide Removal	Decon- DF Cs	Factors DF Zr
#105 (468)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #1 of 5 in the same solutions	30 30 30 30 30	72.2 mg	96% 99% 99% 100% 100%	24.7*	79.3*
#106 (469)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #2 of 5 in the same solutions	30 30 30 30 30	82.8 mg	94% 98% 98% 99% 99%	25.3*	29.0*
#107 (473)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #3 of 5 in the same solutions	30 30 30 30 30	73.8 mg	92% 97% 97% 98% 98%	18.7*	42.7*
#108 (476)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #4 of 5 in the same solutions	30 30 30 30 30	79.4 mg	96% 99% 99% 99% 99%	21.3*	51.3*
#109 (479)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #5 of 5 in the same solutions	30 30 30 30 30	78.5 mg	98% 98% 98% 99% 99%	19.3*	63.7*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

(a) pH adjusted by KOH

Table 2.4.5.2: *Sequential experiments investigating the effectiveness of TECHXTRACT® chemistry using Train II processing and 15 min. sonication per bath. All*

experiments were performed using the five step train 0300-0200-DI-0100-DI (all baths were sonicated at ca. 43°C). The same solutions were used in all five experiments.

Exp. coupon	Decontamination Train Conditions	Time (min.)	Wt. Loss	Visual Oxide Removal	Decon- DF Cs	Factors DF Zr
#110 (478)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #1 of 5 in the same solutions	15 15 15 15 15	71.6 mg	70% 75% 75% 80% 84%	18.7*	33.7*
#111 (484)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #2 of 5 in the same solutions	15 15 15 15 15	83.0 mg	80% 90% 93% 93% 95%	27.7*	30.3*
#112 (498)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #3 of 5 in the same solutions	15 15 15 15 15	80.5 mg	90% 96% 98% 99% 99%	30.7*	40.7*
#113 (499)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #4 of 5 in the same solutions	15 15 15 15 15	81.4 mg	85% 92% 95% 98% 98%	18.3*	37.7*
#114 (502)	1.) 10% 0300 plus 10% H ₂ SO ₄ 2.) 10% 0200 plus HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI run #5 of 5 in the same solutions	15 15 15 15 15	78.3 mg	50% 60% 85% 95% 96%	26.0*	36.7*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

(a) pH adjusted by KOH

Figure 2.4.5.2: *Same solution experiments removing Cs and Zr contamination from SIMCON 2 coupons using Train II processing. Conditions are shown in Table 2.4.5.1.*

Figure 2.4.5.3: *Same solution experiments removing Cs and Zr contamination from SIMCON 2 coupons using a five step decontamination train (15 min. sonication per step). Conditions are as shown in Table 2.4.5.2.*

2.5 Conclusions

Two decontamination trains provided successful removal of Cs and Zr deposits from SIMCON 2 coupons. Both processes were performed at 43°C. Train I processing, shown schematically in Figure 2.4.2.3, uses 45 min. sonication in a 1:10 DI diluted TECHXTRACT® 0300 blend enhanced with 10% H₂SO₄ followed by 15 min. sonication in a DI rinse bath. The Cs and Zr contamination on the SIMCON 2 coupons were reduced to or below the detection limit.

The second successful treatment sequence, Train II, made use of the interactive TECHXTRACT® chemistry. This five step process involves sequential 30 min. sonication in different baths (0300 - 0200 - DI - 0100 - DI). The TECHXTRACT® blends in both decontamination sequences were diluted 1:10 and modified to enhance their effectiveness in decontamination of SIMCON coupons. DF values of up to 31* (Cs) and 41* (Zr) were measured using Train II processing. Multiple coupons can be treated in the same baths without compromising their decontamination effectiveness and shorter treatment times are possible depending on the desired level of decontamination.

Both of these decontamination trains were effective in removing the surrogate contaminants (Cs and Zr) to below XRF detection limits and in removing essentially all of the oxide layer without any visible damage to the stainless steel substrate. Train I is a much simpler process. EET retained the Train II process for subsequent testing because of research suggesting that other contaminants may require this more complex treatment for effective decontamination.

3.0 Heavy Metal Removal from Spent TECHXTRACT® Solutions

This section describes treatment processes for removing heavy metals from spent TECHXTRACT® waste streams. The decontamination processes described in Section 2 generate spent chemical solutions which contain the contaminants. TECHXTRACT® 0100 and 0200 solutions contain moderate to high concentrations of chelating agents. Even though the TECHXTRACT® blends may be diluted, the chelates in the effluent streams must be removed or destroyed prior to successful heavy metal recovery from the spent solutions. This chapter is divided into the following sections: 3.1 defines the problem statement; 3.2 describes the treatment of Train I (TECHXTRACT® 0300 and DI) effluent; 3.3 presents the treatment of spent extraction fluids from Train II (TECHXTRACT® 0300-0200-DI-0100-DI); 3.4 gives an approach for Cs and Zr recovery from concentrated effluent waste streams; 3.5 - 3.7 describe experimental results for simulated effluents from Train I, II, and III processes; and 3.8 discusses the results of ion exchange resin experiments.

3.1 Problem Definition

Two decontamination trains successfully removing cesium (Cs) and zirconium (Zr) deposits from SIMCON 2 coupons have been identified. Train I processing uses only a 10% TECHXTRACT® 0300 solution enhanced with sulfuric acid. Train II processing is based on interactive TECHXTRACT® chemistry and uses all three TECHXTRACT® formulations. Train II may have to be applied in situations where surface contamination products are different in their chemical nature and harder to clean than the contaminants deposited onto SIMCON 2 coupons.

TECHXTRACT® 0300 solutions are common to both treatment trains and do not contain chelates that would interfere with standard heavy metal recovery techniques. Heavy metals forming insoluble hydroxides (e.g. Zr) can be recovered by adjusting the pH, precipitating the heavy metals in their hydroxide state, and filtration. The introduction of TECHXTRACT® 0200 and 0100 blends to Train II processing baths introduces chelating agents and additional organic complexers into the effluent streams. In this case, the waste treatment process is substantially more complicated. The new treatment problems to remove heavy metals from the waste stream are: (1.) The combined effluent from concentrated TECHXTRACT® formulations used in Train II processing forms an emulsion; (2.) Undiluted TECHXTRACT® 0200 contains ca. 10,000 ppm chelates by titration using EPA method 2340 C; (3.) Undiluted TECHXTRACT® 0100 contains ca. 100,000 ppm chelates by titration using EPA method 2340 C; and (4.) Additional organics and complexing agents (e.g. citric and oxalic acid) are introduced into the combined waste stream.

Heavy metal recovery from Train II effluents require additional processing. The four basic steps are listed below and are discussed in detail in Section 3.3.

1. Breaking the emulsion and phase separation of the aqueous and organic phases. Organic phase processing may require

- incineration or extraction of the chelates into an aqueous phase by acid washing.
2. Partial removal of chelates in the aqueous phase by crystallization.
 3. Destruction of remaining chelates in the aqueous phase by UV/H₂O₂ photolysis.
 4. Removal of the heavy metals by traditional removal processes (e.g. Fe³⁺ co-precipitation and filtration of heavy metal hydroxides).

Cesium will not be effected by this treatment scheme since it does not form an insoluble metal hydroxide. Therefore, Cs in the effluent waste streams must be removed by other techniques (e.g. cesium selective ion exchange resins). A cesium selective ion exchange resin (IONSIV®IE-911) was obtained from UOP to evaluate cesium removal from simulated TECHXTRACT® processing streams.

3.2 Processing of Train I Effluent

Train I effluent was simulated by mixing 100 ml TECHXTRACT® 0300 with 900 ml DI water. This solution was spiked with ca. 1000 ppm iron (Fe³⁺). The pH of the mixture was raised to pH 9.5 and the precipitated iron hydroxide was removed by filtration. Chemical analysis of the filtered aqueous stream showed an iron concentration of ca. 0.02 ppm. Based on this analysis, the simulated Train I effluent stream does not contain chelating agents that would interfere with recovery of insoluble heavy hydroxides. Fe recovery in this experiment was over 99.9%.

A schematic for heavy metal recovery from a Train I effluent is shown in Figure 3.2.1. The rinse tank effluent and tank blow down streams are combined into a holding tank. The effluent may be batch treated using Fe³⁺ co-precipitation. Iron, 1000 ppm Fe³⁺ is added to the combined effluent in the holding tank and the pH is raised to 9.5. Fe³⁺ based and other precipitated metal hydroxides are removed by filtration. Co-precipitation of heavy metal hydroxides in the presence of Fe³⁺ can reduce the final metal concentration by 2 orders of magnitude (Figure 3.2.2). Compounds which do not form insoluble heavy metal hydroxides (e.g. cesium) are removed in a second treatment step (e.g. Cs selective ion exchange resins). The final effluent is expected to be clean enough for discharge, depending on site-specific discharge permits.

Figure 3.2.1: Schematic outlining heavy metal removal from Train I (0300 - DI).

3.3 Processing of Train II Effluent

Train II effluents originate from TECHXTRACT® 0300, 0200, 0100, and rinse baths. Train II processing would be required in field applications where the surface contamination is more difficult to remove than that deposited on SIMCON 2 coupons. A worst case waste treatment scenario would require effluent treatment from a Train II process based on fully concentrated TECHXTRACT® solutions. A combined effluent based on baths of undiluted TECHXTRACT® blends was simulated under laboratory conditions. This 2,000 ml sample of the combined effluents consisted of the following approximate composition:

	400 ml	TECHXTRACT® 0100	(20% by volume)
	300 ml	TECHXTRACT® 0200	(15% by volume)
	300 ml	TECHXTRACT® 0300	(15% by volume)
	<u>1,000 ml</u>	<u>DI Water</u>	<u>(50% by volume)</u>
Total:	2,000 ml	Simulated effluent	(100 % by volume)

The volume percentage of TECHXTRACT® 0100 was chosen as 20% because 0100 may sometimes be used as a pretreatment step to degrease parts prior to the decontamination train.

The combined effluent forms an emulsion and has a high chelate content. The chelate content of the simulated effluent is approximately 20,000 ppm (using method 2340 C) when using fully concentrated TECHXTRACT® blends. If the blends are all diluted 1:10, the chelate content of the combined waste stream is about 2,000 ppm. In either case, the chelate content is too high to successfully apply the treatment process outlined in Section 3.2 for recovery of heavy metals from the combined waste stream.

Waste treatment of the Train II effluents is discussed in the following sections of this chapter. The treatment process involves breaking the initial emulsion into organic and aqueous phases (3.3.1); reducing the initial chelate content by crystallization and extraction (3.3.2); destroying the aqueous phase chelates by UV/H₂O₂ photolysis (3.3.3); and recovering the heavy metals from the final solution (3.3.4).

3.3.1 Effluent De-emulsification

The simulated spent TECHXTRACT® waste stream forms a milky white emulsion that can not be readily separated into distinctive organic and aqueous phases at room temperature. Bench scale experiments demonstrated that acidification and heat can be used to break the emulsion. The addition of approximately 25 ml concentrated H₂SO₄ per liter waste stream composite is sufficient to break the emulsion after heating the mixture to ca. 65°C. This mixture has a pH of ca. 0.5 and can readily be separated into distinctive organic and aqueous phases.

3.3.2 Chelate Reduction by Crystallization and Extraction

Phase separation of the broken emulsion produces an organic layer ca. 15% by volume of the simulated Train II effluent (see Section 3.3 for composition). The aqueous layer has a chelate content (by method 2340 C) of ca. 20,000 ppm. EDTA is present in moderate to high concentrations in TECHXTRACT® 0200 and 0100 formulations and NTA is present in the TECHXTRACT® 0100 formulation. If diluted 1:10, the TECHXTRACT® blends as used in Train II processing would have a chelate content of ca. 2,000 ppm. The final chelate content will be dependent on the dilution of the TECHXTRACT® blends and the blow down ratios.

These chelating agents exhibit a minimum solubility in water at a pH between 1.0 to 3.0. Experimental solubility in water of pure component EDTA or NTA as a function of pH is presented in Figure 3.3.2.1. In these experiments, the pH was adjusted by H₂SO₄ or NaOH when required. NTA in water exhibits a minimum solubility of ca. 250 ppm at a pH of about 1.5. EDTA in water exhibits a minimum solubility of ca. 200 ppm at a pH of about 2.2. A minimum combined chelate solubility is expected at a pH of ca. 1.5.

Figure 3.3.2.1: *Pure component aqueous phase equilibrium EDTA and NTA concentrations as a function of pH.*

To address the possibility of using phase separation via chelate crystallization in the spent aqueous (phase separated) phase, the pH was raised to 1.5 by NaOH. After equilibration with seed crystals, the chelate concentration in the aqueous phase was reduced from ca. 20,000 ppm to about 600 ppm.

The chelate content of the organic layer can be lowered via acid extraction. The organic layer was extracted in an experiment using acidic DI water containing 25 ml per liter H₂SO₄. Five equal volume extractions were performed, and each extraction used fresh extraction fluid. The extractions were performed at ca. 65°C to prevent reformation of the organic-water emulsion. Figure 3.3.2.2 presents the chelate content (using method 2340 C) of the aqueous

phases after each extraction. The initial chelate content of the organic phase was calculated to be about 2,200 ppm. Extractions numbers 6, 7, and 8 were extrapolated from the existing data. The chelate partitioning coefficient between the organic and acidic extraction fluid was estimated to be approximately unity, i.e. $\frac{[\text{chelate}_{\text{organic}}]}{[\text{chelate}_{\text{aqueous}}]} = 1$.

Figure 3.3.2.2: *Titrated chelate content of acidified aqueous extraction fluids after equal volume extractions of the organic phase. Five extractions were performed. Extraction number 6, 7, and 8 were extrapolated from the existing data. The extraction fluid was 25 ml H₂SO₄ per liter water.*

If the chelate content of the combined extraction fluids are low, they can be combined with the filtrate from the crystallization step. If the chelate content is too high, the spent solution needs to be reintroduced into the crystallization process. The remaining chelates in the combined aqueous solutions can be destroyed by UV/ H₂O₂ photolysis and is discussed in Section 3.3.3. Another attractive alternate to extraction of the chelates would be incineration of the organic phase.

3.3.3 Chelate Destruction by UV/H₂O₂ Photolysis

The remaining chelate concentration is still too high for effective waste treatment, and cannot be reduced further without destruction of the chelants. Based upon EET's research, a UV/H₂O₂ photolysis process was selected for final chelant reduction.

Exposure of hydrogen peroxide to the appropriate wave length radiation generates hydroxyl radicals ($\text{H}_2\text{O}_2 + h\nu \rightarrow 2\bullet\text{OH}$, $\lambda < \text{ca. } 260 \text{ nm}$). In an aerated aqueous environment, hydroxyl radicals initiate a series of reactions that lead to the room temperature oxidation of most organics. Carboxylic acid groups, functional groups common to the chelates EDTA, NTA, oxalic acid and citric acid are highly susceptible to hydroxyl radical attack.

The UV radiation source used in the UV/ H_2O_2 photolysis experiments consisted of a bank of six Slimline germicidal lamps. The lamps, obtained from American Ultraviolet Co.(model no. G36T6L, AUV PN GML100), are rated at 39 W each and have a near UV output of 13.8 W each ($\lambda_{\text{max}} = 254 \text{ nm}$). Each germicidal lamp was independently powered by a 120V, 60 hz, power supply ballast having a 430 mA output (ballast model no. TX010, AUV PN G1036SL 120/60 TX). Axial spacing between the 36" lamps was approximately 2.5". The distance between the lamps and a fluid surface during UV exposure was typically about $12" \pm 4"$. A light box, dimensions 4' x 2' x 2', was constructed to contain the lamps and provide shielding of the operator from direct exposure to UV light. The total near UV output of the light box is ca. 80W.

Chelate destruction experiments in the presence of UV irradiation were performed on EDTA and NTA solutions, as well as the aqueous phase separated from the broken effluent train emulsion. At a given hydrogen peroxide concentration, the chelate concentration can be modeled by first order kinetics: $C_{\text{chelate}} = C_{\text{chelate},0} \times \exp[-k(t-t_0)]$; where k is a first order rate constant. The initial pH of the EDTA, NTA, and spent TECHXTRACT® (aqueous) solutions were 3.1, 2.4, and 1.5 respectively. Varying the pH did not significantly change the chelate destruction rates.

Selected chelate destruction experimental results and conditions are presented in Table 3.3.3.1. The chelates are destroyed by exposure to UV radiation ($\lambda < \text{ca. } 260 \text{ nm}$) and the appropriate co-catalysts (i.e. H_2O_2 and Fe^{3+}). In all experiments, small amounts of hydrogen peroxide increased the rate of chelate destruction during photolysis. No significant decrease in chelate content was observed in the absence of UV radiation, or in the presence of H_2O_2 without UV radiation. The optimal hydrogen peroxide concentration during EDTA and NTA photolysis experiments corresponded to a solution volume of ca. 0.4% to 1.0% of a 30% H_2O_2 solution.

Chelate destruction in spent TECHXTRACT® aqueous streams can be enhanced by the addition of Fe^{3+} . In the absence of both hydrogen peroxide and Fe^{3+} , chelates during auto-oxidation experiments (UV irradiated, no additives) in the aqueous TECHXTRACT® waste stream showed a half life of ca. 9.4 hr. Upon the addition of a small amount of hydrogen peroxide (0.4% of a 30% H_2O_2 solution,) the half life is decreased to 1.9 hr (Figure 3.3.3.2). In the presence of 660 ppm Fe^{3+} and hydrogen peroxide, chelate half life can be reduced to 1.3 hours (Figure 3.3.3.3).

Table 3.3.3.1: Summary of kinetic data for chelate destruction using UV photolysis.

System	Additives ^(a)	Initial chelate concentration	First order rate constant k (hr ⁻¹)	half life (hr)	<u>volume</u> surface (cm)
EDTA	Auto-oxidation	≈ 500 ppm	0.55	1.3	6
	0.1 % H ₂ O ₂	"	1.88	0.37	"
	0.4 % H ₂ O ₂	"	2.11	0.33	"
	0.4 % H₂O₂	"	2.4	0.29	"
	1.0 % H ₂ O ₂	"	1.35	0.51	"
	1.0 % H ₂ O ₂	"	1.51	0.45	"
	4.0 % H ₂ O ₂	"	1.36	0.51	"
	10.0 % H ₂ O ₂	"	0.43	1.61	"
NTA	Auto-oxidation	≈ 700 ppm	0.11	6.3	6
	0.4 % H ₂ O ₂	"	0.28	2.5	"
	1.0 % H₂O₂	"	0.53	1.3	"
	4.0 % H ₂ O ₂	"	0.18	3.8	"
TECHXTRACT [®] mixture	Auto-oxidation	≈ 1000 ppm	0.074	9.4	1
	0.4 % H₂O₂	"	0.35	1.9	"
	1.0 % H ₂ O ₂	"	0.32	2.2	"
	4.0 % H ₂ O ₂	"	0.16	4.3	"
	330 ppm Fe ³⁺ (no H ₂ O ₂)	≈ 1460 ppm	0.20	3.5	"
	660 ppm Fe ³⁺ 1.5 % H ₂ O ₂	≈ 2200 ppm	0.55	1.3	"

^(a) H₂O₂ % corresponds to volume % of a 30% H₂O₂ solution.

Figure 3.3.3.1: *Chelate destruction kinetics in spent TECHXTRACT® (aqueous phase) mixtures using UV photolysis in the absence of Fe^{3+} .*

Figure 3.3.3.2: *Chelate destruction kinetics in spent TECHXTRACT® (aqueous phase) mixtures using UV photolysis in the presence of Fe^{3+} .*

3.3.4 Fe³⁺ Recovery from Simulated 0200-0100 Blow Down Streams

The chelate crystallization and UV/H₂O₂ destruction described in Section 3.3.3 were obtained using pure EDTA and NTA solutions and simulated combined effluents initially free of heavy metals. In addition to EDTA, TECHXTRACT® 0200 and 0100 blends contain other organic complexers. In this section, the combined waste effluent was spiked with Fe³⁺ prior to chelate crystallization and UV destruction.

The strength of metal-chelate complexes is strongly influenced by the valence state of the metal and the pH of the aqueous solution. Monovalent metals form no complexes; divalent metals, like Ca²⁺, form stable complexes in the alkaline domain, but not in the acidic region. Higher valent metals, like Fe³⁺, form complexes that are stable even at low pH values. Iron (Fe³⁺) chelate complexes in the simulated effluent are not broken at a pH 1.5 at which the EDTA and NTA crystallization is performed. In this case, the chelate concentration of the aqueous phase is the sum of "equilibrium chelate" plus "heavy metal complexed chelate". The "equilibrium chelates" can be titrated by EPA method 2340 C. The presence of high valent heavy metals in the combined effluent increases the amount of chelates that need to be destroyed during the UV treatment prior to successful recovery of the metals. Thus, the presence of complexed metals increase the radiation time necessary for heavy metal recovery after UV/H₂O₂ treatment of the effluent.

To test the effect of high valent metals on the crystallization process, a solution composed of 0.02 mole/l EDTA, 0.02 mole/l NTA, and 0.04 mole/l Fe³⁺ was adjusted to pH 1.5 and was contacted with EDTA and NTA seed crystals to ensure solid-liquid equilibrium. Upon filtration of the crystallized chelates, the filtrate was adjusted to pH = 9.5. No visible formation of an Fe³⁺ hydroxide precipitate was observed, but the solution did exhibit a yellow discoloration typical of Fe³⁺. The pH 9.5 solution was filtered and sent for Fe analysis. The Fe content remaining in solution was about 90% of that found in the original sample, indicating the Fe was mostly complexed and could not be separated from the solution by a simple pH adjustment and filtration in the presence of EDTA and NTA. The free and complexed chelates must first be destroyed prior to heavy metal recovery from the simulated waste stream.

UV/H₂O₂ chelate destruction experiments were performed using a simulated 0100-0200 blow down stream. The simulated effluent stream consisted of 50 ml concentrated TECHXTRACT® 0100, 50 ml concentrated TECHXTRACT® 0200, and 900 ml DI. This solution was spiked with ca. 1000 ppm Fe. The solution was then acidified to pH 0.5, heated, and phase separated. The pH of the aqueous phase was adjusted to 1.5, equilibrated with EDTA and NTA seed crystals, and then filtered.

The solution was UV irradiated in the presence of different concentration of H₂O₂ (0%, 1%, and 4 vol.% of a 30% H₂O₂ solution). Samples were taken at six hour intervals and the H₂O₂ content was adjusted when required. Each sample was then diluted 1:10, adjusted to pH = 9.5, and filtered. Fe remaining in solution was analyzed by ICAP. The Fe retention results (ppm Fe remaining in solution) are presented in Figure 3.3.4.1. Over 99.9% of the original Fe

content was recovered after ca. 20 hr irradiation in the presence of ca. 4% H_2O_2 and after ca. 30 hr irradiation in the presence of 1% H_2O_2 . Irradiation in the absence of H_2O_2 did not significantly reduce the Fe content during the first 36 hr.

Figure 3.3.4.1: *Fe retention during H_2O_2 photolysis of a 0100 - 0200 blow down stream containing ca. 1000 ppm Fe. Initial H_2O_2 content was 0% 1%, 4% by volume (of a 30% H_2O_2 by volume solution). H_2O_2 content was adjusted after each sample was taken.*

Conclusions:

- 1.) The preceding section demonstrated that SIMCON 2 coupons can be decontaminated using either Train I or Train II. The process solutions used in the decontamination train has a pronounced effect upon heavy metal removal from the resulting waste effluent.
- 2.) In Train I, the TECHXTRACT® 0300 effluent does not contain chelating agents that interfere with conventional waste treatment methods. Precipitation of insoluble heavy metal hydroxides from the combined waste effluent, preferably co-precipitation in the presence of Fe^{3+} , is possible.

- 3.) In Train II, the chelates present in the TECHXTRACT® 0200 and 0100 blends must first be reduced and/or destroyed before standard metal recovery techniques may be employed. Waste treatment of the combined Train II effluent may require phase separation, extraction of chelates from the organic phase or organic phase incineration, aqueous phase chelate crystallization and UV/H₂O₂ photolysis. Heavy metal recovery from the Train II effluent is possible but much more complicated and expensive.

3.4 Conceptual Treatment of Concentrated TECHXTRACT® Effluent Streams

The results described in the preceding sections of chapter 3 lead to a conceptual treatment scheme outlined in Figure 3.4.1. The combined effluent containing concentrated TECHXTRACT® streams from the 0300, 0200, and 0100 baths and the two DI rinses are acidified with concentrated sulfuric acid to pH 0.5 and heated to ca. 65°C. This treatment causes a phase separation and allows isolation of an organic and an aqueous phase. The chelate and heavy metal containing organic phase is separated and either sent to direct disposal (e.g. incineration) or treated by washing the chelates into an acidified aqueous stream by organic-water extraction until the specified discharge limits are achieved. The acidic wash water containing chelates and heavy metals is combined with the aqueous phase collected from the phase separation process.

Free chelates are removed in a crystallization operation at pH 1.5. Crystallization lowers the free chelate concentration into the 600 - 1000 ppm range. The solution is filtered and the solids are sent to disposal. In addition to a low concentration of chelates, the aqueous effluent contains chelates bound to metals and free metals. Recovery of the chelate bound metals necessitates the destruction of all remaining chelates. This can be achieved by an advanced oxidation process (AOP) reactor using UV/H₂O₂ photolysis. The efficiency of heavy metal removal depends on the completeness of chelate destruction, which is a function of light intensity and irradiation time. After chelate destruction, the effluent is treated for heavy metal removal by hydroxide precipitation (e.g. in the presence of Fe III). Any remaining soluble metals such as Cesium are removed by ion exchange using commercially available resins.

Field experiments performed at Alaron Corp., Wampum PA verified that concentrated TECHXTRACT® baths are not required to achieve successful surface decontamination of a wide spectrum of substrates and contaminants. Solutions based on enhanced TECHXTRACT® blends diluted 1:10 are sufficient. Laboratory experiments performed on simulated effluents using 1:10 enhanced TECHXTRACT® blends indicated that the phase separation and crystallization steps are not necessary. Therefore, experimental work on the treatment of concentrated streams was discontinued.

Figure 3.4.1: Conceptual treatment scheme for heavy metal removal from effluents based on concentrated TECHXTRACT® streams.

3.5 Expected Train I Waste Treatment Results

EET's initial decontamination experiments showed that dilute chemical baths produced very good results. Therefore, EET's final waste treatment testing was based on the expected effluent streams. Simulated effluents were mixed using non-radioactive surrogates in combined TECHXTRACT® waste fluids to evaluate the performance of the proposed waste treatment schemes using the procedures outlined in the previous section. Train I waste treatment consists of co-precipitation of the heavy metals using Fe^{3+} and cesium removal using ion resin.

The blow down stream from Train I processing contains TECHXTRACT® 0300, sulfuric acid, dissolved metals, and DI water. Train I simulated effluent is based on the assumption that both the chemical and rinse baths are replenished at the same intervals, giving the chemical bath an effective 1:1 dilution in the blow down stream. Train I effluent was spiked with the heavy metals Co, La, Ni, U, and Zr. Cesium was added in order to evaluate the performance of the ion resin. A simulated Train I effluent consisted of the following composition brought to 1 liter volume in DI water:

50 ml TECHXTRACT® 0300
50 ml Conc. H_2SO_4
≈ 500 ml DI water
Plus simulated contaminants
1.01 g $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$
0.67 g $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$
1.01 g $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$
0.53 g $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$
0.68 g $\text{ZrOCl}_2 \cdot x\text{H}_2\text{O}$ (assay 50% ZrO_2)
1.47 g CsNO_3
Total volume adjusted to 1 liter

This mixture contains 250 ppm each of Co, La, Ni, U, and Zr for a total heavy metal content of 1250 ppm. The Cesium loading is 1200 ppm. Waste treatment involves co-precipitation of the heavy metals using Fe^{3+} . The mixture was spiked with 2000 ppm Fe^{3+} using a 10 % iron III-sulfate mixture and the pH of the mixture was adjusted to 9.5 using NaOH. The amount of NaOH was recorded to calculate the expected chemical consumption. Precipitated heavy metal hydroxides were removed by filtration. The resulting effluent was clear and sent for analysis by ICAP. Analytical results are presented in Table 3.5.1. Residual metal concentrations were Co (0.12 ppm), Fe (0.02 ppm), La(0.04 ppm), and Ni (0.09 ppm). Both uranium and zirconium were below the detection limit (< 0.01 ppm). The residual metal concentrations of Co, La, and Ni can be brought to below the ICAP detection limit if a second Fe co-precipitation step is performed (Table 3.5.1, two times Fe co-ppt).

Table 3.5.1: Residual heavy metal concentrations during waste treatment of Train I effluent.

Waste Train	Treatment	Metal	C _o (ppm)	C _f (ppm) after treatment	DF C _o /C _f
Train I (0300-DI)	Fe co-ppt	Co	250	0.120	2100
		Fe	2,000	0.015	13300
		La	250	0.038	6600
		Ni	250	0.092	2700
		U	250	<0.010	>25000
		Zr	250	<0.010	>25000
Train I (0300-DI)	two times Fe co-ppt	Co	250	<0.010	>25000
		Fe	2,000	<0.010	>200000
		La	250	<0.010	>25000
		Ni	250	<0.010	>25000
		U	250	<0.010	>25000
		Zr	250	<0.010	>25000

In a field application involving double Fe co-precipitation, the hydroxide solids from the second precipitation step consist of mainly Fe(OH)₃ and very small traces of heavy metals. The generated sludge from the second Fe precipitation can be used as the Fe³⁺ source for the first precipitation by simply dissolving these filtered solids in the acidic effluent of the blow down streams. A double precipitation dramatically enhances the metal removal efficiency without increasing the amount of sludge generation. Additionally, the chemical consumption of Fe³⁺ and NaOH are not increased. The net result is a very clean aqueous effluent having very low heavy metal effluent concentrations. The initial concentrations of Co, La, Ni, U and Zr were all reduced by factors of over ca. 25,000.

3.6 Expected Train II Waste Treatment Results

Train II waste treatment consists of co-precipitation of the heavy metals using Fe³⁺ to remove filterable solids and the bulk of the heavy metals, UV/H₂O₂ destruction of the metal chelate complexes, a second co-precipitation to recover the released metals, and cesium removal using ion resin. The blow down stream from Train II processing contains TECHXTRACT® 0300, 0200, and 0100 blends, sulfuric acid, hydrochloric acid, potassium hydroxide (KOH), dissolved metals, and DI water. Train II simulated effluent is based on the assumption that all TECHXTRACT® streams are replenished at the same time and are diluted with DI water. Train II effluent was spiked with the heavy metals Cs, Co, La, Ni, U, and Zr. A simulated Train II effluent contained the following composition of chemicals:

200 ml enhanced TECHXTRACT® 0300 (20 ml 0300, 20 ml conc. H₂SO₄, and 160 ml DI)
 200 ml enhanced TECHXTRACT® 0200 (20 ml 0200, 20 ml conc. HCl, and 160 ml DI)
 200 ml enhanced TECHXTRACT® 0100 (20 ml 0100, 3.3 g KOH, and 180 ml DI)
 600 ml DI
 Plus simulated contaminants
 1.21 g CoCl₂•6H₂O
 0.80 g LaCl₃•7H₂O
 1.21 g NiCl₂•6H₂O
 0.64 g UO₂(NO₃)₂•6H₂O
 0.83 g ZrOCl₂•xH₂O (assay 50% ZrO₂)
 1.76 g CsNO₃

Since the TECHXTRACT® 0300 solution does not contain strong organic complexers, the metal salts were first mixed in the diluted enhanced 0300 solution. The combined simulated Train II mixture contains 250 ppm each of Co, La, Ni, U, and Zr for a total heavy metal content of 1250 ppm. The Cesium loading is 1200 ppm. Strong organic complexers are introduced to the combined waste effluent through the TECHXTRACT® 0200 and 0100 blends. The enhanced TECHXTRACT® streams introduce about 1670 ppm (0100) and 170 ppm (0200) chelate (as EDTA) into the combined effluents. The total chelate of the blow down streams is about 1850 ppm. In addition to the chelates, other organic constituents are contained in the mixture. However, unlike the simulated concentrated mixture investigated earlier in Section 3, the TECHXTRACT® streams in Train II decontamination are dilute mixtures. Therefore, the chelate content is already in the range of what was achievable through chelate removal by crystallization, discussed in Section 3.2.2, and this step is not warranted.

Since the TECHXTRACT® streams are diluted using Train II processing, organic-water phase separation of the blow down stream is not required. The mixture was spiked with 2000 ppm Fe³⁺ using a 10 % iron III-sulfate solution and the pH of the mixture was adjusted to 9.5 using NaOH. The amount of NaOH was recorded for chemical consumption calculations. Precipitated heavy metal hydroxides were removed by filtration. After the first co-precipitation, the bulk of the introduced heavy metals are reduced. Additionally and significantly, the filtered water was clear, but exhibited a red-violet color. This indicated that no significant organic-water emulsion was present at this stage of processing, but the solution does contain significant concentrations of complexed metals. ICAP analysis corresponding to the different steps of Train II waste treatment are presented in Table 3.6.1. After the first precipitation, the residual metal concentrations were Co (73 ppm), Fe (29 ppm), La (0.2ppm), and Ni (27 ppm), U (1.8 ppm), and Zr (2.1 ppm). As expected, part of the metals are retained in solution by the chelating and complexing agents introduced with TECHXTRACT® 0200 and 0100.

Table 3.6.1: *Residual heavy metal concentrations during waste treatment of Train II effluent.*

Waste Train	Treatment	Metal	C _o (ppm)	C _f (ppm) after treatment	DF C _o /C _f
Train II (0300-0200-DI-0100-DI)	Fe co-ppt	Co	250	73	3.4
		Fe	2,000	29	69
		La	250	0.2	1250
		Ni	250	27	9.2
		U	250	1.8	140
		Zr	250	2.1	120
Train II (0300-0200-DI-0100-DI)	Fe co-ppt plus 10 hr UV/H ₂ O ₂ plus Fe co-ppt	Co	250	7.8	32
		Fe	2,000	1.1	1800
		La	250	<0.010	>25000
		Ni	250	20.	12.5
		U	250	<0.010	>25000
		Zr	250	<0.010	>25000
Train II (0300-0200-DI-0100-DI)	Fe co-ppt plus 16 hr UV/H ₂ O ₂ plus Fe co-ppt	Co	250	5.4	7.8
		Fe	2,000	0.05	40000
		La	250	<0.010	>25000
		Ni	250	15.	17
		U	250	<0.010	>25000
		Zr	250	<0.010	>25000

The filtrate was next subjected to UV/H₂O₂ treatment in the laboratory unit for 10 and 16 hours. After illumination, the solution was treated by a second iron co-precipitation step and filtered to remove metal hydroxides. The filtrate was analyzed for heavy metals remaining in solution. During the course of UV treatment, the violet-red color became less intense. These results are also summarized in Table 3.6.1. The residual concentrations of Co, Fe, and Ni are reduced, with Ni being the most tenacious. The data indicate that complexed heavy metal can be removed using sufficient radiation times. The residence time and size of an appropriate AOP reactor need to be determined in a pilot test using commercially available UV units.

3.7 Expected Train III Waste Treatment Results

During later testing, a third decontamination train (Train III) was identified. This train uses the TECHXTRACT® 0300 and 0200 blends. A simulated effluent was synthesized as follows:

25 ml TECHXTRACT® 0300
 25 ml Conc. H₂SO₄
 25 ml TECHXTRACT® 0200
 25 ml Conc. HCl
 Plus simulated contaminants
 250 ppm Co, La, Ni, U, and Zr
 1200 ppm Cs
 Total volume adjusted to 1 liter

The TECHXTRACT® 0200 introduces about 250 ppm chelate (primarily EDTA) and organic complexers into the Train III blow down stream. Metals remaining in solution after processing Train III effluents are summarized in Table 3.7.1. Upon co-precipitation with 2000 ppm Fe³⁺ and filtration of the metal hydroxides at pH 9.5, a clear filtrate having a light red-violet color is obtained. The metals remaining in solution were Co (18 ppm), Fe (4.8 ppm), La (0.07 ppm), Ni, (7.5 ppm), U (5.0 ppm), and Zr (1.5 ppm). After 8 hr UV/H₂O₂ treatment and a second Fe .III co-precipitation and filtration step, the residual metal concentrations were considerably reduced: Co (0.44 ppm), Fe (0.02 ppm), Ni (1.2 ppm). The concentrations of La, U and Zr were below detection (<0.010 ppm). Again for a field application, an AOP reactor needs to be properly sized using a commercial unit.

Table 3.7.1: *Residual heavy metal concentrations during waste treatment of Train III effluent.*

Waste Train	Treatment	Metal	C _o (ppm)	C _f (ppm)	DF (C _o /C _f)
'Train III' (0300-0200-DI)	Fe co-ppt	Co	250	18	32
		Fe	2,000	4.8	417
		La	250	0.069	3600
		Ni	250	7.5	33
		U	250	5.0	50
		Zr	250	1.5	170
'Train III' (0300-0200-DI)	Fe co-ppt plus 8 hr UV/H ₂ O ₂ plus Fe co-ppt	Co	250	0.44	570
		Fe	2,000	0.024	83000
		La	250	<0.010	>25000
		Ni	250	1.2	210
		U	250	<0.010	>25000
		Zr	250	<0.010	>25000

3.8 Absorption of Cesium on UOP Ion Exchange Resin

As previously discussed, neither the co-precipitation nor the chelant destruction treatments will be effective for removing soluble metals from the effluent stream. Cesium is a particular concern in many radiological decontamination applications. If discharge of a clean effluent is desired and cesium is present on the items being decontaminated, further effluent treatment will be required. Research by DOE contractors and others suggests that ion exchange is the most effective process for cesium removal.

In order to obtain experimental data for system design, EET obtained a sample of UOP's IONSIV® IE-911 cesium selective ion exchange resin. Liquid-resin equilibrium data are commonly described using a correlation proposed by Freundlich:

$$\Delta m/M = K \times C_f^{1/n}$$

where

M = mass resin, [=] gram

Δm = mass compound absorbed onto the resin, [=] mg

$$= \frac{(C_o - C_f)}{1000} \times V$$

V = volume of solution, [=] ml

C_o = initial concentration of compound in solution, [=] mg/l

C_f = final concentration of compound in solution in equilibrium with M gram resin, [=] mg/l

n = a dimensionless proportionality constant determined from equilibrium data

K = a proportionality constant determined from equilibrium data, [=] $\frac{\text{mg}}{\text{g}} (\text{mg/l})^{-1/n}$

The Freundlich isotherm is typically valid for low equilibrium concentrations of the compound of interest. Additionally, the integer 'n' is often unity. Evaluation of the equilibrium behavior of the resin at low concentrations is required to properly design a resin column to meet effluent criteria. Resin consumption is based on the resin loading in equilibrium with the feed concentration of the compound of interest. This initial concentration may correspond to a plateau region where the resin becomes saturated and a maximum resin loading, $\Delta m/M$, is observed.

In generating the isotherm data, M gram resin (ca. 20 to 800 mg) was placed in 100 ml solution (pH = 3.5) with a known initial cesium concentration (ca. 700 - 1200 ppm). The mixture was stirred for 48 hours and the solution was filtered. The aqueous phase was sent for analysis to determine the final cesium concentration. Final cesium concentrations for five

different matrices are presented in Table 3.8.1. Matrix I consisted of 1200 ppm Cs in DI water. Matrix II consisted of 1200 ppm Cs in DI water containing ca. 1000 ppm NTA and 1000 ppm EDTA. Matrix III consisted of the metal containing Train I solution after the first Fe^{3+} co-precipitation step. Note that the initial cesium concentration was lowered from 1200 ppm to ca. 750 ppm during co-precipitation. Matrix IV and matrix V consisted of metal solutions of Train III (0300-0200-DI) and Train II (0300-0200-0100-DI) effluents. After the first co-precipitation step the initial cesium concentrations were 700 and 910 ppm, respectively.

Table 3.8.1: *Cesium Isotherm Data (all experiments performed in 100 ml solution)*

Matrix	Mass resin (mg)	C_f cesium <u>mg</u> liter	Δm mg cesium	$\Delta m/M$ <u>mg cesium</u> g resin
I DI C_o cesium = 1200 ppm	20.1	1100	10	498
	57.6	1000	20	347
	99.0	900	30	303
	291.2	630	57	198
	791.6	34	117	148
II EDTA + NTA + DI C_o cesium = 1200 ppm	32.0	440	76	2375
	61.4	950	25	407
	95.0	900	30	315
	311.7	590	61	198
	814.8	17	118	145
III 0300 + DI C_o cesium = 750 ppm	29.1	720	3	103
	47.7	660	9	189
	110.9	520	23	207
	306.9	270	48	156
	802.4	88	66.2	83
IV 0300 + 0200 + DI C_o cesium = 700 ppm	25.3	800	***	***
	54.9	760	***	***
	101.4	710	***	***
	300.0	580	12	40
	790.6	280	42	53
V 0300 + 0200 + 0100 + DI C_o cesium = 920 ppm	28.6	910	1	35
	59.6	820	10	168
	102.6	690	23	224
	287.6	280	64	223
	803.3	290	63	78

The resin loading and final cesium concentrations were plotted on a double logarithmic scale (Figure 3.8.1). The data scatter widely and did not exhibit behavior expected from a Freundlich type isotherm in the concentration ranges investigated (ca. 10 - 1000 ppm). In this

concentration range, the resin apparently is saturated at a maximum cesium loading of approximately 150 mg cesium / gram resin for effluents containing TECHXTRACT® solutions.

Figure 3.8.1 *Resin loading as a function of equilibrium cesium concentrations for different waste water matrices.*

To estimate the resin consumption and cost during treatment of Train I and Train II effluents, a cesium concentration of 100 ppm and a maximum resin loading of 150 mg cesium / g resin is assumed. The resin costs ca. \$150/lb. The resin cost associated with treating 750 gallons of Train I effluent is calculated to be:

$$\begin{aligned}
 \text{cesium to be removed per batch} &= 750 \frac{\text{gal.}}{\text{batch}} \times 3.8 \frac{\text{l}}{\text{gal.}} \times 100 \frac{\text{mg cesium}}{\text{l}} \\
 &= 285,000 \frac{\text{mg cesium}}{\text{batch}} \\
 \text{mass UOP resin required per batch} &= 285,000 \frac{\text{mg cesium}}{\text{batch}} \times \frac{\text{g resin}}{150 \text{ mg cesium}} \times \frac{1 \text{ lb}}{454 \text{ g}} \\
 &= 4.2 \frac{\text{lb}}{\text{batch}} \\
 \text{resin cost per batch} &= 4.2 \frac{\text{lb}}{\text{batch}} \times \frac{\$150}{\text{lb}} \\
 &= \$630/\text{batch}
 \end{aligned}$$

Similarly, for processing 1875 gal. Train II effluent, the amount of resin and resin cost is:

$$\begin{aligned}
 \text{cesium to be removed} &= 1875 \frac{\text{gal.}}{\text{batch}} \times 3.8 \frac{\text{l}}{\text{gal.}} \times 100 \frac{\text{mg cesium}}{\text{l}} \\
 &= 712,500 \frac{\text{mg cesium}}{\text{batch}}
 \end{aligned}$$

$$\begin{aligned}
 \text{mass UOP resin required} &= 712,500 \frac{\text{mg cesium}}{\text{batch}} \times \frac{\text{g resin}}{150 \text{ mg cesium}} \times \frac{1 \text{ lb}}{454 \text{ g}} \\
 &= 10.5 \frac{\text{lb}}{\text{batch}} \\
 \text{resin cost} &= 10.5 \frac{\text{lb}}{\text{batch}} \times \frac{\$150}{\text{lb}} \\
 &= \$1,575/\text{batch}
 \end{aligned}$$

4.0 Surface Decontamination Experiments Using Radionuclides

In Section 2, treatment strategies were developed to remove surrogate contaminants from SIMCON coupons. Decontamination trains were identified using sonication with dilute, enhanced TECHXTRACT® formulations to reduce the contamination on SIMCON coupons by DF values approaching 100. The optimized systems from the surrogate decontamination experiments were the basis for EET's continuation with radioactively contaminated samples. This section discusses the results of this testing.

4.1 Subcontract Testing Facility - Alaron Corporation

All experiments involving radioactively contaminated materials were performed at Alaron Corporation's facility in Wampum, PA. This is a full-scale, fixed base radiological decontamination facility. Alaron processes contaminated materials - mostly metals - for nuclear utilities, private industry, and the DOE. The majority of Alaron's decontamination involves blasting or other physical methods of removing surface contamination. Alaron's facility includes a small testing laboratory, which is where the work for this contract was performed.

Alaron was chosen as a subcontractor to EET for this project through a competitive selection process. EET's evaluation criteria included cost, technical capabilities, and ability to meet the project schedule requirements. An added advantage of Alaron as the subcontractor was their ability to supply radioactively-contaminated samples. Coupons were cut from Alaron's inventory of materials that were waiting treatment or that had been treated unsuccessfully. This allowed EET to experiment with a wide variety of substrates and isotopes.

4.2 Overview of Experiments

A total of 88 experiments with radioactively contaminated samples were performed at Alaron's facility. A synopsis of the substrates and the primary contaminants in these experiments is shown in Table 4.2.1, and a more detailed description is provided in Appendix B. The mass of the samples typically ranged from ca. 50 to 400 g per sample, and the largest dimension was about 2". The majority of the contamination on the samples was "fixed." Several of the sample groups had been unsuccessfully treated by Alaron using blasting and chemical techniques.

Testing was performed using sonication and TECHXTRACT® blends at 43°C. The bench scale Crest ultrasonic units, described in Section 2.3, were shipped to Alaron for this phase of the contract. Testing was performed by an Alaron technician, under the direction of one of EET's researchers. Alaron personnel were also responsible for all radiological surveys to determine experimental results.

The radiation (β - γ) emitted from a sample was measured by an integral counter and the counts per minute (cpm) were calculated. The background cpm was subtracted to obtain a net cpm emitted by a sample. The cpm were converted to dpm per 100 cm² using the probe efficiency

($\eta = 20\%$) and normalizing the 16 cm² probe area to 100 cm². All experiments used the same probe, integral counter and a fixed geometry of the sample with respect to the probe.

Table 4.2.1: *Substrates and primary radionuclide contamination in Alaron decontamination testing.*

Substrate	Primary Isotopes
Copper tubing	Fe-55, Co-60, Ni-63, Cs-137
Titanium tubing	Cr-51, Fe-55, Cs-134, Cs-137
Aluminum	U-234, U-238
Carbon steel	Fe-55, Co-58, Ni-63, Cs-134, Cs-137
Concrete	Co-60, Cs-137
Stainless steel fuel rack	Fe-55, Co-58, Co-60, Ni-63

As with the SIMCON coupons, the objective of the experiments performed at Alaron was to identify TECHXTRACT® based decontamination train(s) that could achieve the best decontamination performance. Furthermore, an absolute target of reducing radioactivity to “free release” levels could be set for these experiments. Per DOE Order 5400.5 and Nuclear Regulatory Commission (NRC) Regulation 1.86, free release for fixed β - γ contamination requires levels less than 5000 dpm/100 cm². DF values of approaching 1,000 were measured in several experiments. The remaining contaminants on a variety of metal substrates were often reduced to background levels after processing. Even the materials that had not been successfully decontaminated by Alaron were treated to below free release levels in many experiments. The details of this testing, including several new insights, are discussed by substrate in the following sections. A complete table of all Alaron decontamination testing is given in Appendix C.

4.3 Copper and Titanium Tubing, Carbon Steel, and Stainless Steel

The samples of copper (Cu) tubing, titanium (Ti) tubing, carbon steel, and stainless steel were successfully decontaminated using Train I processing (10% 0300 plus 10% H₂SO₄), as shown in Table 4.3.1. The Cu and Ti tubing sections were lightly contaminated, and exhibited a slight scale deposit. Contamination levels on the samples were up to 6700 (Cu) and 8000 (Ti) dpm/100 cm². After processing seven Cu sections concurrently in a 10% TECHXTRACT® 0300 and 10% H₂SO₄ solution for 7.5 min., the average initial contamination was reduced from 5444 dpm/100 cm² to below background measurements (Exp. 13). Similarly, after processing seven sections of Ti tubing in a 10% TECHXTRACT® 0300 plus 10% H₂SO₄ solution for 15 min., the average initial radiation was reduced from 2500 dpm/100 cm² to below background levels (Exp. 23). Multiple sections of lightly contaminated carbon steel were also processed concurrently to below background levels (Exp. 27) during 7.5 min. processing in a 10% TECHXTRACT® 0300 and 10% H₂SO₄ solution. These results show that

short sonication times and chemical reuse are possible, depending on the desired level of contaminant removal.

Table 4.3.1: *Summary of Decontamination Experiments on Copper & Titanium Tubing, Carbon, and Stainless Steel*

Exp.	Initial (C _o) (dpm/100 cm ²)	Final (C _f) (dpm/100 cm ²)	Comments*
1	2,888	13	15 min
3	2,313	116	15 min
7	2,369	63	7.5 min
10	3,591	37	7.5 min
13	5,444	-147	7.5 min; Multiple samples
18	2,631	50	15 min
23	2,574	-279	15 min; Multiple samples
24	14,206	944	15 min
25	37,513	959	NiCl ₂ added
27	1,193	-419	7.5 min; Multiple samples
35	45,144	822	0200 and 0300
39	22,525	3,644	15 min
46	907,256	131,450	Train II, sample used as missile target
55	2,666	237	0300 - 0200
56	5,338	169	0300 - 0200
57	120,795	-431	Modified Train III, filter screen
59	25,225	2,202	Modified Train III, machine part

* All experiments use Train I process unless otherwise noted.

4.4 Aluminum

Decontamination of aluminum (Al) using TECHXTRACT® blends was successful using Train III processing (30 min. sonication in 10% TECHXTRACT® 0300 plus 10% H₂SO₄ followed by 30 min. in 10% TECHXTRACT® 0200 plus 10% HCl). One side of the sample tested in Exp. 45 was reduced from 35000 DPM to background levels, and DF values over 100 were measured on the second side. Exp. 45 demonstrated that high DF values can be achieved using TECHXTRACT® blends. However, during processing in the 0200 bath a large amount of gas generation, presumably hydrogen evolution, was observed. Additionally, the gas

generation was accompanied by severe foaming of the 0200 bath. Similar foaming was observed when the aluminum sections were processed in an 0300 blend containing NiCl_2 (Exp. 58).

Table 4.4.1: *Summary of Decontamination Experiments on Aluminum*

Exp.	Initial (C_o) (dpm/100 cm^2)	Final (C_f) (dpm/100 cm^2)	Comments
19	6,578	2,234	Train I; 15 min; Repeat 2x
20	3,372	1,284	Enhanced 0200; 15 min; 2x
45	28,806	66	Train III
58	60,405	202	Modified Train III

4.5 Stainless Steel Tubing

Stainless steel heat exchanger tubing sections were successfully treated using Train I processing (10% 0300 plus 10% H_2SO_4), as shown in Table 4.5.1. The tubing samples were obtained from a heat exchanger that Alaron was decontaminating using another chemical technique. The gross contamination was readily removed by Alaron's process, but the tubing sections still had fixed radiation levels of up to 200,000 dpm/100 cm^2 . The residual contamination was reduced to free release using Train I processing. Multiple samples of the heat exchanger coupons were processed in 300 ml of the same 0300 solution (beginning with Exp. 66). The results are presented in Figure 4.5.1A and 4.5.1B. The average mass and surface area per coupon were 39 g and 3.6 in^2 , respectively. The data are presented as a function of (A) the total surface area processed and (B) the total mass processed. The open circles are the initial contamination levels (dpm/100 cm^2), averaged over both outside sections (average of top and bottom). The closed circles were the final levels after Train I processing. The coupons were run in batches of three. After processing 30 sections, free release limits were achieved on all sections but on sample #1, which exhibited some scratches on the surface from the removal of a baffle plate. This one sample was treated in an 0300 plus NiCl_2 solution and the contamination was almost completely removed (Exp. 66). Final average contamination was 32 dpm/100 cm^2 . In addition to removing contamination from the outside area of the heat exchanger coupons, the contamination was removed from the inside as well. One sample from each set of three coupons was split axially, and the activity of the inside surface was measured. The final contamination levels on the inside surface area of the samples were below free release limits for all samples. The graphs show that over 100 in^2 material representing about 1250 g material was successfully processed in the same 300 ml solution. No significant deterioration in bath performance was observed after processing the 30 specimens. Additionally, very little mass (<ca. 0.01 g per 39 g sample) was lost during processing.

Table 4.5.1: *Summary of Decontamination Experiments on Stainless Steel Tubing*

Exp.	Initial (C_o) (dpm/100 cm²)	Final (C_f) (dpm/100 cm²)	Comments*
14	2,575	-50	---
15	3,281	69	10 min
66	87,777	483	Multiple samples
68	35,582	212	Multiple samples, cont. of #66
71	17,832	212	Multiple samples, cont. of #68
73	13,017	426	Multiple samples, cont. of #71
75	18,345	327	Multiple samples, cont. of #73
79	16,591	927	Multiple samples, cont. of #75
80	20,354	833	Multiple samples, cont. of #79
86	13,802	1,589	Multiple samples, cont. of #80
87	23,253	615	Multiple samples, cont. of #86
88	13,581	1,034	Multiple samples, cont. of #87

* All experiments use Train I process unless otherwise noted.

Figure 4.5.1 Decontamination of stainless steel heat exchanger tubing sections using TECHXTRACT® 0300 blend and sonication as a function of (A) total surface area processed and (B) total mass processed.
 Legend: (○) initial contamination; (●) final contamination.

4.6 Lead

Lead is too soft to be processed using conventional blasting techniques. As a result, Alaron decontaminates the lead manually. Several lead samples were successfully decontaminated using dilute TECHXTRACT® 0300 solutions, without sulfuric acid. Sulfuric acid in the enhanced TECHXTRACT® 0300 is detrimental to the performance of this solution. Lead sulfate is highly insoluble, and treatment in the Train I process (TECHXTRACT® 0300 with sulfuric acid) caused the formation of a protective lead sulfate layer. The DF values on lead samples in an 0300 solution using 10% H₂SO₄ were typically below 5 (Exp. 26). All lead treatment results are shown in Table 4.6.1.

Table 4.6.1: *Summary of Decontamination Experiments on Lead*

Exp.	Initial (C ₀) (dpm/100 cm ²)	Final (C _f) (dpm/100 cm ²)	Comments
26	44,775	8525	0300 - 3x plus 0200 - 2x
43	1,650	194	Train I; 15 min, Repeat 3x
44	124,488	862	Train II
60	401,281	1,047	Train III
61	1,200,313	2,547	Modified Train III, plus 2 nd 0200
62	2,710,281	4,250	Modified Train III, plus 2 nd 0200
63	2,757,484	43,375	Modified Train III, plus 2 nd 0200
64	384,803	18,584	Train III, no H ₂ SO ₄
65	571,555	2,023	Train III, no H ₂ SO ₄
67	129,656	2,023	20% 0300, no H ₂ SO ₄ , multiple sample
69	258,766	1,977	20% 0300, no H ₂ SO ₄ , multiple sample
70	42,820	1,430	20% 0300, no H ₂ SO ₄ , multiple sample
72	35,484	3,031	20% 0300, no H ₂ SO ₄ , multiple sample
74	33,164	6,789	20% 0300, no H ₂ SO ₄ , multiple sample
76	19,203	1,406	20% 0300, no H ₂ SO ₄ , multiple sample

Figure 4.6.1 shows experimental results during the processing of multiple lead samples in 300 ml of the same 0300 solution (20% concentration) without sulfuric acid (beginning with Exp. 64). The average mass and surface area per lead sample were 430 g and 11.5 in², respectively. The results are presented as a function of the total surface area processed (Figure 4.6.1A) and as a function of the total mass processed (4.6.1B). DF values

approaching 100 were obtained in some experiments. Lead is highly malleable, therefore, any sharp impact on the surface during handling can cover up a contaminated area such that chemical decontamination is impossible. About 3500 g lead having a total surface area of about 100 in² was decontaminated in the same 300 ml solutions. Unlike the heat exchanger samples, the lead samples did lose a modest amount of weight during processing (typically about 1 g weight loss per 430 g sample).

Figure 4.6.1 Decontamination of lead coupons using TECHXTRACT® 0300 blend (20%, no H₂SO₄) and sonication as a function of (A) total

surface area processed and (B) total mass processed.

Legend: (○) initial contamination; (●) final contamination.

4.7 Stainless Steel Fuel Rack

Contamination on stainless steel fuel rack samples was at much higher concentrations and was tenacious. Neither Train I nor Train II processing was successful in decontaminating these samples to free release levels. However, a modified Train III process successfully decontaminated samples to free release levels. The modified Train III consists of the following sequential baths: (1) 10% TECHXTRACT® 0300 plus 10% H₂SO₄; (2) 10% TECHXTRACT® 0200 plus 10% HCl; (3) 10% TECHXTRACT® 0300 plus 10% H₂SO₄ plus 3% NiCl₂; (4) Rinse in DI water. Typical bath times were 30 minutes at 43°C. The coupons contained a large amount of slag and sputter deposits making complete contaminant removal difficult if not impossible. These samples were considered by Alaron to be the most difficult to decontaminate. Results from all of the tests involving fuel rack samples are shown in Table 4.7.1.

Table 4.7.1: *Summary of Decontamination Experiments on Stainless Steel Fuel Rack*

Exp.	Initial (C _o) (dpm/100 cm ²)	Final (C _f) (dpm/100 cm ²)	Comments
2	265,650	9,534	Train I
4	248,047	11,294	Train I, 2x
5	265,650	8,209	Train I, 3x
6	223,109	9,041	Train II
8	336,444	14,844	Enhanced 0200, 2x
9	326,731	77,344	Enhanced 0100, 2x
11	248,047	4,863	Train I, 5x
12	316,503	17,603	Train I, 3x
16	333,469	8,750	Train I, 2x
17	245,806	8,481	Train I, 2x
29	247,922	2,806	Train I, NiCl ₂ added
30	254,509	2,619	0200 - 0300 plus NiCl ₂
31	272,184	2,825	Modified Train III
32	263,800	-91	Train I, 3x, NiCl ₂ added
33	245,681	5,975	Train I, 3x, NiCl ₂ added
36	285,200	466	0200 - 0300, plus NiCl ₂

Exp.	Initial (C_o) (dpm/100 cm²)	Final (C_f) (dpm/100 cm²)	Comments
37	316,209	431	0200 - 0300, plus NiCl ₂
38	285,681	1,053	0200 - 0300, plus NiCl ₂
40	299,844	1,969	Modified Train III
41	246,194	-128	Modified Train III
42	261,697	103	Modified Train III
47	226,529	2,091	Train III, HCl
48	313,992	94	Train III, HCl
49	342,895	1,094	Train III, HCl
50	733,156	1,297	Train III
51	153,732	1,982	Train III, HCl
52	60,422	7,875	Train III, HCl
53	447,422	19,703	Train III, HCl
54	860,016	35,672	Train III, HCl
77	261,484	766	Train III
78	256,797	547	Train III, cont. #77
81	274,500	14,266	Train III, cont. #78
82	258,000	1,094	Train III, cont. #81
83	219,859	2,359	Train III, cont. #82
84	324,609	19,156	Train III, cont. #83
85	204,281	30,547	Train III, cont. #84

The Train III process was also evaluated for a multiple sample run (beginning with Exp. 77). Results are presented graphically in Figures 4.7.1A and B. The average mass and surface area per fuel rack sample were 260 g and 15.2 in². The open circles are the initial radiation per coupon. The open squares are the radiation after 30 min. treatment in the 0300 bath (10% 0300 + 10% H₂SO₄). The open triangles are after treatment in the 0200 bath (total processing: 30 min. in the 0300 solution followed by 30 min. in 10% 0200 + 10% HCl). The closed circles are after an additional 30 min. in a 10% 0300 + 10% H₂SO₄ + 3% NiCl₂ (total processing time 90 min.). The 0300 NiCl₂ bath was required to obtain sufficient decontamination values for free release of the material. Weight losses were about 1-2 g per

260 g coupon. The performance of the solutions deteriorated after processing about 80 in² (1250 g) material.

Figure 4.7.1 Decontamination of stainless steel fuel rack samples using TECHXTRACT® blends and sonication as a function of (A) total surface area processed and (B) total mass processed.
 Legend: (○) initial contamination; (△) after processing 30 min. in 10% 0300 plus 10% H₂SO₄; (□) after 30 min. in 10% 0300 plus 10% H₂SO₄, followed by 30 min. in 10% 0200 plus 10% HCl; (●) final contamination after 30 min. in 10% 0300 plus

10% H₂SO₄, followed by 30 min. in 10% 0200 plus 10% HCl, followed by 30 min. in 10% 0300 plus 10% H₂SO₄ plus 3% NiCl₂.

4.8 Conclusions:

- 1.) Cu tubing, Ti Tubing, carbon steel, and stainless steel samples were successfully decontaminated using Train I processing based on 10% TECHXTRACT® 0300 plus 10% H₂SO₄ and sonication times as short as 7.5 min. Shorter decontamination times are possible.
- 2.) Aluminum can be decontaminated using Train III processing in 10% 0300 plus 10% H₂SO₄ followed by 10% 0200 plus 10% HCl. Severe foam generation and gas evolution was observed during processing in the 0200 solution.
- 3.) Stainless steel tubing was successfully decontaminated using Train I processing based on 10% 0300 plus 10% H₂SO₄.
- 4.) Lead samples were successfully decontaminated using a 20% 0300 solution without sulfuric acid. The presence of sulfuric acid is detrimental to the performance of the 0300 solution during lead processing.
- 5.) Stainless steel fuel rack was successfully decontaminated in a modified Train III process: 10% 0300 plus 10% H₂SO₄, followed by 10% 0200 plus 10% HCl; followed by 10% 0300 plus 10% H₂SO₄ plus 3% NiCl₂.
- 6.) Despite the fact that many of the samples in this phase of the project were considered to be difficult or impossible to decontaminate, TECHXTRACT® based treatment trains were successfully tested for each scenario.

5.0 Concrete Rubble Decontamination

Another objective of EET's contract was to evaluate the effectiveness of the TECHXTRACT® decontamination train(s) for the treatment of contaminated concrete rubble. As in the metal decontamination testing, the initial evaluation was done using non-radioactive surrogates. The rubble was prepared from cement and sand sinter blocks (2' x 2' x 2" garden stepping stones) that did not contain gravel. Rubble samples prepared from these block were impregnated with lanthanum (La), cobalt (Co), nickel (Ni), zirconium (Zr), and cesium (Cs). Section 5.1 discusses rubble preparation, impregnation, and metals analysis. Section 5.2 presents experimental results and conditions. A limited number of tests with radioactively contaminated concrete samples was performed at Alaron. These results are discussed in Section 5.3.

5.1 Rubble Preparation, Impregnation, and Metals Analysis

'Rubble' was prepared from non-gravel containing cement and sand sinter blocks. The blocks were mechanically broken into debris and screened. Debris having a size fraction retained between 3/8" and 3/4" mesh screens were used in the experiments. This rubble was washed with DI water and dried at 120°C.

Two batches of metal impregnated rubble were prepared. Batch I rubble was prepared by contact with a 1000 ppm Cs and 1000 ppm Zr solution. The Batch I metal solution was prepared from cesium nitrate and zirconyl chloride. Batch II rubble was prepared by contact with a 1000 ppm Co, 1000 ppm La, and 1000 ppm Ni solution. The Batch II metal solution was prepared from the respective metal chloride salts. During impregnation, ca. 250 g rubble was contacted with 1 liter metals solution for about 16 hours. Afterwards the aqueous phase was decanted and the rubble was lightly washed with DI water and then dried at 120°C.

The metals content of the untreated (after impregnation) and treated rubble was obtained by acid digestion and ICAP analysis. A sample of the rubble was finely crushed. An accurately weighed sample of the crushed rubble (ca. 2.5g) was digested in nitric acid (HNO₃) and hydrochloric acid (HCl) in accordance with EPA method 3010. The solids were filtered and washed. Finally, the filtrate was brought to a known volume. ICAP analysis of the metals content in the filtrate was performed by an independent laboratory and the metal loading (mg metal per kg) of the rubble was calculated. Cesium analysis of Batch I impregnated rubble showed an initial loading of less than $8 \frac{\text{mg}}{\text{kg rubble}}$. This low loading could be the result of either poor Cs adsorption onto the debris during impregnation or due to calcium (Ca) interference during ICAP analysis. Ca in the filtrate is present in very high concentrations and may mask Cs signals in the analytical method used. Therefore, Batch I experiments #3 - #9 were not analyzed for Cs loading.

5.2 Rubble Decontamination - Experimental Results and Conditions

Decontamination experiments were performed by treating about 15g impregnated rubble in 300 ml solution at 43°C using sonication. TECHXTRACT® blends were applied in two or five

step treatment trains. Test conditions and results are summarized in Tables 5.2.1 for Batch I rubble and 5.2.2 and Batch II rubble.

Table 5.2.1: *Metal removal from Batch I (Cs and Zr) impregnated rubble using TECHXTRACT® based decontamination trains. All experiments were performed at 43°C and under sonication unless otherwise noted.*

Exp.	Decontamination Train Conditions	Time (min.)	Metal	Final Loading <u>mg metal</u> <u>kg rubble</u>	Removal	Decon Factor
---	initial metal loading	---	Cs Zr	<8 180	---	---
1	1.) 10% 0300 2.) DI	45 15	Cs Zr	< 8 40	---	---
2	1.) 10% 0300 + 10% H ₂ SO ₄ 2.) DI	45 15	Cs Zr	<8 50	---	---
3	1.) 10% 0300 2.) 10% 0200 + 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5 5.) DI	45 45 45 45 45	Cs Zr	---	---	---
4	1.) 10% 0300 2.) 10% 0200 3.) DI 4.) 10% 0100 5.) DI	45 45 45 45 45	Cs Zr	---	---	---
5	1.) 10% 0200 2.) DI	45 15	Cs Zr	---	---	---
6	1.) 10% 0200 + 10% HCl 2.) DI	45 15	Cs Zr	---	---	---
7	1.) 10% 0100 2.) DI	45 15	Cs Zr	---	---	---
8	1.) 10% 0100 @ pH 13.5 (KOH) 2.) DI	45 15	Cs Zr	---	---	---
9	1.) 10% 0300 2.) DI	45 15	Cs Zr	---	---	---

Table 5.2.2: *Metal removal from Batch II (Co, La, and Ni) impregnated rubble using TECHXTRACT® based decontamination trains. All experiments were performed at 43°C and under sonication unless otherwise noted.*

Exp.	Decontamination Train Conditions	Time (min.)	Metal	Final Loading <small>mg metal kg rubble</small>	Removal	Decon Factor
---	initial metal loading	---	Co La Ni	224 920 156	--- --- ---	--- --- ---
1	1.) 10% 0300 2.) DI	45 15	Co La Ni	50 136 38	78% 85% 76%	4.5 6.7 4.1
2	1.) 10% 0300 + 10% H ₂ SO ₄ 2.) DI	45 15	Co La Ni	95 202 66	58% 78% 58%	2.4 4.6 2.4
3	1.) 10% 0300 2.) 10% 0200 + 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5 5.) DI	45 45 45 45 45	Co La Ni	31 27 28	86% 97% 82%	7.2 34 5.6
4	1.) 10% 0300 2.) 10% 0200 3.) DI 4.) 10% 0100 5.) DI	45 45 45 45 45	Co La Ni	3 7 6	99% 99% 96%	75 131 26
5	1.) 10% 0200 2.) DI	45 15	Co La Ni	140 620 116	38% 33% 26%	1.6 1.5 1.3
6	1.) 10% 0200 + 10% HCl 2.) DI	45 15	Co La Ni	44 121 37	80% 87% 76%	5.1 7.6 4.2
7	1.) 10% 0100 2.) DI	45 15	Co La Ni	181 576 144	19% 37% 8%	1.2 1.6 1.1
8	1.) 10% 0100 @ pH 13.5 (KOH) 2.) DI	45 15	Co La Ni	52 161 40	77% 83% 74%	4.3 5.7 3.9
9	1.) 10% 0300 2.) DI	45 15	Co La Ni	73 150 60	67% 84% 62%	3.1 6.1 2.6

Rubble treated using full strength TECHXTRACT® 0300 (no dilution) was aggressively attacked by the chemicals and resulted in excessive foam formation. Treatment using concentrated TECHXTRACT® blends was therefore not pursued further. TECHXTRACT® solutions were tested individually using 1:10 DI diluted formulations. Experiments investigating the effectiveness of enhanced 1:10 TECHXTRACT® solutions were performed using 10% H₂SO₄ in the 0300 formulation, 10% HCl in the 0200 formulation, and pH adjustment to ca. 13.5 using KOH in the 0100 formulation.

Treatment in the 1:10 DI diluted 0300 plus 10% H₂SO₄ based train caused some foaming of the solution during treatment. (Exp. #2, Tables 5.2.1 and 5.2.2). Subsequent experiments used unenhanced 1:10 diluted 0300 formulations. Treatment in a 1:10 diluted 0200 plus 10% HCl based trains caused severe surface attack. The surface layer was softened and crumbled leading to partial disintegration of the rubble.

Rubble decontamination results using the full five train treatment sequence are shown in experiments #3 and #4 of Tables 5.2.1 and 5.2.2. The total treatment time in these experiments was 225 min., with 45 minutes per bath. Removal efficiencies ranging from 82% to 99% were achieved. Individual decontamination factors for Exp. #4 were 12 (Zr), 75 (Co), 131 (La), and 26 (Ni).

The effectiveness of individual TECHXTRACT® based treatment trains were investigated in Exp. #1, #2, and #5-#9 of Tables 5.2.1 and 5.2.2. The total treatment time in these experiments was 60 min. The best results were observed in experiments #6 and #8. The treatment train used in Exp. #6 was based on a 10% 0200 solution enhanced with 10% HCl. The corresponding DF factors were 5.1 (Co), 7.6 (La), 4.2 (Ni), and 4.4 (Zr). The treatment train used in Exp. #8 was based on a 10% 0100 solution pH adjusted to ca. 13.5 with KOH. The DF values for Exp. #8 were 4.3 (Co), 5.7 (La), 4.2 (Ni), and 3.9 (Zr).

5.3 Decontamination of Radioactively Contaminated Concrete Rubble

Alaron was able to provide a small amount of radioactively contaminated concrete rubble for testing as part of EET's overall program at their facility. The primary isotopes on this material were Co-60 and Cs-137. The samples were all from one source, were limited in quantities, and had relatively low levels of contamination. These factors limited the value of the data obtained in this portion of the project.

Results of the testing with radioactively contaminated concrete rubble are shown in Table 5.3.1. Experiments were conducted in each of the individual TECHXTRACT® solutions and in the sequential Train II process. DF values ranged from 1.1 to 2.4, with the best results obtained in the TECHXTRACT® 0100 solution and the Train II process.

These values are lower than those observed with the surrogates, and much lower than the results for metal decontamination. Concrete would be expected to be more difficult to decontaminate due to its porosity, but these results were not consistent with EET's projections or past experience. Given the limited amount of data, the concrete decontamination results

are considered to be inconclusive. Further evaluation with a broader range of samples (e.g., higher contamination levels, other isotopes) would be needed to better address this topic.

Table 5.3.1: *Summary of Decontamination Experiments on Concrete*

Exp.	Initial (C_o) (dpm/100 cm²)	Final (C_f) (dpm/100 cm²)	Comments
21	5550	4869	10% 0300
22	7225	5188	Enhanced 0200
28	8388	3613	Enhanced 0100, Repeat 3x
34	3513	1456	Train II

6.0 Full-Scale System Engineering Design

This section describes the key design parameters and costs for a full-scale decontamination and effluent treatment system. The design is based on the bench scale results discussed in the previous sections of this report.

The system can be scaled up to virtually any size. In actual application, the dimensions of the ultrasonic baths will be set by the largest item(s) to be decontaminated. If these items are extremely large, or if there are a small number of large items mixed in with a large volume of smaller pieces, size reduction may be used to minimize the required bath size.

Since a specific full-scale field application has not been selected, EET picked a reasonable scale-up scenario for the system design. Ultrasonic baths with a 75 gal. (10 cu ft) capacity and interior dimensions of 3 ft. x 2 ft. x 20 in. were used for design and cost modeling. This size is small enough to be an off-the-shelf unit, but large enough to handle reasonable size batches of scrap metal or debris. Large items - such as I-beams or large pieces of equipment - would require size reduction or larger baths. EET did confirm that customized ultrasonic baths could be manufactured in a wide range of sizes.

Section 6.1 introduces Train I configuration and processing, and Section 6.2 estimates expected Train I chemical consumption. Train I corresponds to the best performance from the majority of EET's tests, and is the simplest and lowest cost system. Section 6.3 shows Train II configuration and processing, and Section 6.4 details the corresponding chemical consumption. Train II represents the highest capital and operating cost case, due to the use of 5 different baths (3 chemical, 2 rinse) and more complex effluent treatment requirements.

6.1 Train I Configuration

Decontamination of radioactively contaminated metal and treatment of spent TECHXTRACT® chemicals will be performed in a batch operation. A schematic of the system is given by Figure 6.1.1. TECHXTRACT® baths will be replenished as needed from 55-gal. product drums (1, 2) and a DI water unit (3). Make up chemicals for Train I processing are concentrated TECHXTRACT® 0300 and sulfuric acid. The DI unit is sized to deliver up to 300 gal. per 8 hr day of 18 MΩ water.

The make up solution for the decontamination bath is formulated by mixing 7.5 gallons TECHXTRACT® 0300 and 7.5 gallons conc. H₂SO₄ with 60 gallons DI water (4). This mixture is transferred by a motorized barrel pump from the make up tank to the 0300 ultrasonic bath (6). DI water can be directly added to the ultrasonic rinse bath as needed. An overhead conveyer (5) will be used to transport baskets of scrap metal through the decontamination train. The conveyer will be operated via a hand held controller.

Figure 6.1.1 - Train I Decontamination and Effluent System Schematic

A diaphragm pump transfers the spent chemicals and rinse solution from the treatment baths to one of two stirred holding tanks (7 and 8). All holding tanks are 1000 gal volume and are sized to contain up to five Train I discharge volumes (750 gal.). Spent solutions are chemically treated to precipitate dissolved heavy metals/radionuclides as metal hydroxides. The metals in solution are expected to be of similar nature and diversity as metals found in effluents of plating industry waste-water and those encountered from the Alaron field tests. Co-precipitation using iron III can reduce the solubility of metal hydroxides by several orders of magnitude. This treatment approach allows for efficient metal removal in one vessel at one pH. In the absence of complexing agents, the residual metal concentrations of species forming metal hydroxide precipitates can be brought to below ICAP detection (0.01 ppm). Adjustment of the effluent pH is achieved by addition of 50% NaOH (9). Addition of Fe^{3+} will be via an iron III sulfate solution (10% Fe^{3+} , 10). If the effluent contains chromium in the plus 6 oxidation state, iron may be added as Fe^{2+} to reduce Cr^{6+} to Cr^{3+} . Iron and NaOH

will be supplied in 55 gallon barrels and delivered to the holding tanks by motorized barrel pumps.

After addition of iron and final adjustment of the pH of the holding tank to ca. 9.5, a solid liquid separation step is required to remove filterable solids and precipitated heavy metal hydroxides (11, 12, and 13). Figure 6.1.1 shows a filter press for this separation. The press is sized to hold sludge from processing one batch of waste water. The solids removed from the filter press will be radioactive and must be disposed of properly (13). The filtered water will pass through a bank of in-line filters (14) to remove any fines that are not retained by the filter press. Depending upon the level of radioactivity of the incoming metals, the filter press may need to be replaced with a device capable of continuous operation to avoid prolonged exposure to the concentrated radioactive sludge.

Depending on the specific radionuclides and the effluent discharge requirement, further processing after the in-line filters may not be necessary. If the effluent contains insoluble metal hydroxides, such as cesium (Cs), and further removal is required, the system will continue as shown.

Cesium can be removed by an ion exchange unit (19) available from vendors such as UOP or 3M. After filtration, the waste water is routed to hold tank III (15). The pH of the waste stream in hold tank III needs to be adjusted by the addition of 10% H_2SO_4 (16) by a feed back control system to optimize the performance of the resin (pH between 2 and 4 for the UOP resin). The H_2SO_4 is supplied from 55 gal. drums. Prior to the resin bed, the wastewater is micro filtered to remove particulates to $0.1\mu\text{m}$ using a second in-line filtration system (18). After passing through the resin bed, the processed waste water is collected in the Whoopsie tank (20) where the pH is adjusted by a feed back control system (21) by dosing 10% NaOH from a 55 gal. drum to meet discharge regulations. The treated effluent should then be ready for final testing and discharge.

In summary, the major sections of a Train I processing system consist of:

- Chemical supply tanks
- Make up tanks
- Ultrasonic baths (2)
- Hold and treatment tanks for heavy metal precipitation
- Filtration unit(s)
- Cs removal unit (optional)
- Final pH adjustment (optional)

The major equipment items and estimated costs are listed in Table 6.1.1. The costs of the individual items add up to \$176,000, and do not include delivery, installation, or mark-up.

Table 6.1.1 *Major Equipment Cost for Train I Decontamination and Waste Treatment*

Item #	Description	Estimated Cost
1	drum pump for 0300	\$600
	barrel mixer	\$300
2	drum pump for conc. H ₂ SO ₄	\$600
3	DI Supply	\$16,000
4	drum pump for DI	\$475
	0300 make up tank, 100 gal.	\$400
	mixer & mount	\$425
5	drum pump	\$475
	Decon line conveyer	\$10,000
6	two 75 gal Crest U.S. baths @ 18 K ea.	\$36,000
7	SS liners (@ 10% of units)	\$3,600
	3/4" diaphragm pump	\$1,500
	Eff. Hold I (1000 gal. support w/conical bottom)	\$2,000
8	3 HP tank mixer	\$6,000
	Eff. Hold II (1000 gal. support w/conical bottom)	\$2,000
9	3 HP tank mixer	\$6,000
	NaOH feed system	\$1,750
10	Fe ³⁺ feed system	\$875
11	Diaphragm pump	\$3,750
12	air compressor	\$1,500
	6 cu ft filter press	\$14,500
13	Solids container	\$400
14	In line filtration cartridge system	\$250
15	Eff. Hold III (1000 gal. support w/conical bottom)	\$2,000
	3 HP tank mixer	\$6,000
16	dilute H ₂ SO ₄ feed system	\$1,500
17	Cs resin bed pump	\$2,500
18	In line filtration cartridge system (0.1 µm)	\$350
19	Cs resin bed	\$25,000
20	Whoopsie Tank (1000 gal. support w/conical bottom)	\$2,000
	3 HP tank mixer	\$6,000
21	dilute NaOH feed system	\$1,500
22	Scaffolding	\$20,000
Major Equipment Cost:		\$176,250

6.2 Train I Chemical Consumption

The effluent tanks in Train I processing are each sized to contain 5 complete wash cycles of spent fluids (chemicals and rinse, 750 gal. total) from the ultrasonic baths. The spent fluids will be processed in 750 gal. batches. The expected chemical consumption for one 750 gal. effluent batch, or 5 ultrasonic decontamination batches, is outlined below.

Charge make up:

	<i>Chemical</i>	<i>Volume</i>	<i>Unit Price</i>	<i>Batch Cost</i>
1.)	TECHXTRACT® 0300	5x7.5 gal.	\$ 49.00 /gal.	\$1,837.50
2.)	Conc. H ₂ SO ₄	5x7.5 gal.	\$ 1.27 /gal.	\$ 47.63
3.)	De-ionized Water	<u>5x135 gal.</u>	\$ 0.42 /gal.	<u>\$ 283.50</u>
	Total	750 gal.		\$2,168.63

Waste Treatment per 750 gal. batch:

	<i>Chemical</i>	<i>Volume</i>	<i>Unit Price</i>	<i>Batch Cost</i>
4.)	50% NaOH	83 gal.	\$ 2.36 /gal.	\$ 195.88
5.)	10% Fe ³⁺ sulfate	10 gal.	\$ 1.97 /gal.	\$ 19.70
6.)	10% H ₂ SO ₄	0.5 gal.	\$ 0.51 /gal.	\$ 0.25
7.)	10% NaOH	0.4 gal.	\$ 0.90 /gal.	\$ <u>0.36</u>
	Total			\$ 216.19

Cesium ion exchange resin:

The amount of the optional cesium ion exchange resin required to treat a batch will depend on the cesium concentration in the spent solution. From this value and the resin loading at this cesium concentration, resin usage can be estimated. Cesium isotherms using UOP IONSIV® resin were performed for equilibrium Cs concentrations in the 20 to 1000 ppm Cs range. The cesium concentrations that will be encountered in the field are currently unknown. Resin consumption spanning two decades of Cs concentration (10 to 1000 ppm) are calculated based on a resin loading of 150 mg cesium per gram resin and a resin cost of \$150.00/lb.

Cs resin per 750 gal. batch:

	<i>Cs concentration</i>	<i>Volume</i>	<i>Mass resin</i>	<i>Batch Cost</i>
A.)	10 ppm	750 gal.	0.42 lb	\$ 63.00
B.)	100 ppm	750 gal.	4.2 lb	\$ 630.00
C.)	1000 ppm	750 gal.	42 lb	\$ 6300.00

To estimate the total chemical cost associated with the Train I process, a cesium concentration of 100 ppm is assumed. Based on the above calculations, the total chemical and resin cost for Train I decontamination and waste treatment is estimated to be \$ 3,015 per 750 gal. batch.

6.3 Train II Configuration

Decontamination and effluent treatment for a Train II configured system will also be performed as a batch operation. The process flow characteristics are similar to Train I processing. The major difference of a Train II system versus Train I is driven by the addition of TECHXTRACT® 0200 and TECHXTRACT® 0100 products in the decontamination step. This impacts the decontamination step and the effluent treatment. The two additional solutions introduce organic complexing agents and chelates into the waste stream, substantially increasing the complexity of waste treatment. These compounds must be destroyed before the complexed metals can be recovered. This necessitates additional treatment (e.g. chelate destruction by AOP using UV/H₂O₂) and a second hydroxide precipitation step.

In the decontamination system, Train II is parallel to the Train I system shown in Fig. 6.1.1 and described in Section 6.1. The specific differences are: (i) 6 different product feed drums (vs. 3); (ii) 3 chemical make-up tanks (vs. 1); (iii) 5 ultrasonic baths (vs. 2); plus (iv) corresponding increases in piping and conveyor size.

The primary effluent system is identical to the Train I system, except that the hold tanks will be 2500 gal in size (vs. 1000 gal). This is due to the larger effluent volume per batch (1875 gal vs. 750 gal). The optional effluent treatment for the removal of soluble metals such as cesium is also identical.

There is, however, a significant new system for chelant destruction before the final stage. This system is shown schematically in Fig. 6.3.1. After the solid-liquid separation in the primary effluent treatment, the liquid stream is routed to either hold tank III or hold tank IV (A and B). At this stage of treatment, the effluent still contains complexed metals and metals that do not form insoluble metal hydroxides. The organic complexing agents are destroyed using an AOP process (e.g. UV/H₂O₂) to release the complexed metals (F). The pH is adjusted to ca. 3.0 using 10% H₂SO₄ (C) to avoid the formation of metal hydroxide sludge during processing. An in line filter (E) is used to remove any particulate fines prior to the AOP reactor (F). Hydrogen peroxide, at 30% concentration will be dosed from 55 gal drums to the appropriate hold tank or, preferably, directly to the AOP reactor. The chelate destruction by AOP will be performed in a batch mode and may require multiple passes through the reactor depending on effluent criteria.

Figure 6.3.1 - Schematic of Train II Chelant Destruction System

After destroying the chelates, the waste water is next routed to hold tank V (G) where the liberated metals are co-precipitated with iron. Iron addition will be from a 10% Fe solution from a 55 gal. drum (I). Again, if the solution contains chromium in the plus 6 oxidation state, Fe^{2+} may be added to reduce Cr^{6+} to Cr^{3+} . NaOH addition from a 10% solution in a 55 gal drum will be dosed to bring the pH to 9.5. Precipitated metal hydroxides will be removed by a filter press (J, K). This separation may need to be automated depending on the level of radioactivity of the sludge. The removed solids (L) require proper disposal. The water is next routed through an in line particle filter (M) and on to the optional effluent treatment/ion exchange step for the removal of soluble metals.

In summary, the major sections of a Train II processing system consist of:

- Chemical supply tanks
- Make up tanks
- Ultrasonic baths (5)
- Hold and treatment tanks for heavy metal precipitation (2x)

Solid-liquid separation (filtration) unit(s)
 An AOP unit for the destruction of chelates and complexers
 Cs removal unit (optional)
 Final pH adjustment (optional)

The major equipment items and estimated costs are listed in Table 6.3.1. The prices of the individual items add up to \$450,225, and do not include delivery, installation, or mark-up.

Table 6.3.1 *Major Equipment Cost for Train II Decontamination and Waste Treatment*

Item #	Description	Estimated Cost
1	drum pump for 0300	\$600
	0300 barrel mixer	\$300
2	drum pump for conc. H ₂ SO ₄	\$600
3	drum pump for 0200	\$600
	0200 barrel mixer	\$300
4	drum pump for conc. HCl	\$600
5	drum pump for 0100	\$600
	0100 barrel mixer	\$300
6	drum pump for 50% KOH	\$600
7	DI Supply	\$16,000
8	0300 make up tank, 100 gal.	\$400
	mixer & mount	\$425
	pump	\$475
9	0200 make up tank, 100 gal.	\$400
	mixer & mount	\$425
	pump	\$475
10	0100 make up tank, 100 gal.	\$400
	mixer & mount	\$425
	pump	\$475
11	five 75 gal Crest U.S. baths @ 18 K ea.	\$90,000
	SS liners (@ 10% of units)	\$9,000
	two 3/4" diaphragm pumps (acid side/base side)	\$3,000
12	Decon line conveyer	\$22,500
13	Eff. Hold I (2500 gal. support w/conical bottom)	\$4,225
	5 HP tank mixer	\$6,500
14	Eff. Hold II (2500 gal. support w/conical bottom)	\$4,225
	5 HP tank mixer	\$6,500
15	NaOH feed system	\$1,750
16	Fe ³⁺ feed system (hand barrel pump)	\$875
17	Air compressor	\$1,500
18	Diaphragm pump	\$3,750
19	6 cu ft filter press	\$14,500
20	Solids container	\$400

Item #	Description	Estimated Cost
21	In line filtration cartridge system	\$250
22	Eff. Hold III (2500 gal. support w/conical bottom)	\$4,225
	5 HP tank mixer	\$6,500
23	Eff. Hold IV (2500 gal. support w/conical bottom)	\$4,225
	5 HP tank mixer	\$6,500
24	dilute H ₂ SO ₄ feed system (smart pump)	\$1,500
25	H ₂ O ₂ feed system for AOP reactor (1-21-97 quote)	\$4,400
26	In line filtration cartridge system (0.1 µm)	\$350
27	10 kW AOP reactor (incl. training)	\$71,100
28	Eff. Hold V (2500 gal. support w/conical bottom)	\$4,225
	5 HP tank mixer	\$6,500
29	dilute NaOH feed system	\$1,500
30	Fe ³⁺ feed system	\$875
31	1" Diaphragm pump	\$2,050
32	1 cu ft filter press	\$5,000
33	Solids container	\$250
34	In line filtration cartridge system (0.1 µm)	\$350
35	Eff. Hold VI (2500 gal. support w/conical bottom)	\$4,225
	5 HP tank mixer	\$6,500
36	dilute H ₂ SO ₄ feed system	\$1,500
37	Cs resin bed pump	\$2,500
38	In line filtration cartridge system (0.1 µm)	\$350
39	Cs resin bed	\$25,000
40	dilute NaOH feed system	\$1,500
41	Whoopsie Tank (2500 gal. support w/conical bottom)	\$4,225
	5 HP tank mixer	\$6,500
	Scaffolding	\$30,000
	Control box	\$40,000
	Sizing of AOP reactor and pilot test rental	\$15,000
	Major Equipment Cost:	\$450,225

6.4 Train II Chemical Consumption

The primary effluent tanks in Train II processing are 2500 gal. each, sized to contain 5 complete wash cycles of spent fluids (5 x 5 x 75 gal. = 1875 gal.) from the ultrasonic baths. The spent fluids can be processed in 1875 gal. batches. The expected chemical consumption for one 1875 gal. effluent batch (5 ultrasonic decontamination batches) is outlined below.

Charge make up:

	<i>Chemical</i>	<i>Volume</i>	<i>Unit Price</i>	<i>Batch Cost</i>
1.)	TECHXTRACT® 0300	5x7.5 gal.	\$ 49.00 /gal.	\$ 1837.50
2.)	Conc. H ₂ SO ₄	5x7.5 gal.	\$ 1.27 /gal.	\$ 46.62
3.)	TECHXTRACT® 0200	5x7.5 gal.	\$ 49.00 /gal.	\$ 1837.50
4.)	Conc. HCl	5x7.5 gal.	\$ 1.18 /gal.	\$ 44.25
5.)	TECHXTRACT® 0100	5x7.5 gal.	\$ 49.00 /gal.	\$ 1837.50
6.)	50% KOH	5x1.5 gal.	\$ 2.36 /gal.	\$ 17.70
7.)	De-ionized Water	<u>5x336gal.</u>	\$ 0.42/gal.	<u>\$ 705.60</u>
	Total	1875 gal.		\$ 6326.67

Waste Treatment per 1875 gal. batch:

	<i>Chemical</i>	<i>Volume</i>	<i>Unit Price</i>	<i>Batch Cost</i>
First Precipitation:				
8.)	50% NaOH	110 gal.	\$ 2.36/gal.	\$ 259.60
9.)	10% Fe ³⁺ sulfate	16 gal.	\$ 1.97/gal.	\$ 31.52
UV treatment:				
10.)	10% H ₂ SO ₄	8 gal.	\$ 0.51/gal.	\$ 4.08
11.)	30% H ₂ O ₂	50 gal.	\$ 1.87/gal.	\$ 93.50
Second precipitation:				
12.)	50% NaOH	5.7 gal.	\$ 2.36 /gal.	\$ 13.45
13.)	10% Fe ³⁺ sulfate	6.2 gal.	\$ 1.97/gal.	\$ 12.21
Cs Treatment:				
12..)	10% H ₂ SO ₄	1.2 gal.	\$ 0.51/gal.	\$ 0.61
Final pH adjustment:				
13.)	10% NaOH	1.0 gal.	\$ 0.90/gal.	<u>\$ 0.90</u>
	Total			\$ 415.87

Cesium ion exchange resin:

As discussed in Section 6.2, the amount of ion exchange resin required to treat a batch will depend on the cesium concentration in the spent waste stream and the resin loading at this concentration. The cesium concentrations that will be encountered in the field are currently

unknown. Resin consumption spanning two decades of Cs concentration (10 to 1000 ppm) are calculated based on a resin loading of 150 g / kg resin and a resin cost of \$150.00/lb.

Cs resin per 1875 gal. batch:

	<i>Cs concentration</i>	<i>Volume</i>	<i>Mass resin</i>	<i>Batch Cost</i>
A.)	10 ppm	1875 gal.	1.05 lb	\$ 157.50
B.)	100 ppm	1875 gal.	10.5 lb	\$ 1575.00
C.)	1000 ppm	1875 gal.	105 lb	\$ 15750.00

To estimate the total chemical cost associated with the Train II process, a cesium concentration of 100 ppm is assumed. Based on the above calculations, the chemical and resin cost for Train II decontamination and waste treatment is estimated to be \$ 8318/1875 gal. batch.

7.0 DECONTAMINATION SYSTEM ECONOMIC MODEL

Even though a full-scale system has not been built and operated, it is important to make a preliminary assessment of the economics of the TECHXTRACT[®] based system versus commercially available alternatives. This section of the report provides the results of economic modeling for the full-scale system and research on alternative economics.

It should be noted at the outset that there are many uncertainties in the economics for the TECHXTRACT[®] based system and the alternatives at this stage. The analysis in this section uses several different scenarios to depict a reasonable range of economic results. Many of the input factors are based on EET's research as described in the previous sections. Additional information, particularly pertaining to the alternatives, was obtained from outside sources and published reports. These include "Analysis of the Potential Recycling of Department of Energy Radioactive Scrap Metal" prepared by S. Cohen and Associates (1995), papers from the 1995 Beneficial Reuse Conference, the March 1996 issue of *Radwaste Magazine* article entitled "An Industry Response to Recycle 2000," the U.S. Department of Energy Scrap Metal Inventory Report for the Office of Technology Development (Parsons report), and interviews with key industry players.

7.1 Key Factors

There are many different factors in the economics of the chemical decontamination system, several of which are particularly critical. The factors which have the greatest impact on the economic viability of the system are: capital costs (including system design factors), labor requirements, chemical consumption, survey costs, batch processing times, and material density. Each of these is further defined below.

Capital costs: Capital costs are highly dependent on the type of system that is required (2-stage vs. 5-stage; waste processing requirements). Beyond this, the effective capital cost per unit of throughput is a function of the life and usage of the system. A 2-stage system with simple waste processing requirements operating 250 days per year for 5 years is particularly economical, and can be effective in many cases.

Labor requirements: The number of people required to run the system and their wage and benefits scale are important economic inputs. The system has been designed to minimize the actual labor requirement. It will be important to take advantage of this and not overstaff the operation.

Chemical consumption: The TECHXTRACT[®] chemicals which are used in the chemical decontamination system are relatively expensive. The rate at which these chemicals are used will be an important cost factor. This rate will also directly affect the volume of secondary waste that requires processing and disposal. To the extent that the chemicals can be used for repeated baths, the consumption rate and decontamination cost will decrease.

Survey costs: The cost of performing radiological surveys to confirm that an item has been decontaminated to "free release" standards is a substantial part of the total cost. It is outside of the

scope of this report to deal with the policy issues that are associated with free release of materials. If random or some other form of "less than 100%" surveys ever gain acceptance, the total decontamination costs for free release of materials (regardless of the decontamination method chosen) will be decreased drastically.

Batch processing time: Many of the costs of operating the system are essentially fixed (e.g., labor, capital). Faster processing, in the form of shorter batch times, will increase throughput and therefore minimize these costs on a unit basis.

Material density: A final critical economic factor is the material density. Batch production rates will be a function of material volume - each bath will have a fixed volume capacity for scrap metal or debris. But the value of free-released scrap metal is expressed in \$/lb. Therefore, higher density translates into more favorable economics. (Higher density does not improve the economic comparison between decontamination and waste disposal.)

Three different scenarios have been developed for the primary economic analysis. The scenarios correspond to low cost, mid, and high cost cases. For each scenario, changes are made in the factors listed above plus several less critical variables. Input variables are based on EET's research and the external sources referenced above. Table 7.1.1 below presents the variables for the economic model.

A fourth scenario was also developed corresponding to the 5-stage system (which was identified as "Train 2" in the technical section of this report). Scenario 4 is based on the same assumptions as Scenario 2 (2-stage, "mid" case), except for the factors which are impacted by the more complex configuration. The variables that are different in Scenario 4 versus Scenario 2 are shown in Table 7.1.2.

Table 7.1.1 Key Variables for Economic Model	
Factor	Values for Each Scenario (Low, Mid, High)
Capital:	
- Assembly (% of components)	20, 25, 40%
- Life (yrs)	5, 4, 3
- Operation	
* Days per year	300, 250, 225 days
* Hours per day	10, 8, 8 hrs
Personnel:	
- Decon crew size (excl. survey)	3, 4, 5
- Base wage (\$/hr)	\$12, 15, 18
- Fringe & overhead (% of base wage)	30, 50, 80%
- PPE & consumables (\$/hr)	\$3.00, 5.00, 8.00
Chemicals:	
- Cost (\$/batch)	\$222, 297, 477
- Usage/recharge rate (gal/lb proc)	0.005, 0.010, 0.025 gal
Other:	
- Survey for free release (\$/lb)	\$0.10, 0.25, 0.40
- Prep/segment (\$/lb)	\$0.00, 0.20, 0.20
Secondary Waste:	
- Volume (% of feed)	3, 5, 10%
- Disposal (\$/cu ft)	\$100, 50, 24
Throughput:	
- Density of material (lb/cu ft)	100, 50, 20 lb/cu ft
- Basket size (cu ft)	10, 9.5, 9 cu ft
- Cycle time (min)	20, 30, 45 min

Table 7.1.2 Economic Analysis - Differences Between 3-Stage and 5-Stage		
	3-stage, Mid-case	5-stage
Capital cost	\$176,250	\$450,225
Crew size	4	5
Chemical cost per batch	\$297	\$809
Chemical usage rate	0.010 gal/lb	0.030 gal/lb
Electricity	\$1.16/hr	\$5.22/hr
Secondary Waste Volume	5%	10%

7.2 Economics of Alternatives

The cost of decontamination using the TECHXTRACT[®] based system cannot be viewed in isolation. It must be compared to other commercially available alternatives. The Cohen report considers a number of options for decontamination of scrap metal, and reports on their estimated unit costs. The primary methods used for traditional decontamination are abrasive blasting (shot, grit, CO₂), chemical decontamination, and metal melting. In addition, Cohen and several of the other sources cited above provide information on typical costs for disposal of low level radioactive waste. Based upon our literature review, these are the primary options for the disposition of radioactively-contaminated scrap metal and debris. A summary of these results is presented in Table 7.2.1.

Table 7.2.1 Cost Comparison for Commercially Available Options		
Option	Cost Range (\$/lb)	
	Low	High
Mechanical decon	1.40	3.35
Chemical decon	1.20	2.20
Metal melting	1.00	1.65
Disposal (as LLRW)	\$0.40	\$5.48
Basis for LLRW disposal		
- Density (lb/cu ft)	100	20
- Disposal (\$/cu ft)	\$24.00	\$100.00
- Transport (\$/cu ft)	\$1.00	\$2.50
- Other (\$/lb)	\$0.15	\$0.35

In addition to avoiding disposal, effective decontamination allows for the recovery and sale of the clean metal as scrap. In the case of some metals, the value that can be realized through sale as scrap exceeds the cost of the decontamination. This provides a very strong incentive for decontamination and free release. Typical scrap metal values are shown in Table 7.2.2.

Table 7.2.2 Typical Scrap Metal Values	
Metal	Value (Purchase as Scrap)
Carbon steel	\$0.04 - 0.06/lb
Lead	\$0.08 - 0.10/lb
Stainless steel	\$0.20 - 0.40/lb
Aluminum	\$0.25 - 0.50/lb
Copper	\$0.75 - 0.80/lb
Nickel	\$2.20 - 2.95/lb

7.3 Results of Economic Analysis

A spreadsheet model has been developed to determine the costs under each of these scenarios and to allow for "what if" assessments for other variables. A depiction of the model is shown below, with variables from Scenario 2 (mid case, 2-stage system).

ECONOMIC ANALYSIS - TECHXTRACT® DECONTAMINATION SYSTEM

Factor	Units	Value	\$/hr
EQUIPMENT:			
Capital	\$	176,250	
Assembly	%	25%	
Total	\$	220,313	
Hourly rate	\$/hr		\$27.54
- Life (yrs)	Yrs	4	
- Days/yr	Days/yr		250
- Hrs/day	Hrs/day	8	
OPERATING COSTS:			
Personnel	\$/hr		\$110.00
- Crew size	#	4	
- Base wage/hr	\$/hr	15	
- Fringe & ovhd	%	0.5	
- PPE & cons.	\$/hr	5	
Chemicals	\$/hr		\$37.62

- Cost/batch	\$/batch	297	
- Recharge rate	Gal/lb proc	0.01	
- Batch life	hrs/batch	7.9	
Secondary waste	\$/hr		\$9.50
- Volume/batch	%	5%	
- Density	lb/cu ft	250	
- Cost/cu ft	\$/cu ft	\$50.00	
Other	\$/hr		\$428.66
- Electricity	\$/hr	1.16	
- Survey	\$/lb	\$0.25	
- Prep/segment	\$/lb	\$0.20	
TOTAL OPERATING COST	\$/hr		\$544.62
	\$/lb		\$0.57
TOTAL COST	\$/hr		\$572.16
	\$/lb		\$0.60
THROUGHPUT RATE	lb/hr	950	
- Density	lb/cu ft	50	
- Basket size	cu ft	9.5	
- Cycle time	min		30
	hrs	0.5	
- Batch size	gal	75	

The results for each scenario are presented in Table 7.3.1.

Table 7.3.1 Summary of Economic Analysis (\$/hr)				
Cost Factors:	Scenario			
	1 - Low	2 - Mid	3 - High	4 - 5-stage
Capital	\$14.10	27.54	45.69	70.35
Personnel	55.80	110.00	202.00	137.50
Chemicals	44.40	37.62	38.16	307.42
Other	301.16	428.66	145.16	432.72
Secondary Waste	8.64	9.50	9.60	19.00
Total - \$/hr	\$424.10/hr	\$613.32/hr	\$440.61/hr	\$966.99/hr
Total - \$/lb	\$0.14/lb	\$0.65/lb	\$1.84/lb	\$1.02/lb
Throughput (lb/hr)	3000	950	240	950

Several key conclusions can be drawn from this analysis:

- 1.) The TECHXTRACT[®] based system can offer economics which are very favorable when compared to other commercially available alternatives and when compared to the scrap values of higher value metals.
- 2.) There are many uncertainties related to the system economics. Many of these uncertainties will be reduced or eliminated once project specific data is available.
- 3.) Survey and preparation costs are a major factor for this system. Any steps that can be taken to automate or accelerate the survey process will generate significant returns.
- 4.) Neither this system nor any other decontamination method can compete economically with the lowest end of the LLRW disposal scenarios. Even though the low end represents current DOE "pricing," these prices are primarily internal transfer costs which may not reflect realistic, fully burdened life cycle costs. The availability of cheap disposal is a major barrier to any recycling/reuse efforts.

8.0 CONCLUSIONS

Work under this PRDA contract was expected to lead to several specific results, as described in the project's "Management Plan" dated October 4, 1996. These results were:

- An evaluation of the effectiveness of the TECHXTRACT[®] decontamination system in meeting "free release" standards for radioactively contaminated metal.
- Evaluation of the effectiveness of the same system for decontaminating concrete rubble.
- Evaluation and selection of a secondary waste treatment system for the spent chemicals and water from the decontamination process.
- The design of an optimized system for this decontamination based on the TECHXTRACT[®] process.
- Assessment of the economics of the full-scale system versus disposal of the materials without decontamination.

The work described in this report addresses each of these issues. The primary focus of the project was the effectiveness and optimization of a chemical decontamination system based on the TECHXTRACT[®] technology. Testing using surrogates (SIMCON's) and actual samples of radioactively contaminated materials demonstrated that such a system could achieve extremely high decontamination factors and could reduce radioactivity to below published "free release" requirements. In many cases, background or non-detectable levels were achieved. By performing the final testing at a site which had a large inventory of contaminated materials, EET was able to show the robustness of the system for the decontamination of a wide range of radionuclides and substrates.

The testing program led to the definition of an optimized system for decontamination. The value of sonication, of slightly elevated temperatures, and of selective reagent chemistry was confirmed. Of particular significance to system economics, the program showed that a system with a reduced number of chemical baths can be highly effective.

The results of experiments for the decontamination of concrete rubble were less conclusive. Modest decontamination factors were achieved in surrogate testing, but adequate samples of radioactively-contaminated concrete could not be obtained. Given these results and the low value associated with recovering concrete, this aspect of the program does not appear to have good applications. (This conclusion does not extend to the decontamination of in-place concrete, which has a completely different set of characteristics for decontamination and different economic considerations.)

EET also identified a variety of secondary waste treatment options for the spent decontamination and rinse solutions. These included a co-precipitation method, an advanced oxidation photoreactor, and ion exchange. Each of these methods was shown to be effective in certain situations. The research did not conclude with a single treatment method because of the impact of many different application specific variables. These include final effluent treatment criteria, the availability of existing on-site treatment systems, the selected decontamination system (Train I vs. Train II), and the individual radionuclides.

The base phase of the contract concluded with simplified system design and economic analysis of full-scale decontamination systems, based on the results of the bench scale studies. The design shows that it is feasible to construct a full-scale system (i.e., 75 gallon bath capacities, maximum dimensions of 3' x 2' x 20") in a modular system for around \$200,000 (Train I configuration). Economic analysis highlighted the many different factors that affect overall decontamination costs, but showed that the TECHXTRACT[®] based system can compete very favorably with existing decontamination options and with disposal. Furthermore, this analysis showed that the system can generate income (resale value greater than decontamination cost) for certain high-value scrap metals.

The only significant project objective which was not completed during the base phase of the contract was the selection of a site for the optional, field demonstration. This objective was addressed during the base phase, but delays in the selection of DOE Large Scale D&D Demonstration sites and uncertainty surrounding the optional phase of this contract prevented this objective from being completed.

In conclusion, EET's PRDA contract has shown that a decontamination and treatment system based on the TECHXTRACT[®] chemistry can meet performance requirements for the decontamination of scrap metal, secondary waste treatment, and waste minimization, and that this system can be economically attractive versus currently available options. A field demonstration, under the optional phase of the contract, would allow for construction and operation of a system in actual conditions to verify these conclusions and to allow the DOE to realize the full benefits of this unique technology.

TABLE OF ACRONYMS

AOP	advanced oxidation process
C_o, C_f	initial, final concentrations (of contaminant)
D&D	decontamination and decommissioning
DF	decontamination factor
DOE	Department of Energy
dpm	disintegrations per minute
FETC	Federal Energy Technology Center
INEL	Idaho National Engineering Laboratory
NRC	Nuclear Regulatory Commission
ppm	parts per million
ppt	precipitation
PRDA	Program Research and Development Agreement
SIMCON	Simulated Contaminant
XRF	X-ray fluorescence

APPENDIX A

Results from SIMCON 2 Decontamination Experiments

Table A1: *Batch I experimental results and conditions for Exp. #1 - 14*
All experiments were performed at 43°C with sonication unless otherwise noted.

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#1 (301)	1.) 100% 0300	20				
	2.) 100% 0200	20				
	3.) DI	20				
	4.) 100% 0100	20				
	5.) DI	20	28.5 mg	85%	9.8	7.1
#2 (302)	1.) 50% 0300	20				
	2.) 50% 0200	20				
	3.) DI	20				
	4.) 50% 0100	20				
	5.) DI	20	31.5 mg	40%	12.0	33.6
#3 (303)	1.) 100% 0300	20				
	2.) 100% 0200	20				
	3.) DI	20				
	4.) 100% 0100	20				
	5.) DI (Temp @ 55°C)	20	46.0 mg	65%	13.9	32.0*
#4 (304)	1.) 100% 0300	20				
	2.) 100% 0200	20				
	3.) DI	20				
	4.) 100% 0100	20				
	5.) DI (Temp @ 60°C)	20	47.3 mg	80%	18.0	33.2
#5 (306)	1.) 50% 0300	20				
	2.) 50% 0200	20				
	3.) DI	20				
	4.) 50% 0100	20				
	5.) DI (Temp @ 25°C)	20	20.8 mg	50%	13.7	32.0
#6 (309)	1.) 100% 0300	20				
	2.) 100% 0200	20				
	3.) DI	20				
	4.) 100% 0100	20				
	5.) DI (Temp @ 25°C)	20	23.0 mg	30%	18.7	35.0

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#7 (310)	1.) 50% 0300 2.) 50% 0200 3.) DI 4.) 50% 0100 5.) DI (Mechanical stirring)	20 20 20 20 20	10.2 mg	0%	15.7	2.6
#8 (311)	1.) 100% 0300 2.) 100% 0300 3.) 100% 0300 4.) 100% 0300 (Temp @ 25°C) Sonication by Vibra Cell sonicator	5 10 15 20	---	20% 30% 35% 35%	7.3	10
#9 (312)	1.) 100% 0300 Sonication by Vibra Cell sonicator	20	---	25%	8.0	6.4
#10 (314)	1.) 100% 0300 2.) 100% 0200 3.) DI 4.) 100% 0100 5.) DI	60 60 60 60 60	39.2 mg	75%	72.7*	83.7*
#11 (315)	1.) 100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 100% 0200 & 3% HCl 3.) DI 4.) 100% 0100 @ pH 13.5 ^(a) 5.) DI	20 20 20 20 20	58.2 mg	95%	80.0*	59.3*
#12 (316)	50% HCl	60	53.2 mg	95%	161*	108*
#13 (317)	1.) 100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 100% 0200 & 3% HCl 3.) DI 4.) 100% 0100 @ pH 13.5 ^(a) 5.) DI (Temp @ 60°C)	20 20 20 20 20	43.2 mg	80%	120*	67.3*
#14 (318)	1.) 100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 100% 0200 & 3% HCl 3.) DI 4.) 100% 0100 @ pH 13.5 ^(a) 5.) DI (Temp @ 25°C)	20 20 20 20 20	32.8 mg	65%	15.0	21.5

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

^(a) pH adjusted by KOH

Table A2: *Batch II experimental results and conditions for Exp. #15 - 28*
All experiments were performed at 43°C with sonication unless otherwise noted.

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon DF Cs	Factors DF Zr
#15 (319)	1.) 100% 0300 2.) 100% 0200 3.) DI 4.) 100% 0100 5.) DI (with abrasives added)	30 30 30 30 30	24.2 mg	5%	15.1	9.1
#16 (321)	100% 0300 (steel pan)	30(b)	16.2 mg	15%	10.3	5.1
#17 (322)	100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ (steel pan)	30(b)	14.9 mg	15%	12.8	14.0
#18 (323)	100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂	30(b)	12.6 mg	10%	11.2	15.9
#19 (324)	1.) 50% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 50% 0200 & 3% HCl 3.) DI 4.) 50% 0100 @ pH 13.5 ^(a) 5.) DI	30(b) 30 30 30 30	38.8 mg	40%	20.8	20.6
#20 (325)	1.) 100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 100% 0200 & 3% HCl 3.) DI 4.) 100% 0100 @ pH 13.5 ^(a) 5.) DI	30(b) 30 30 30 30	32.8 mg	50%	21.6	20.9
#21 (327)	1.) 25% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5 ^(a) 5.) DI	30(b) 30 30 30 30	28.4 mg	90%	34.7	34.1
#22 (328)	1.) 100% 0300 2.) 100% 0200 3.) DI 4.) 100% 0100 5.) DI	30(b) 30 30 30 30	32.7 mg	60%	31.4	24.7
#23 (329)	1.) 50% 0300 2.) 50% 0200 3.) DI 4.) 50% 0100 5.) DI	30(b) 30 30 30 30	21.6 mg	30%	8.7	42.2

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#24 (330)	1.) 25% 0300 2.) 25% 0200 3.) DI 4.) 25% 0100 5.) DI (timer problems)	30(b) 30 30 30 30	25.0 mg	20%	14.3	34.4
#25 (332)	1.) 25% 0300 2.) 25% 0200 3.) DI 4.) 25% 0100 5.) DI	30(b) 30 30 30 30	24.6 mg	20%	19.6	29.7*
#26 (339)	50% HNO ₃	60	18.3 mg	20%	8.4	6.7
#27 (340)	1.) 25% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5 ^(a) 5.) DI	30(b) 30 30 30 30	23.6 mg	90%	37.3	30.3*
#28 (341)	1.) 25% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5 ^(a) 5.) DI (Temp @ 65°C)	30(b) 30 30 30 30	50.0 mg	95%	47.3*	27.3*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

(a) pH adjusted by KOH

(b) 10 min. presoak in solution at temperature without sonication

Table A3: *Batch III experimental results and conditions for Exp. #29 - 42*
All experiments were performed at 43°C with sonication unless otherwise noted.

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon DF Cs	Factors DF Zr
#29 (342)	1.) 50% 0300 2.) 50% 0200 ₁ 3.) DI 4.) 50% 0100 5.) DI (Temp @ 60°C)	30 30 30 30 30	39.6 mg	55%	32.3	35.3*
#30 (343)	1.) 50% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 50% 0200 & 3% HCl 3.) DI 4.) 50% 0100 @ pH 13.5(a) 5.) DI (Temp @ 60°C)	30 30 30 30 30	25.4 mg	65%	18.4	33.4
#31 (344)	1.) 100% 0300 2.) 100% 0200 ₁ 3.) DI 4.) 100% 0100 5.) DI (Temp @ 60°C)	30 30 30 30 30	28.0 mg	40%	24.6	16.2
#32 (345)	1.) 100% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 100% 0200 & 3% HCl 3.) DI 4.) 100% 0100 @ pH 13.5(a) 5.) DI (Temp @ 60°C)	30 30 30 30 30	42.6 mg	50%	30.6	50.5
#33 (346)	1.) 10% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 10% 0200 & 3% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI	30 30 30 30 30	48.5 mg	60%	17.8	16.3
#34 (347)	1.) 0% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 0% 0200 & 3% HCl 3.) DI 4.) 0% 0100 @ pH 13.5(a) 5.) DI (Reagent chemicals only)	30 30 30 30 30	68.4 mg	98%	70.3*	70.7*

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#35 (348)	1.) 0% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 0% 0200 & 3% HCl 3.) DI 4.) 0% 0100 @ pH 13.5(a) 5.) DI (Temp @ 60°C)	30 30 30 30 30	70.6 mg	80%	30.2	24.5
#36 (349)	1.) 25% 0300 & 3% H ₂ SO ₄ + 7% H ₂ O ₂ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5(a) 5.) DI	30 30 30 30 30	47.4 mg	90%	50.8	76.5
#37 (350)	1.) 25% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5(a) 5.) DI (3 g Sident 10 in solutions)	30 30 30 30 30	22.0 mg	85%	17.8	37.3
#38 (351)	1.) 25% 0300 & 3% H ₂ SO ₄ + 3% H ₂ O ₂ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5(a) 5.) DI (3 g Aerosil 200 in solutions)	30 30 30 30 30	42.6 mg	50%	20.8	24.2
#39 (353)	1.) 25% 0300 & 3% H ₂ SO ₄ + 7% H ₂ O ₂ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5(a) 5.) DI	30 30 30 30 30	43.5 mg	50%	26.7	36.3*
#40 (357)	1.) 25% 0300 & 3% H ₂ SO ₄ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5(a) 5.) DI	30 30 30 30 30	65.5 mg	97%	47.7*	53.7*
#41 (358)	1.) 25% 0300 & 3% H ₂ SO ₄ 2.) 25% 0200 & 3% HCl 3.) DI 4.) 25% 0100 @ pH 13.5(a) 5.) DI	30 30 30 30 30	62.8 mg	95%	48.0*	35.0*

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#42 (359)	1.) 25% 0300 & 3% H ₂ SO ₄	75				
	2.) 25% 0200 & 3% HCl	30				
	3.) DI	30				
	4.) 25% 0100 @ pH 13.5 ^(a)	30				
	5.) DI	30	69.0 mg	99%	93.3*	58.0*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

^(a) pH adjusted by KOH

Table A4: *Batch IV experimental results and conditions for Exp. #43 - 56*
All experiments were performed at 43°C with sonication unless otherwise noted.

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon DF Cs	Factors DF Zr
#43 (361)	10% H ₂ SO ₄	90	65.9 mg	65%	73.0*	47.0*
#44 (362)	1.) 10% 0300 & 3% H ₂ SO ₄ 2.) 10% 0200 & 3% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI	45 30 30 30 30	68.1 mg	97%	61.0*	77.0*
#45 (364)	1.) 10% 0300 & 3% H ₂ SO ₄ 2.) 10% 0200 & 3% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI	75 30 30 30 30	84.0	100%	71.3*	25.3*
#46 (366)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) 10% 0200 & 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI (Temp @ 50°C)	30 15 20 15 15	84.5	99%	69.7*	34.0*
#47 (371)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) 10% 0200 & 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI	45 20 20 20 20	61.8	98%	72.3*	44.3*
#48 (372)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) 10% 0200 & 10% HCl 3.) DI 4.) 10% 0100 @ pH 13.5(a) 5.) DI	15 20 15 25 15	66.0 mg	97%	98.0*	43.0*
#49 (373)	1.) 10% H ₂ SO ₄ 2.) 10% HCl 3.) DI 4.) DI @ pH 13(a) 5.) DI	30 30 30 30 30	66.0 mg	40% 45% 45% 60% 60%	46.7*	25.0*

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#50 (376)	1.) 10% 0300	30				
	2.) 10% 0200	30				
	3.) DI	30				
	4.) 10% 0100	30				
	5.) DI	30	26.9 mg	55%	4.7	20.7
#51 (377)	1.) 10% 0300 & 10% H ₂ SO ₄	30				
	2.) 10% 0200 & 10% HCl	30				
	3.) DI	30				
	4.) 10% 0100 @ pH 13.5 ^(a)	30				
	5.) DI	30	59.1 mg	98%	28.0*	40.3*
#52 (378)	1.) 10% 0300 & 10% H ₂ SO ₄	30				
	2.) 10% 0200 & 10% HCl	30				
	3.) DI	30				
	4.) 10% 0100 @ pH 13.5 ^(a)	30				
	5.) DI	30	72.0 mg	96%	44.3*	48.0*
#53 (379)	1.) 10% 0200	45				
	2.) DI	15	21.3 mg	40%	4.2	12.8
#54 (380)	1.) 10% 0200 & 10% HCl	45				
	2.) DI	15	28.0 mg	70%	31.2	26.4
#55 (387)	1.) 10% 0100	45				
	2.) DI	15	19.3 mg	45%	3.9	16.0
#56 (390)	1.) 10% 0100 @ pH 13.5 ^(a)	45				
	2.) DI	15	16.2 mg	40%	5.5	4.2

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

^(a) pH adjusted by KOH

Table A5: *Batch V experimental results and conditions for Exp. #57 - 70*
All experiments were performed at 43°C with sonication unless otherwise noted.

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon DF Cs	Factors DF Zr
#57 (391)	1.) 10% HCl 2.) DI	45 15	47.3 mg	90% 90%	46.0	66.0*
#58 (392)	1.) DI @ pH 13 ^(a) 2.) DI	45 15	19.9 mg	10%	5.4	7.8
#59 (393)	1.) 10% 0300 & 20% H ₂ SO ₄ 2.) DI	45 15	57.0 mg	95%	78.3	61.0*
#60 (394)	1.) 10% HCl 2.) DI	45 15	30.2 mg	65%	46.7	64.0*
#61 (395)	1.) DI	60	19.6 mg	20%	4.1	6.3
#62 (396)	1.) 10% 0300 2.) DI	45 15	30.6 mg	35%	10.1	5.6
#63 (397)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	71.3 mg	90%	23.9	24.7*
#64 (398)	1.) 10% 0300 & 10% H ₂ SO ₄ (60°C) 2.) DI (60°C)	45 15	36.6 mg	45%	8.6	7.4
#65 (399)	1.) 10% 0300 (60°C) 2.) DI (60°C)	45 15	71.8 mg	98%	51.0*	25.7*
#66 (400)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	60.4 mg	98%	29.3*	39.0*
#67 (401)	1.) 10% 0300 & 10% H ₂ SO ₄ + 5% NaCl 2.) DI	45 15	90.5 mg	95%	91.7*	65.7*
#68 (405)	1.) 10% 0300 & 10% H ₂ SO ₄ + 1% NiCl ₂ 2.) DI	45 15	232 mg	80%	46.0	21.8
#69 (406)	1.) 10% 0300 & 10% H ₂ SO ₄ + 3% NiCl ₂ 2.) DI	45 15	191.8 mg	60%	26.8	11.7
#70 (407)	1.) 10% 0300 & 10% H ₂ SO ₄ 2.) DI	45 15	70.4 mg	96%	69.0*	44.3*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

(a) pH adjusted by KOH

Table A6: *Batch VI experimental results and conditions for Exp. #71 - 84*
All experiments were performed at 43°C with sonication unless otherwise noted.

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#71 (408)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	90.6 mg	96%	24.5	27.7*
#72 (409)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	72.6 mg	85%	21.0	33.7*
#73 (410)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	90.6 mg	96%	77.0*	58.7*
#74 (411)	1.) DI	60	14.4 mg	20%	3.5	5.3
#75 (412)	1.) DI	60	19.2 mg	10%	7.0	8.2
#76 (413)	1.) DI	60	18.5 mg	30%	5.2	5.8
#77 (416)	1.) DI	60	20.6 mg	20%	5.8	6.7
#78 (421)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	40.0 mg	10%	---	---
#79 (422)	1.) DI (stirring only)	60	1.0 mg	5%	2.8	1.1
#80 (423)	1.) 10% H ₂ SO ₄	45				
	2.) DI	15	84.6 mg	20%	30.6	26.3*
#81 (424)	1.) 10% 0300 & 10% H ₂ SO ₄ + 3% H ₂ O ₂	45				
	2.) DI	15	37.2 mg	65%	8.4	4.6
#82 (425)	1.) 100% 0300	45				
	2.) DI	15	14.7 mg	20%	5.9	4.7
#83 (435)	1.) 100% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	28.6 mg	10%	6.3	10.1
#84 (437)	1.) 50% 0300	45				
	2.) DI	15	48.3 mg	35%	9.2	5.4

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

Table A7: *Batch VII experimental results and conditions for Exp. #85 - 98*
All experiments were performed at 43°C with sonication unless otherwise noted

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#85 (438)	1.) 50% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	56.7 mg	70%	7.3	8.1
#86 (439)	1.) 100% 0200	45				
	2.) DI	15	28.5 mg	10%	46.0	13.0
#87 (440)	1.) 100% 0200 & 10% HCl	45				
	2.) DI	15	40.3 mg	30%	53.9	53.0*
#88 (442)	1.) 50% 0200	45				
	2.) DI	15	49.5 mg	40%	42.5	57.7*
#89 (443)	1.) 50% 0200 & 10% HCl	45				
	2.) DI	15	45.5 mg	30%	41.4	70.7*
#90 (444)	1.) 100% 0200 & 10% H ₂ SO ₄	45				
	2.) DI	15	48.0 mg	40%	55.5	51.3*
#91 (445)	1.) 50% 0200 & 10% H ₂ SO ₄	45				
	2.) DI	15	40.5 mg	40%	73	85.3*
#92 (447)	1.) 10% 0200 & 10% H ₂ SO ₄	45				
	2.) DI	15	41.7 mg	40%	96.9	97.2*
#93 (448)	1.) 100% 0100	45				
	2.) DI	15	15.5 mg	5%	2.8	9.8
#94 (449)	1.) 50% 0100	45				
	2.) DI	15	17.1 mg	5%	3.8	20.0
#95 (450)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	63.0 mg	97%	78.5	52.0
#96 (451)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	76.7 mg	96%	43.7*	45.0*
#97 (455)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	73.4 mg	97%	23.7*	54.0*
#98 (456)	1.) 10% 0300 & 10% H ₂ SO ₄	45				
	2.) DI	15	68.9 mg	98%	42.0*	26.3*

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

Table A8: *Batch VIII experimental results and conditions for Exp. #99 - 112*
All experiments were performed at 43°C with sonication unless otherwise noted

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#99 (458)	1.) 10% 0300 & 10% H ₂ SO ₄	45	71.6	96%	29.7*	31.3*
	2.) DI	15				
#100 (460)	1.) 10% 0300 & 10% H ₂ SO ₄	45	69.7	98%	43.0*	53.7*
	2.) DI	15				
#101 (461)	1.) 10% 0300 & 10% H ₂ SO ₄	45	73.7	94%	56.0*	41.3*
	2.) DI	15				
#102 (471)	1.) 10% 0300 & 10% H ₂ SO ₄	45	80.8	97%	22.3*	39.0*
	2.) DI	15				
#103 (472)	1.) 10% 0300 & 10% H ₂ SO ₄	45	59.8	95%	18.3*	56.7*
	2.) DI	15				
#104 (464)	1.) 10% 0300 & 10% H ₂ SO ₄	45	76.8	97%	25.7*	31.7*
	2.) DI	15				
#105 (468)	1.) 10% 0300 plus 10% H ₂ SO ₄	30	72.2 mg	96%	24.7*	79.3*
	2.) 10% 0200 plus HCl	30		99%		
	3.) DI	30		99%		
	4.) 10% 0100 @ pH 13.5(a)	30		100%		
	5.) DI	30		100%		
	run #1 of 5 in the same solutions					
#106 (469)	1.) 10% 0300 plus 10% H ₂ SO ₄	30	82.8 mg	94%	25.3*	29.0*
	2.) 10% 0200 plus HCl	30		98%		
	3.) DI	30		98%		
	4.) 10% 0100 @ pH 13.5(a)	30		99%		
	5.) DI	30		99%		
	run #2 of 5 in the same solutions					
#107 (473)	1.) 10% 0300 plus 10% H ₂ SO ₄	30	73.8 mg	92%	18.7*	42.7*
	2.) 10% 0200 plus HCl	30		97%		
	3.) DI	30		97%		
	4.) 10% 0100 @ pH 13.5(a)	30		98%		
	5.) DI	30		98%		
	run #3 of 5 in the same solutions					
#108 (476)	1.) 10% 0300 plus 10% H ₂ SO ₄	30	79.4 mg	96%	21.3*	51.3*
	2.) 10% 0200 plus HCl	30		99%		
	3.) DI	30		99%		
	4.) 10% 0100 @ pH 13.5(a)	30		99%		
	5.) DI	30		99%		
	run #4 of 5 in the same solutions					

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#109 (479)	1.) 10% 0300 plus 10% H ₂ SO ₄	30	78.5 mg	98%	19.3*	63.7*
	2.) 10% 0200 plus HCl	30		98%		
	3.) DI	30		98%		
	4.) 10% 0100 @ pH 13.5(a)	30		99%		
	5.) DI	30		99%		
	run #5 of 5 in the same solutions					
#110 (478)	1.) 10% 0300 plus 10% H ₂ SO ₄	15	71.6 mg	70%	18.7*	33.7*
	2.) 10% 0200 plus HCl	15		75%		
	3.) DI	15		75%		
	4.) 10% 0100 @ pH 13.5(a)	15		80%		
	5.) DI	15		84%		
	run #1 of 5 in the same solutions					
#111 (484)	1.) 10% 0300 plus 10% H ₂ SO ₄	15	83.0 mg	80%	27.7*	30.3*
	2.) 10% 0200 plus HCl	15		90%		
	3.) DI	15		93%		
	4.) 10% 0100 @ pH 13.5(a)	15		93%		
	5.) DI	15		95%		
	run #2 of 5 in the same solutions					
#112 (498)	1.) 10% 0300 plus 10% H ₂ SO ₄	15	80.5 mg	90%	30.7*	40.7*
	2.) 10% 0200 plus HCl	15		96%		
	3.) DI	15		98%		
	4.) 10% 0100 @ pH 13.5(a)	15		99%		
	5.) DI	15		99%		
	run #3 of 5 in the same solutions					

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

(a) pH adjusted by KOH

Table A9: *Batch IX experimental results and conditions for Exp. #113 - 115*
All experiments were performed at 43°C with sonication unless otherwise noted

Exp. coupon	Decontamination Train Conditions	Time (min)	Wt. Loss	Visual Oxide Removal	Decon Factors DF Cs DF Zr	
#113 (499)	1.) 10% 0300 plus 10% H ₂ SO ₄	15	81.4 mg	85%	18.3*	37.7*
	2.) 10% 0200 plus HCl	15		92%		
	3.) DI	15		95%		
	4.) 10% 0100 @ pH 13.5 ^(a)	15		98%		
	5.) DI	15		98%		
	run #4 of 5 in the same solutions					
#114 (502)	1.) 10% 0300 plus 10% H ₂ SO ₄	15	78.3 mg	50%	26.0*	36.7*
	2.) 10% 0200 plus HCl	15		60%		
	3.) DI	15		85%		
	4.) 10% 0100 @ pH 13.5 ^(a)	15		95%		
	5.) DI	15		96%		
	run #5 of 5 in the same solutions					
#115 (507)	1.) DI	150	28.1mg	20%	6	16.4

* DF value based on C_f = 3 µg; final contaminant level at or below detection limit

^(a) pH adjusted by KOH

APPENDIX B

Substrates and Radionuclides from Alaron Testing

Substrate/Alaron Lot#	Primary Isotopes	Percent of Activity
Concrete 92-075	Co-60 Cs-137	8.0% 92.0%
Stainless steel fuel rack 94-010	Fe-55 Co-58 Co-60 Ni-63 Others (all <1.0%)	59.3% 12.3% 10.7% 14.6% 3.1%
Stainless steel sheet 94-023	Fe-55 Co-58 Co-60 Ni-63 Others (all <1.0%)	58.9% 12.3% 10.7% 14.6% 3.5%
Copper tubing 94-042	H-3 Fe-55 Co-58 Co-60 Ni-63 Zr-95 Nb-95 Cs-134 Cs-137 Others (all <1.0%)	3.5% 21.3% 9.0% 34.3% 13.9% 2.2% 4.1% 1.0% 8.5% 2.2%
Titanium tubing 94-157	Cr-51 Mn-54 Fe-55 Co-60 Zn-65 Cs-134 Cs-137 Others (all <1.0%)	13.4% 1.7% 29.1% 9.4% 3.3% 21.9% 20.2% 1.0%
Stainless steel sheet 95-176	H-3 Fe-55 Co-58 Co-60 Ni-63 Cs-134 Cs-137 Pu-241 Others (all <1.0%)	5.1% 51.6% 4.4% 11.6% 10.8% 1.7% 10.4% 1.5% 2.9%

Substrate/Alaron Lot#	Primary Isotopes	Percent of Activity
Aluminum 96-071	U-234	39.1%
	U-235	1.7%
	U-238	59.2%
Carbon steel 96-090	Mn-54	1.3%
	Fe-55	15.8%
	Co-58	20.2%
	Co-60	5.6%
	Ni-63	10.6%
	Nb-95	2.0%
	Zr-95	1.7%
	Ag-110m	1.4%
	Cs-134	14.5%
	Cs-137	26.6%
	Others (all <1.0%)	0.3%

APPENDIX C

Results from “Hot” Testing at Alaron Corp.

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
# 1	94-042	3/25/97 94-042 #1	Cu tubing	10% 0300 + 10% H2SO4	15 min.	101	inside	967	507	5	2,888	13	231
# 2	94-010	3/25/97 94-010 #1	Stainless Steel fuel rack (Sheet)	10% 0300 + 10% H2SO4	30 min.	101	side 1	30,967	1,352	5	190,388	5,294	36
						101	side 2	55,051	4,090	5	340,913	22,406	15.2
				Same 0300 SIn	30 min.	101	side 1	30,967	1,422	5	190,388	5,731	33.2
						101	side 2	55,051	2,639	5	340,913	13,337	25.6
# 3	96-090	3/25/97 96-090 #1	Steel Metal (Sheet)	10% 0300 + 10% H2SO4	15 min.	101	side 1	873	550	5	2,300	281	8.2
						101	side 2	877	497	5	2,325	(50)	***
# 4	94-010	3/26/97 94-010 #1	Stainless Steel fuel rack (Sheet)	10% 0300 + 10% H2SO4	30 min.	101	side 1	49,559	3,128	5	306,588	16,394	18.7
						101	side 2	30,826	4,638	5	189,506	25,831	7.3
				10% 0300 + 10% H2SO4	30 min.	101	side 1	49,559	1,879	5	306,588	8,588	35.7
						101	side 2	30,826	2,745	5	189,506	14,000	13.5
# 5	94-010	3/25/97 94-010 #1 (Exp #2 sample)	Stainless Steel fuel rack (Sheet)	Exp #2 Train		101	side 1	30,967	1,422	5	190,388	5,731	33.2
						101	side 2	55,051	2,639	5	340,913	13,337	25.6
				10% 0300	30 min.	100	side 1	30,967	1,292	5	190,419	4,950	38.5
				+ 10% H2SO4		100	side 2	55,051	2,335	5	340,944	11,469	29.7
# 6	94-010	3/26/97 94-010 #2	Stainless Steel fuel rack (Sheet)	10% 0300 + 10% H2SO4	30 min.	100	side 1	28,588	5,646	5	175,550	32,163	5.5
						100	side 2	43,807	5,296	5	270,669	29,975	9
				10% 0200 + 10% HCl	30 min.	100	side 1	28,588	1,467	5	175,550	6,044	29
						100	side 2	43,807	2,583	5	270,669	13,019	20.8
				DI	30 min.	100	side 1	28,588	1,196	5	175,550	4,350	40.4
						100	side 2	43,807	2,178	5	270,669	10,488	25.8
				10% 0100 + KOH	30 min.	100	side 1	28,588	1,228	5	175,550	4,550	38.6
						100	side 2	43,807	2,090	5	270,669	9,938	27.2

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				DI	30 min.	100	side 1	28,588	1,402	5	175,550	5,637	31.1
						100	side 2	43,807	2,491	5	270,669	12,444	21.8
# 7	94-042	3/26/97 94-042	Copper Tubing	10% 0300 + 10% H2SO4	7.5 min.	100	inside	879	510	5	2,369	63	37.9
# 8	94-010	3/26/97 94-010 #3	Stainless Steel fuel rack (Sheet)	10% 0200 + 10% HCl	30 min.	100	side 1	54,331	3,370	5	336,444	17,938	18.8
						100	side 2	28,162	3,318	5	172,888	17,613	9.8
				10% 0200 + 10% HCl	30 min.	100	side 1	54,331	2,875	5	336,444	14,844	22.7
						100	side 2	28,162	2,629	5	172,888	13,306	13
# 9	94-010	3/26/97 94-010 #4	Stainless Steel fuel rack (Sheet)	10% 0100 + KOH	30 min.	100	side 1	52,777	17,381	5	326,731	105,506	3.1
						100	side 2	24,852	12,282	5	152,200	73,638	2.1
				10% 0100 + KOH	30 min.	100	side 1	52,777	12,875	5	326,731	77,344	4.2
						100	side 2	24,852	7,675	5	152,200	44,844	3.4
# 10	96-090	3/26/97 96-090 #1	Steel Metal (Sheet)	10% 0300 + 10% H2SO4	7.5 min.	100	side 1	1,045	494	5	3,406	(38)	***
						100	side 2	1,104	518	5	3,775	112	33.6
# 11	94-010	3/26/97 94-010 #1	Stainless Steel fuel rack (Sheet)	Exp #4 Train		101	side 1	49,559	1,879	5	306,588	8,588	35.7
						101	side 2	30,826	2,745	5	189,506	14,000	13.5
				10% 0300 + 10% H2SO4	30 min.	102	side 1	49,559	1,565	5	306,556	6,594	46.5
						102	side 2	30,826	1,691	5	189,475	7,381	25.7
				10% 0300 + 10% H2SO4	45 min.	102	side 1	49,559	1,471	5	306,556	6,006	51
						102	side 2	30,826	1,280	5	189,475	4,813	39.4
				10% 0300 + 10% H2SO4	45 min.	102	side 1	49,559	1,314	5	306,556	5,025	61
						102	side 2	30,826	1,262	5	189,475	4,700	40.3
# 12	94-010	3/27/97 94-010 #1	Stainless Steel fuel rack (Sheet)	10% 0300 + 30% H2SO4	30 min.	102	side 1	72,688	13,691	5	451,113	82,381	5.5
						102	side 2	29,613	3,137	5	181,894	16,419	11.1
				10% 0300 + 10% H2SO4	30 min.	102	side 1	72,688	4,907	5	451,113	27,481	16.4
						102	side 2	29,613	1,947	5	181,894	8,981	20.3
				10% 0300 + 10% H2SO4	30 min.	102	side 1	72,688	4,937	5	451,113	27,669	16.3
						102	side 2	29,613	1,716	5	181,894	7,538	24.1

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF	
# 13	94-042	3/27/97 94-042 Lot #1	Cu tubing	10% 0300 + 10% H2SO4	7.5 min.	102	inside	527	189	2	5,047	(234)	***	
						102	inside	577	219	2	5,828	234	24.9	
						102	inside	635	199	2	6,734	(78)	***	
						102	inside	554	196	2	5,469	(125)	***	
						102	inside	500	176	2	4,625	(438)	***	
						102	inside	564	196	2	5,625	(125)	***	
						102	inside	510	187	2	4,781	(266)	***	
						102	Average=	552	195	2	5,444	(147)	***	
# 14	95-053	3/27/97 95-053 #1	Stainless Steel tubing	10% 0300 + 10% H2SO4	30 min.	102	side 1	1,097	500	5	3,669	(63)	***	
						102	side 2	747	504	5	1,481	(38)	***	
# 15	95-053	3/27/97 95-053 #2	Stainless Steel tubing	10% 0300 + 10% H2SO4	10 min.	102	side 1	1,313	530	5	5,019	125	40.2	
						102	side 2	757	512	5	1,544	13	123.5	
# 16	94-010	3/28/97 94-010 #1	Stainless Steel fuel rack (Sheet)	10% 0200 + 10% H2SO4	30 min.	101	side 1	31,606	3,451	5	194,381	18,413	10.6	
						101	side 2	53,860	2,520	5	333,469	12,594	26.5	
				10% 0200 + 10% HCl	30 min.	101	side 1	31,606	2,820	5	194,381	14,469	13.4	
						101	side 2	53,860	1,905	5	333,469	8,750	38.1	
# 17	94-010	3/28/97 94-010 #2	Stainless Steel fuel rack (Sheet)	10% 0300 + 10% H2SO4	30 min.	101	side 1*	5,639	3,518	5	32,088	18,831	1.7	
						101	side 2	39,834	2,942	5	245,806	15,231	16.1	
				10% 0300 + 10% HCl	30 min.	101	side 1*	5,639	2,718	5	32,088	13,831	2.3	
						101	side 2	39,834	1,862	5	245,806	8,481	29	
				* note: side1 was covered with a sputter deposit										
# 18	94-156	3/28/97 94-156 #1	Titanium Tubing	10% 0300 + 10% H2SO4	15 min.	101	outside	926	513	5	2,631	50	52.6	
						101	inside	537	504	5	200	(6)	***	
# 19	96-071	3/28/97 96-071	Aluminium Sheet	10% 0300 + 10% H2SO4	15 min.	101	side 1	1,758	1,423	5	7,831	5,738	1.4	
						101	side 2	1,357	869	5	5,325	2,275	2.3	

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF	
# 20	96-071	#1	Aluminium Sheet	10% 0300 + 10% H2SO4	15 min.	101	side 1	1,758	929	5	7,831	2,650	3	
					101	side 2	1,357	796	5	5,325	1,819	2.9		
					101	side 1	1,187	870	5	4,263	2,281	1.9		
					101	side 2	902	723	5	2,481	1,362	1.8		
					10% 0200* + 10% HCl	15 min.	101	side 1	1,187	857	5	4,263	2,200	1.9
							101	side 2	902	564	5	2,481	369	6.7
					* note: gas generation observed during experiment (apparent exothermic reaction)									
# 21	92-075	3/28/97 92-075 #1	Concrete	10% 0300	30 min.	101	Flat side*	1,393	1,284	5	5,550	4,869	1.1	
						101	side 2	570	528	5	406	144	2.8	
						* note: Flat side has some paint								
# 22	92-075	3/28/97 92-075 #2	Concrete	10% 0200 + 10% HCl	30 min.	101	Flat side*	1,661	1,335	5	7,225	5,188	1.4	
						101	side 2	518	593	5	81	550		
							* note: Flat side has some paint							
# 23	94-156	3/31/97 94-156 Lot #1	Titanium tubing	10% 0300 + 10% H2SO4	15 min.	110	outside	734	204	2	8,031	(250)	***	
						110	outside	228	208	2	125	(188)	-0.7	
						110	outside	229	214	2	141	(94)	***	
						110	outside	218	230	2	(31)	156	***	
						110	outside	210	176	2	(156)	(688)	***	
						110	outside	564	196	2	5,375	(375)	***	
						110	outside	510	187	2	4,531	(516)	***	
						110	Average=	385	202	2	2,574	(279)	***	
# 24	95-176	3/31/97 95-176 #1	Stainless Steel (Thin sheet)	10% 0300 + 10% H2SO4	15 min.	110	side 1	825	560	5	1,719	63	27.5	
						110	side 2	4,821	842	5	26,694	1,825	14.6	

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
# 25	94-023	3/31/97 94-023 #1	Stainless Steel Sheet	10% 0300 + 10% H2SO4	15 min.	110	side 1	5,333	2,433	5	29,894	11,769	2.5
						110	side 2	7,771	5,961	5	45,131	33,819	1.3
				10% 0300 + 10% H2SO4 (same)	15 min.	110	side 1	5,333	2,097	5	29,894	9,669	3.1
						110	side 2	7,771	5,537	5	45,131	31,169	1.4
				10% 0300 + 10% H2SO4 (new)	30 min.	110	side 1	5,333	1,483	5	29,894	5,831	5.1
						110	side 2	7,771	2,243	5	45,131	10,581	4.3
				10% 0300 + 10% H2SO4 +3% NiCl2	30 min.	110	side 1	5,333	632	5	29,894	513	58.3
						110	side 2	7,771	1,013	5	45,131	2,894	15.6
				10% 0300 + 10% H2SO4 + 3% NiCl2 (same)	30 min.	110	side 1	5,333	632	5	29,894	513	58.3
						110	side 2	7,771	775	5	45,131	1,406	32.1
# 26	ARF Pb	3/31/97 ARF Pb #1	Lead Sample	10% 0300 + 10% H2SO4	15 min.	110	side 1	7,714	6,935	5	44,775	39,906	1.1
				10% 0300 + 10% H2SO4 (same)	15 min.	110	side 1	7,714	7,059	5	44,775	40,681	1.1
				10% 0300 + 10% H2SO4 (same)	30 min.	110	side 1	7,714	5,909	5	44,775	33,494	1.3
				10% 0200 + 10% HCl	30 min.	110	side 1	7,714	2,231	5	44,775	10,506	4.3
				10% 0200 + 10% HCl (same)	30 min.	110	side 1	7,714	1,914	5	44,775	8,525	5.3
# 27	96-090	3/31/97 96-090	Steel Metal	10% 0300 + 10% H2SO4	7.5 min.	110	side 1	352	217	2	2,063	(47)	***
						110	side 1	231	199	2	172	(328)	***

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
		Lot #2				110	side 1	249	213	2	453	(109)	***
						110	side 1	241	226	2	328	94	3.5
						110	side 1	252	196	2	500	(375)	***
						110	side 1	272	203	2	813	(266)	***
						110	Average=	266	209	2	721	(172)	***
						110	side 2	304	205	2	1,313	(234)	***
						110	side 2	211	235	2	(141)	234	???
						110	side 2	244	186	2	375	(531)	***
						110	side 2	253	204	2	516	(250)	***
						110	side 2	231	191	2	172	(453)	***
						110	side 2	258	204	2	594	(250)	***
						110	Average=	250	204	2	471	(247)	***
# 28	92-075	3/31/97 92-075 #1	Concrete	10% 0100 + KOH	30 min.	110	Flat side	1,892	1,350	5	8,388	5,000	1.7
				10% 0100 + KOH	30 min.	110	Flat side	1,892	1,212	5	8,388	4,138	2
				10% 0100 + KOH	30 min.	110	Flat side	1,892	1,128	5	8,388	3,613	2.3
# 29	94-010	3/26/97	Stainless Steel	Exp # 4 and #11 Train		101	side 1	49,559	1,314	5	306,588	5,056	60.6
		94-010 #1	(exp. 4 & 11)			101	side 2	30,826	1,262	5	189,506	4,731	40.1
				10% 0300 + 10% H2SO4 + 3% NiCl2	30 min.	105	side 1	49,559	798	5	306,463	1,706	179.6
						105	side 2	30,826	1,150	5	189,381	3,906	48.5
# 30	94-010	3/26/97 94-010 #3	Stainless Steel (exp. 8)	Exp # 8 Train		100	side 1	54,331	2,875	5	336,444	14,844	22.7
						100	side 2	28,162	2,629	5	172,888	13,306	13
				10% 0300 + 10% H2SO4 + 3% NiCl2 (exp # 29 sln)	30 min.	105	side 1	54,331	977	5	336,288	2,825	119
						105	side 2	28,162	911	5	172,731	2,413	71.6
# 31	94-010	4/1/97 94-010 #1	Stainless Steel	10% 0300 + 10% H2SO4	30 min.	105	side 1	42,251	1,678	5	260,788	7,206	36.2
			and then	10% 0200	30 min.	105	side 2	45,898	908	5	283,581	2,394	118.5

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				+ 10% HCl									
				10% 0300	30 min.	105	side 1	42,251	1,026	5	260,788	3,131	83.3
				+ 10% H2SO4		105	side 2	45,898	928	5	283,581	2,519	112.6
				+ 3% NiCl2									
# 32	94-010	3/28/97 94-010 #1	Stainless Steel (exp. 16)	Exp # 16 Train		101	side 1	31,606	2,820	5	194,381	14,469	13.4
						101	side 2	53,860	1,905	5	333,469	8,750	38.1
				10% 0300		105	side 1	31,606	480	5	194,256	(281)	***
				+ 10% H2SO4	30 min.	105	side 2	53,860	541	5	333,344	100	3333.4
				+ 3% NiCl2									
				(exp. #30 sln)									
# 33	94-010	3/28/97 94-010 #2	Stainless Steel (exp. 17)	Exp # 17 Train		101	side 1*	5,639	2,718	5	32,088	13,831	2.3
						101	side 2	39,834	1,862	5	245,806	8,481	29
				10% 0300		105	side 1*	5,639	2,901	5	31,963	14,850	2.2
				+ 10% H2SO4	30 min.	105	side 2	39,834	1,481	5	245,681	5,975	41.1
				+ 3% NiCl2									
				(exp. #32 sln)									
				* note: side1 was covered with a sputter deposit									
# 34	92-075	4/2/97 92-075 #1	Concrete	10% 0300	30 min.	99	flat side	1,057	735	5	3,513	1,500	2.3
				+ 10% H2SO4									
			and then	10% 0200	30 min.								
			and then	+ 10% HCl									
				DI	30 min.								
				10% 0100	30 min.	99	flat side	1,057	728	5	3,513	1,456	2.4
				+ KOH									
			and then	DI	30 min.								
# 35	94-023	4/2/97 94-023 #1	Stainless Steel	10% 0200	30 min.	99	side 1	7,178	583	5	41,769	550	75.9
				+ 10% HCl		99	side 2	8,258	670	5	48,519	1,094	44.4
			and then	10% 0300	30 min.								
				+ 10% H2SO4									

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
# 36	94-010	4/2/97 94-010	Stainless Steel	10% 0200 + 10% HCl (exp 35 sln)	30 min.	99	side 1	65,142	816	5	404,044	2,006	201.4
				10% 0300 + 10% H2SO4 (exp 35 sln)	30 min.	99	side 2	27,112	1,760	5	166,356	7,906	21
		#1	and then										
				10% 0300 + 10% H2SO4	30 min.	99	side 1	65,142	563	5	404,044	425	950.7
				+ 3% NiCl2		99	side 2	27,112	576	5	166,356	506	328.6
# 37	94-010	4/2/97 94-010	Stainless Steel	10% 0200 + 10% HCl (exp 36 sln)	30 min.	99	side 1	44,132	771	5	272,731	1,725	158.1
				10% 0300 + 10% H2SO4 (exp 36 sln)	30 min.	99	side 2	58,045	841	5	359,688	2,163	166.3
		#2	and then										
				10% 0300 + 10% H2SO4	30 min.	99	side 1	44,132	523	5	272,731	175	1558.5
				+ 3% NiCl2 (exp 36 sln)		99	side 2	58,045	605	5	359,688	688	523.2
# 38	94-010	4/2/97 94-010	Stainless Steel	10% 0200 + 10% HCl (exp 37 sln)	30 min.	99	side 1	54,544	1,058	5	337,806	3,519	96
				10% 0300 + 10% H2SO4 (exp 37 sln)	30 min.	99	side 2	37,864	1,170	5	233,556	4,219	55.4
		#3	and then										
				10% 0300 + 10% H2SO4	30 min.	99	side 1	54,544	618	5	337,806	769	439.4
				+ 3% NiCl2 (exp 37 sln)		99	side 2	37,864	709	5	233,556	1,338	174.6
# 39	95-176	4/2/97 95-176 #1	Stainless Steel Sheet (thin)	10% 0300 + 10% H2SO4	5 min.	99	side 1	4,099	1,313	5	22,525	5,113	4.4
							side 2	765	565	5	4,781	3,531	1.4
				10% 0300 + 10% H2SO4	5 min.	99	side 1	4,099	1,142	5	22,525	4,044	5.6
							side 2	765	565	5	4,781	3,531	1.4

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				(same)									
				10% 0300 + 10% H2SO4	5 min.	99	side 1	4,099	1,078	5	22,525	3,644	6.2
				(same)			side 2	765	624	5	4,781	3,900	1.2
# 40	94-010	4/3/97 94-010 #1	Stainless Steel	10% 0300 + 10% H2SO4	30 min.	113	side 1	40,506	1,588	5	249,631	6,394	39
			and then	10% 0200 + 10% HCl	30 min.	113	side 2	56,574	1,573	5	350,056	6,300	55.6
				10% 0300 + 10% H2SO4	30 min.	113	side 1	40,506	643	5	249,631	487	512.1
				+ 3% NiCl2		113	side 2	56,574	1,117	5	350,056	3,450	101.5
# 41	94-010	4/3/97 94-010	Stainless Steel	10% 0300 + 10% H2SO4	30 min.	113	side 1	32,946	2,153	5	202,381	9,925	20.4
		#2	and then	(exp #40 sln) 10% 0200 + 10% HCl	30 min.	113	side 2	46,966	1,517	5	290,006	5,950	48.7
				(exp #40 sln) 10% 0300 + 10% H2SO4	30 min.	113	side 1	32,946	513	5	202,381	(325)	***
				+ 3% NiCl2		113	side 2	46,966	576	5	290,006	69	4218.3
# 42	94-010	4/3/97 94-010	Stainless Steel	10% 0300 + 10% H2SO4	30 min.	113	side 1	36,040	1,605	5	221,719	6,500	34.1
		#3	and then	(exp #40 sln) 10% 0200 + 10% HCl	30 min.	113	side 2	48,833	1,616	5	301,675	6,569	45.9
				(exp #40 sln) 10% 0300 + 10% H2SO4	30 min.	113	side 1	36,040	568	5	221,719	19	11825
				+ 3% NiCl2		113	side 2	48,833	595	5	301,675	188	1608.9
# 43	97-009	4/3/97 97-009	Lead Sample	10% 0300 + 10% H2SO4	15 min.	113	side 1	769	598	5	1,275	206	6.2
						113	side 2	889	598	5	2,025	206	9.8

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
		#1		10% 0300 + 10% H2SO4 (same)	15 min.	113 113		769 889	659 584	5 5	1,275 2,025	588 119	2.2 17.1
				10% 0300 + 10% H2SO4 (same)	15 min.	113 113	side 1 side 2	769 889	590 602	5 5	1,275 2,025	156 231	8.2 8.8
# 44	97-009	4/7/97 97-009 #1	Lead Sample	10% 0300 + 10% H2SO4	30 min.	102 102	side 1 side 2	20,428 949	608 582	5 5	124,488 2,744	612 450	203.2 6.1
				and then 10% 0200 + 10% HCl and then DI	30 min.								
				10% 0100 + KOH and then DI	30 min.	102 102	side 1 side 2	20,428 949	648 560	5 5	124,488 2,744	862 313	144.3 8.8
# 45	94-225	4/7/97 94-225 #1	Aluminium	10% 0300 + 10% H2SO4	30 min.	102 102	side 1 side 2	6,121 4,117	499 542	5 5	35,069 22,544	(69) 200	*** 112.7
				and then 10% 0200 + 10% HCl *	30 min.								
				and then DI									
				* gas and foam generation									
# 46	96-071	4/7/97 96-071 #1	APG Steel (impact target?)	10% 0300 + 10% H2SO4	30 min.	102	side 1* side 1 alpha** 220 cpm	145,671	52,216	5	907,256	323,163	2.8
				and then 10% 0200 + 10% HCl and then DI	30 min.								
				10% 0100 + KOH	30 min.	102	side 1	145,671	21,542	5	907,256	131,450	6.9

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				and then DI	30 min.		side 1 alpha 50 cpm * side 1 has sever slag/sputter deposits ** efficiency alpha 10.3% ** area alpha 50 cm3 ** background alpha < 1 cpm						
# 47	94-010	4/9/97	Stainless steel	10% 0300	30 min.	102	#1 side 1	15,507	222	2	239,109	281	850.2
		94-010	fuel rack	+ 10% H2SO4		102	#1 side 2	23,557	320	2	364,891	1,813	201.3
		lot #1	same sln expts	and then 10% 0200	30 min.	102	#2 side 1	19,658	514	2	303,969	4,844	62.8
			set #1	+ 10% HCl		102	#2 side 2	25,467	386	2	394,734	2,844	138.8
			side 1 = slag side	and then 10% 0300	30 min.	102	#3 side 1	1,538	321	2	20,844	1,828	11.4
				+ 10% H2SO4 + 3% NiCL2		102	#3 side 2	2,484	264	2	35,625	938	38
# 48	94-010	4/9/97	Stainless steel	10% 0300	30 min.	102	#4 side 1	14,953	1,807	2	230,453	25,047	9.2
		94-010	fuel rack	+ 10% H2SO4		102	#4 side 2	25,646	2,145	2	397,531	30,328	13.1
		lot #1	same sln expts										
			set #2	10% 0200	30 min.	102	#4 side 1	14,953	481	2	230,453	4,328	53.2
			side 1 = slag side	+ 10% HCl		102	#4 side 2	25,646	968	2	397,531	11,938	33.3
				10% 0300	30 min.	102	#4 side 1	14,953	216	2	230,453	188	1229.1
				+ 10% H2SO4 + 3% NiCL2		102	#4 side 2	25,646	204	2	397,531	-	***
# 49	94-010	4/9/97	Stainless steel	10% 0300	30 min.	102	#5 side 1	19,378	208	2	299,594	63	4793.5
		94-010	fuel rack	+ 10% H2SO4		102	#5 side 2	24,093	220	2	373,266	250	1493.1
		lot #1	same sln expts	and then 10% 0200	30 min.	102	#6 side 1	16,553	372	2	255,453	2,625	97.3

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
# 50	94-010	4/9/97 94-010 lot #1	set #3 side 1 = slag side Stainless steel fuel rack same sln expts set #4 side 1 = slag side	+ 10% HCl	30 min.	102	#6 side 2	28,573	296	2	443,266	1,438	308.4
				and then 10% 0300		102	#7 side 1*	5,159	1,766	2	77,422	24,406	3.2
				+ 10% H2SO4 + 3% NiCL2	30 min.	102	#7 side 2	17,030	494	2	262,906	4,531	58
				* severe slag deposits (75% of area)									
				10% 0300		102	#8 side 1*	11,694	3,673	2	179,531	54,203	3.3
				+ 10% H2SO4		102	#8 side 2	47,126	3,306	2	733,156	48,469	15.1
				10% 0200	30 min.	102	#8 side 1	11,694	2,094	2	179,531	29,531	6.1
				+ 10% HCl		102	#8 side 2	47,126	931	2	733,156	11,359	64.5
				10% 0300	30 min.	102	#8 side 1	11,694	644	2	179,531	6,875	26.1
				+ 10% H2SO4		102	#8 side 2	47,126	287	2	733,156	1,297	565.3
+ 3% NiCL2	* slag deposits (50% of area)												
# 51	94-010	4/9/97 94-010 lot #1	Stainless steel fuel rack same sln expts set #5 side 1 = slag side	10% 0300	30 min.	102	#9 side 1	12,609	306	2	193,828	1,594	121.6
				+ 10% H2SO4	102	#9 side 2	40,536	631	2	630,188	6,672	94.5	
				and then 10% 0200	102	#10 side 1	1,423	289	2	19,047	1,328	14.3	
				+ 10% HCl	102	#10 side 2	2,006	290	2	28,156	1,344	21	
				and then 10% 0300	102	#11 side 1	1,579	207	2	21,484	47	458.3	
				+ 10% H2SO4	102	#11 side 2	2,104	262	2	29,688	906	32.8	
				+ 3% NiCL2									
# 52	94-010	4/9/97 94-010 lot #1	Stainless steel fuel rack same sln expts set #6 side 1 = slag side	10% 0300	30 min.	102	#12 side 1	17,008	8,981	2	262,563	137,141	1.9
				+ 10% H2SO4	102	#12 side 2	4,071	2,573	2	60,422	37,016	1.6	
				10% 0200	30 min.	102	#12 side 1	17,008	6,468	2	262,563	97,875	2.7
				+ 10% HCl	102	#12 side 2	4,071	1,078	2	60,422	13,656	4.4	
				10% 0300	30 min.	102	#12 side 1	17,008	2,827	2	262,563	40,984	6.4

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				+ 10% H2SO4 + 3% NiCL2		102	#12 side 2	4,071	708	2	60,422	7,875	7.7
# 53	94-010	4/9/97 94-010 lot #1	Stainless steel fuel rack same sln expts set #7 side 1 = slag side	10% 0300 + 10% H2SO4	30 min.		97 #13 side 1	28,829	4,994	2	447,422	75,000	6
							97 #13 side 2	20,380	9,682	2	315,406	148,250	2.1
				10% 0200 + 10% HCl	30 min.		97 #13 side 1	28,829	2,469	2	447,422	35,547	12.6
							97 #13 side 2	20,380	4,498	2	315,406	67,250	4.7
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.		97 #13 side 1	28,829	1,455	2	447,422	19,703	22.7
							97 #13 side 2	20,380	3,761	2	315,406	55,734	5.7
# 54	94-010	4/9/97 94-010 lot #1	Stainless steel fuel rack same sln expts set #7 side 1 = slag side (set #6 filtered)	10% 0300 + 10% H2SO4	30 min.		97 #14 side 1	8,669	4,687	2	132,422	70,203	1.9
							97 #14 side 2	55,235	4,422	2	860,016	66,063	13
				10% 0200 + 10% HCl	30 min.		97 #14 side 1	8,669	3,602	2	132,422	53,250	2.5
							97 #14 side 2	55,235	2,380	2	860,016	34,156	25.2
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.		97 #14 side 1	8,669	3,266	2	132,422	48,000	2.8
							97 #14 side 2	55,235	2,477	2	860,016	35,672	24.1
# 55	96-032	4/10/97 96-032 #1	steel (alpha)	10% 0300 + 10% H2SO4	30 min.		97 side 1	696	514	5	1,319	181	7.3
							97 side 2	1,127	726	5	4,013	1,506	2.7
							side 2 alpha	140 cpm	62 cpm				
				10% 0200 + 10% HCl	30 min.		97 side 1	696	488	5	1,319	19	70.3
							97 side 2	1,127	558	5	4,013	456	8.8
							side 2 alpha	140 cpm	27 cpm				
# 56	96-032	4/10/97 96-032 #2	steel (alpha)	10% 0300 + 10% H2SO4	30 min.		97 side 1	568	432	5	519	(331)	***
							97 side 2	1,339	743	5	5,338	1,612	3.3
							side 2 alpha	240 cpm	51 cpm				
				10% 0200 + 10% HCl	30 min.		97 side 1	568	501	5	519	100	5.2
							97 side 2	1,339	512	5	5,338	169	31.6

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm) 240 cpm	Final (cpm) 32 cpm	Cnt (min)	Initial (dpm)	Final (dpm)	DF
							side 2 alpha						
# 57	96-003	5/19/97 96-003 lot #1	Stainless steel filter screen same sln expts #1	10% 0300 + 10% H2SO4	30 min.	104	side 1	7,758	316	2	117,975	1,694	69.7
						104	side 2	8,119	618	2	123,616	6,413	19.3
				10% 0200 + 10% HCl	30 min.	104	side 1	7,758	217	2	117,975	147	803.2
						104	side 2	8,119	416	2	123,616	3,256	38
				10% 0300 + 10% H2SO4	30 min.	104	side 1	7,758	173	2	117,975	(541)	***
				+ 3% NiCL2		104	side 2	8,119	187	2	123,616	(322)	***
# 58	96-003	5/19/97 96-003 lot #1	Aluminium sheet same sln expts #2	10% 0300 + 10% H2SO4	30 min.	104	side 1	6,274	491	2	94,788	4,428	21.4
						104	side 2	1,873	225	2	26,022	272	95.7
				10% 0200* + 10% HCl	20 min.	104	side 1	6,274	254	2	94,788	725	130.7
				* gas generation observed sln foamed severely		104	side 2	1,873	230	2	26,022	350	74.3
				10% 0300** + 10% H2SO4	20 min.	104	side 1	6,274	206	2	94,788	(25)	***
				+ 3% NiCL2 *gas generation observed sln foamed severely		104	side 2	1,873	235	2	26,022	428	60.8
# 59	96-003	5/19/97 96-003 lot #1	machine part (steel) same sln expts #3	10% 0300 + 10% H2SO4	30 min.	104	side 1	3,305	1,367	2	48,397	18,116	2.7
						104	side 2	339	383	2	2,053	2,741	0.7
				10% 0200 + 10% HCl	30 min.	104	side 1	3,305	650	2	48,397	6,913	7
						104	side 2	339	315	2	2,053	1,678	1.2
				10% 0300 + 10% H2SO4	30 min.	104	side 1	3,305	468	2	48,397	4,069	11.9
						104	side 2	339	229	2	2,053	334	6.1

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				+ 3% NiCL2									
# 60	97-038	5/20/97 97-038 lot #1 set #1	Lead samples same sln expts #1 430 g	10% 0300 + 10% H2SO4	30 min.	105	side 1	25,891	16,990	2	401,281	262,203	1.5
						105	side 2	2,496	2,094	2	35,734	29,453	1.2
				10% 0200 + 10% HCl	30 min.	105	side 1	25,891	276	2	401,281	1,047	383.3
						105	side 2	2,496	298	2	35,734	1,391	25.7
# 61	97-038	5/20/97 97-038 lot #1 set #1	Lead samples same sln expts #2 431 g	10% 0300 + 10% H2SO4	30 min.	105	side 1	77,029	47,148	2	1,200,313	733,422	1.6
						105	side 2	1,560	1,275	2	21,109	16,656	1.3
				10% 0200 + 10% HCl	30 min.	105	side 1	77,029	1,596	2	1,200,313	21,672	55.4
						105	side 2	1,560	400	2	21,109	2,984	7.1
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.	105	side 1	77,029	1,247	2	1,200,313	16,219	74
						105	side 2	1,560	425	2	21,109	3,375	6.3
				10% 0200 + 10% HCl (new)	30 min.	105	side 1	77,029	372	2	1,200,313	2,547	471.3
						105	side 2	1,560	229	2	21,109	313	67.6
# 62	97-038	5/20/97 97-038 lot #1 set #1	Lead samples same sln expts #3 451 g	10% 0300 + 10% H2SO4	30 min.	105	side 1	173,667	137,569	2	2,710,281	2,146,250	1.3
						105	side 2	7,362	2,711	2	111,766	39,094	2.9
				10% 0200 + 10% HCl	30 min.	105	side 1	173,667	3,350	2	2,710,281	49,078	55.2
						105	side 2	7,362	714	2	111,766	7,891	14.2
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.	105	side 1	173,667	2,586	2	2,710,281	37,141	73
						105	side 2	7,362	794	2	111,766	9,141	12.2
				10% 0200 + 10% HCl (new)	30 min.	105	side 1	173,667	481	2	2,710,281	4,250	637.7
						105	side 2	7,362	302	2	111,766	1,453	76.9
# 63	97-038	5/20/97 97-038 lot #1 set #1	Lead samples same sln expts #4 442 g	10% 0300 + 10% H2SO4	30 min.	105	side 1	176,688	127,661	2	2,757,484	1,991,438	1.4
						105	side 2	4,638	3,997	2	69,203	59,188	1.2
				10% 0200 + 10% HCl	30 min.	105	side 1	176,688	7,852	2	2,757,484	119,422	23.1
						105	side 2	4,638	427	2	69,203	3,406	20.3

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.	105	side 1	176,688	6,109	2	2,757,484	92,188	29.9
						105	side 2	4,638	400	2	69,203	2,984	23.2
				10% 0200 + 10% HCl (new)	30 min.	105	side 1	176,688	2,985	2	2,757,484	43,375	63.6
						105	side 2	4,638	333	2	69,203	1,938	35.7
				20% 0300 (new)	30 min.	100	side 1	965	351	2	11,944	2,350	5.1
						100	side 2	24,828	3,194	2	384,803	46,772	8.2
# 64	97-038	5/20/97 97-038 lot #1 set #2	Lead samples same sln expts #5 495 g	10% 0200 (new) + 10% HCl	30 min.	100	side 1	965	324	2	11,944	1,928	6.2
						100	side 2	24,828	1,390	2	384,803	18,584	20.7
				20% 0300	30 min.	105	side 1	23,867	608	2	369,656	6,234	59.3
						105	side 2	49,710	813	2	773,453	9,438	82
# 65	97-038	5/20/97 97-038 lot #1 set #2	Lead samples same sln expts #6 300 g	10% 0200 + 10% HCl	30 min.	105	side 1	23,867	352	2	369,656	2,234	165.4
						105	side 2	49,710	313	2	773,453	1,625	476
				20% 0300	30 min.	105	side 1	5,687	351	2	85,594	2,219	38.6
						105	side 2	11,327	326	2	173,719	1,828	95
(# 67)	97-038	5/20/97 97-038 lot #1 set #2	Lead samples same sln expts #7 495 g	20% 0300	30 min.	105	side 1	11,679	419	2	179,219	3,281	54.6
						105	side 2	21,861	252	2	338,313	672	503.5
				20% 0300	30 min.	105	side 1	11,679	419	2	179,219	3,281	54.6
						105	side 2	21,861	252	2	338,313	672	503.5
(# 69)	97-038	5/20/97 97-038 lot #1 set #2	Lead samples same sln expts #8 354 g	20% 0300	30 min.	105	side 1	11,679	419	2	179,219	3,281	54.6
						105	side 2	21,861	252	2	338,313	672	503.5
				20% 0300	30 min.	105	side 1	11,679	419	2	179,219	3,281	54.6
						105	side 2	21,861	252	2	338,313	672	503.5
# 70	97-038	5/20/97 97-038 lot #1 set #2	Lead samples same sln expts #9	20% 0300	30 min.	105	side 1	2,174	301	2	30,703	1,438	21.4
						105	side 2	3,725	300	2	54,938	1,422	38.6
				20% 0300	30 min.	105	side 1	2,174	301	2	30,703	1,438	21.4
						105	side 2	3,725	300	2	54,938	1,422	38.6

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF	
			396 g											
# 72)	97-038	5/20/97	Lead samples	20% 0300	30 min.	105	side 1	3,820	518	2	56,422	4,828	11.7	
		97-038 lot #1 set #2	same sln expts #10 489 g			105	side 2	1,140	288	2	14,547	1,234	11.8	
# 74)	97-038	5/20/97	Lead samples	20% 0300	30 min.	105	side 1	3,971	994	2	58,781	12,266	4.8	
		97-038 lot #1 set #2	same sln expts #11 446 g			105	side 2	692	293	2	7,547	1,313	5.8	
# 76)	97-038	5/20/97	Lead samples	20% 0300	30 min.	105	side 1	2,066	366	2	29,016	2,453	11.8	
		97-038 lot #1 set #2	same sln expts #12 470 g			105	side 2	810	232	2	9,391	359	26.1	
# 66)	95-053	5/20/97	Stainless steel	10% 0300	30 min.	100	#1 'top'	26,229	667	2	406,694	7,288	55.8	
		95-053 lot #1 set #1	heat exchanger tube sections same sln expts #1-3	+ 10% H2SO4		100	#1 'bottom'	1,701	882	2	23,444	10,647	2.2	
						100	#2 'top'	1,924	286	2	26,928	1,334	20.2	
						100	#2 'bottom'	1,752	226	2	24,241	397	61.1	
						100	#3 'top'	2,364	240	2	33,803	616	54.9	
						100	#3 'bottom'	940	240	2	11,553	616	18.8	
					10% 0300	30 min.	100	#1 'top'	26,229	203	2	406,694	38	10845.2
					+ 10% H2SO4 + 3% NiCl2		100	#1 'bottom'	1,701	194	2	23,444	(103)	***
		95-053 lot #1 set #2	heat exchanger tube sections same sln expts	+ 10% H2SO4		100	#4 'top'	1,928	210	2	26,991	147	183.8	
	100				#4 'bottom'	3,084	248	2	45,053	741	60.8			
	100				#5 'top'	3,256	194	2	47,741	(103)	***			
	100				#5 'bottom'	3,683	196	2	54,413	(72)	***			

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
			#4-6			100	#6 'top'	1,277	239	2	16,819	600	28
						100	#6 'bottom'	1,639	198	2	22,475	(41)	***
(# 71)	95-053	5/20/97	Stainless steel	10% 0300	30 min.	100	#7 'top'	1,725	219	2	23,819	288	82.8
		95-053	heat exchanger	+ 10% H2SO4		100	#7 'bottom'	2,347	199	2	33,538	(25)	***
		lot #1	tube sections			100	#8 'top'	964	233	2	11,928	506	23.6
		set #3	same sln expts			100	#8 'bottom'	1,532	201	2	20,803	6	3328.5
			#7-9			100	#9 'top'	778	208	2	9,022	116	78
						100	#9 'bottom'	705	225	2	7,881	381	20.7
(# 73)	95-053	5/20/97	Stainless steel	10% 0300	30 min.	100	#10 'top'	1,286	224	2	16,959	366	46.4
		95-053	heat exchanger	+ 10% H2SO4		100	#10 'bottom'	750	213	2	8,584	194	44.3
		lot #1	tube sections			100	#11 'top'	1,066	228	2	13,522	428	31.6
		set #4	same sln expts			100	#11 'bottom'	1,128	251	2	14,491	788	18.4
			#10-12			100	#12 'top'	752	199	2	8,616	(25)	***
						100	#12 'bottom'	1,220	252	2	15,928	803	19.8
(# 75)	95-053	5/20/97	Stainless steel	10% 0300	30 min.	100	#13 'top'	1,013	224	2	12,694	366	34.7
		95-053	heat exchanger	+ 10% H2SO4		100	#13 'bottom'	2,356	226	2	33,678	397	84.9
		lot #1	tube sections			100	#14 'top'	691	216	2	7,663	241	31.8
		set #5	same sln expts			100	#14 'bottom'	1,291	265	2	17,038	1,006	16.9
			#13-15			100	#15 'top'	902	200	2	10,959	(9)	***
						100	#15 'bottom'	1,995	198	2	28,038	(41)	***
(# 79)	95-053	5/20/97	Stainless steel	10% 0300	30 min.	96	#16 'top'	653	218	2	7,203	406	17.7
		95-053	heat exchanger	+ 10% H2SO4		96	#16 'bottom'	869	226	2	10,578	531	19.9
		lot #1	tube sections			96	#17 'top'	2,408	391	2	34,625	3,109	11.1
		set #6	same sln expts			96	#17 'bottom'	911	222	2	11,234	469	24
			#16-18			96	#18 'top'	1,102	241	2	14,219	766	18.6
						96	#18 'bottom'	1,580	210	2	21,688	281	77.1
# 80	95-053	5/20/97	Stainless steel	10% 0300	30 min.	96	#19 'top'	1,722	263	2	23,906	1,109	21.5
		95-053	heat exchanger	+ 10% H2SO4		96	#19 'bottom'	1,443	265	2	19,547	1,141	17.1
		lot #1	tube sections			96	#20 'top'	1,072	288	2	13,750	1,500	9.2
		set #7	same sln expts			96	#20 'bottom'	1,604	242	2	22,063	781	28.2
			#19-21			96	#21 'top'	1,164	208	2	15,188	250	60.8
						96	#21 'bottom'	1,963	206	2	27,672	219	126.5

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
# 86)	95-053	5/20/97 95-053 lot #1 set #8	Stainless steel	10% 0300	30 min.	96 #22 'top'		1,247	281	2	16,484	1,391	11.9
			heat exchanger	+ 10% H2SO4		96 #22 'bottom'		1,352	206	2	18,125	219	82.9
			tube sections			96 #23 'top'		522	251	2	5,156	922	5.6
			same sln expts			96 #23 'bottom'		1,752	482	2	24,375	4,531	5.4
			#22-24			96 #24 'top'		622	216	2	6,719	375	17.9
						96 #24 'bottom'		957	326	2	11,953	2,094	5.7
# 87	95-053	5/20/97 95-053 lot #1 set #9	Stainless steel	10% 0300	30 min.	96 #25 'top'		1,035	204	2	13,172	188	70.3
			heat exchanger	+ 10% H2SO4		96 #25 'bottom'		1,731	211	2	24,047	297	81
			tube sections			96 #26 'top'		588	237	2	6,188	703	8.8
			same sln expts			96 #26 'bottom'		900	274	2	11,063	1,281	8.6
			#25-27			96 #27 'top'		2,635	213	2	38,172	328	116.3
						96 #27 'bottom'		3,192	249	2	46,875	891	52.6
# 88)	95-053	5/20/97 95-053 lot #1 set #10	Stainless steel	10% 0300	30 min.	96 #28 'top'		612	242	2	6,563	781	8.4
			heat exchanger	+ 10% H2SO4		96 #28 'bottom'		1,237	263	2	16,328	1,109	14.7
			tube sections			96 #29 'top'		625	224	2	6,766	500	13.5
			same sln expts			96 #29 'bottom'		1,321	387	2	17,641	3,047	5.8
			#28-30			96 #30 'top'		1,082	211	2	13,906	297	46.8
						96 #30 'bottom'		1,490	222	2	20,281	469	43.3
# 77)	96-???	5/20/97 96-???	Stainless steel	10% 0300	30 min.	96 #1 side 1		16,927	1,312	2	261,484	17,500	14.9
			fuel rack	+ 10% H2SO4		96 #1 side 2		1,757					
			same sln expts										
			#1 (ID #7)	10% 0200	30 min.	96 #1 side 1		16,927	602	2	261,484	6,406	40.8
			(slag side =	+ 10% HCl									
			side #2)										
# 78	96-???	5/21/97 96-???	Stainless steel	10% 0300	30 min.	96 #1 side 1		16,627	2,044	2	256,797	28,938	8.9
			fuel rack	+ 10% H2SO4		96 #1 side 2		2,269					
			same sln expts										
			#2 (ID #6)	10% 0200	30 min.	96 #1 side 1		16,627	843	2	256,797	10,172	25.2
			(slag side =	+ 10% HCl									
			side #2)										

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.	96	#1 side 1	16,627	227	2	256,797	547	469.6
						96	#1 side 2	2,269	667	2	32,453	7,422	4.4
# 81)	96-???	5/21/97 96-??? lot #1	Stainless steel fuel rack same sln expts #3 (ID #5) (slag side = side #2)	10% 0300	30 min.		96 #1 side 1	17,760	4,146	2	274,500	61,781	4.4
				+ 10% H2SO4			96 #1 side 2	2,503					
				10% 0200 + 10% HCl	30 min.		96 #1 side 1	17,760	1,685	2	274,500	23,328	11.8
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.		96 #1 side 1 96 #1 side 2	17,760 2,503	1,105 492	2 2	274,500 36,109	14,266 4,688	19.2 7.7
# 82	96-???	5/21/97 96-??? lot #1	Stainless steel fuel rack same sln expts #4 (ID #4) (slag side = side #2)	10% 0300 + 10% H2SO4	30 min.		96 #1 side 1 96 #1 side 2	16,704 1,479	3,515	2	258,000	51,922	5
				10% 0200 + 10% HCl	30 min.		96 #1 side 1	16,704	967	2	258,000	12,109	21.3
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.		96 #1 side 1 96 #1 side 2	16,704 1,479	262 352	2 2	258,000 20,109	1,094 2,500	235.9 8
# 83	96-???	5/21/97 96-??? lot #1	Stainless steel fuel rack same sln expts #5 (ID #3) (slag side = side #2)	10% 0300 + 10% H2SO4	30 min.		96 #1 side 1 96 #1 side 2	14,263 2,332	3,489	2	219,859	51,516	4.3
				10% 0200 + 10% HCl	30 min.		96 #1 side 1	14,263	1,476	2	219,859	20,063	11
				10% 0300 + 10% H2SO4 + 3% NiCL2	30 min.		96 #1 side 1 96 #1 side 2	14,263 2,332	343 591	2 2	219,859 33,438	2,359 6,234	93.2 5.4
# 84	96-???	5/21/97 96-??? lot #1	Stainless steel fuel rack same sln expts #6 (ID #2) (slag side = side #2)	10% 0300 + 10% H2SO4	30 min.		96 #1 side 1 96 #1 side 2	20,967 1,838	5,111	2	324,609	76,859	4.2
				10% 0200 + 10% HCl	30 min.		96 #1 side 1	20,967	2,465	2	324,609	35,516	9.1

Exp.	Sample	Sample ID	Substrate	Treatment	Time	Bkgrnd (cpm)	Location	Initial (cpm)	Final (cpm)	Cnt (min)	Initial (dpm)	Final (dpm)	DF
				10% 0300* + 10% H2SO4 + 3% NiCL2 *sample discolored	30 min.	96	#1 side 1	20,967	1,418	2	324,609	19,156	16.9
						96	#1 side 2	1,838	1,084	2	25,719	13,938	1.8
# 85	96-???	5/21/97 96-??? lot #1	Stainless steel fuel rack same sln expts #7 (ID #1) (slag side = side #2)	10% 0300 + 10% H2SO4	30 min.	96	#1 side 1	13,266	4,422	2	204,281	66,094	3.1
				10% 0200 + 10% HCl	30 min.	96	#1 side 1	13,266	1,841	2	204,281	25,766	7.9
				10% 0300* + 10% H2SO4 + 3% NiCL2 *sample highly discolored (reddish hue)	30 min.	96	#1 side 1	13,266	2,147	2	204,281	30,547	6.7
						96	#1 side 2	1,641	863	2	22,641	10,484	2.2