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LIST OF ACRONYMS

BSI.....	Bartlett Services, Inc.
DF	decontamination factor
DPA.....	digital pixel analysis
ESI.....	Entropic Systems Inc.
GAC.....	granular activated carbon
HEPA	high efficiency particulate air
IRAD	Independent research and development
LET	linear energy transfer
LLW.....	low-level waste
Mrad.....	Mega (million) rad
MIT.....	Massachusetts Institute of Technology
MITNRL	Massachusetts Institute of Technology Neutron Research Laboratory
NAA.....	neutron activation analysis
PFC.....	perfluorocarbon
PLC.....	programmable logic controller
SNAP.....	Significant New Alternatives Program
SRS.....	Savannah River Site
TRU	Transuranic
NRC.....	U.S. Nuclear Regulatory Commission
WAC.....	Waste Acceptance Criteria
VOC.....	volatile organic compound
WIPP	Waste Isolation Pilot Plant
WSRC.....	Westinghouse Savannah River Company

ABSTRACT

The Sonatol process uses ultrasonic agitation in fluorinated surfactant solutions to remove radioactive particles from surfaces. Filtering the suspended particles allows the solutions to be reused indefinitely. The current work applies the Sonatol process to the decontamination of heterogeneous legacy Pu-238 waste that exhibits excessive hydrogen gas generation, which prevents transportation of the waste to the Waste Isolation Pilot Plant. Bartlett Services, Inc. (BSI) designed and fabricated a prototype decontamination system within a replica of a Savannah River Site glovebox. In Phase I, BSI conducted cold testing with surrogate waste material to verify that the equipment, operating procedures, and test protocols would support testing with Pu-238 in Phase II.

The surrogate waste material is representative of known constituents of legacy job control waste. Two sub-micron sized Pu-238 simulants were added to the surrogate waste so that decontamination could be tested. The first simulant was an Osram Sylvania Phosphor 2284C powder that fluoresces under ultraviolet light. The use of the fluorescent simulant allows rapid, inexpensive system startup testing because residuals can be assayed using a digital camera. The results of digital pixel analysis (DPA) are available immediately and do not require use of licensed material. The second simulant, which was used for integrated cold testing, was a cerium oxide powder that was activated in a research reactor neutron flux and assayed by photon spectroscopy.

The surrogate transuranic (TRU) waste material was contaminated with Pu-238 simulants and loaded into the cleaning chamber, where the surrogates were ultrasonically agitated and rinsed. The decontaminated materials were then assayed for surface contamination by DPA to establish optimum operating parameters and provide process quality control. Selected samples were sent to the Massachusetts Institute of Technology for neutron activation analysis (NAA). NAA testing resulted in weighted average decontamination factors (DFs) in the range of 125 to 157 for the surrogate waste mixtures. The weighted DFs for the organic portion of the surrogate waste mixtures ranged from 66 to 140. The NAA DF for inorganic material was 370.

Other than the removal of particulate contamination, the processed samples were unchanged by decontamination. Most NAA samples were irradiated after decontamination. However, several samples were irradiated in the reactor core prior to decontamination in order to investigate the possible interference of radiation induced imbedding of particles in organic materials. The radiation dose was in excess of 110 Mrad. The NAA DF for samples irradiated before decontamination was six.

EXECUTIVE SUMMARY

INTRODUCTION

The United States Department of Energy's Savannah River Site (SRS), near Aiken, South Carolina, stores Pu-238 contaminated transuranic waste. Portions of this waste cannot be shipped to the Waste Isolation Pilot Plant (WIPP) in TRUPACT-II containers due to excessive generation of hydrogen gas. The gas results from radiolysis of organic constituents in the waste matrix. Decontaminating the organic matrix sufficiently to meet the hydrogen gas concentration limits would permit shipment for disposal at WIPP and eliminate the need for continued storage at SRS. Decontamination is a simple, proven and relatively inexpensive option with a high probability of successful implementation. Treatment of the waste by destroying the organic content is an alternative to decontamination. Other possible answers to the problem are installing hydrogen gas getters in the TRUPACT-II containers or increasing the allowable hydrogen gas limit of the TRUPACT-II. These alternatives are, however, either prohibited or impractical at this time.

Particles that are micron size or smaller generally adhere tenaciously due to secondary valence forces with other solid surfaces, and cannot be removed by simple mechanical means. However, in 1987, Dr. Robert Kaiser of Entropic Systems, Inc. (ESI) demonstrated that contacting particle-contaminated surfaces with a dilute solution of fluorinated surfactant of high molecular weight in an inert perfluorocarbon (PFC) liquid under sonic shear conditions resulted in effective removal of micron or sub-micron sized particles from solid surfaces. The scope of the current project is to design, fabricate and perform non-radioactive testing of a glovebox-sized decontamination system to demonstrate the capability to decontaminate legacy Pu-238 waste (see Figure 1). The principal objectives are to:

- Treat batches of simulated Pu-238 waste in one hour in a glove box;
- Demonstrate that Pu-238 simulant is at least 99% removed by the Sonatol® system;
- Demonstrate 99.9% filtration of Pu-238 simulant from PFC liquid in one pass.

BACKGROUND

The TRU waste consists of heterogeneous job control debris contaminated with Pu-238 oxide. Waste is packaged in the drum in four to six layers of polyvinyl chloride bags. Drums may contain numerous waste packages. Waste contents include polyvinyl chloride, cellulose, metals, and other job control waste, and may contain cans and bottles (both metal and plastic) containing hazardous liquids. None of the indicated

constituents reacts chemically with the Sonatol® cleaning fluid. The Sonatol® process operates as a batch process in a closed system with non-toxic liquids at ambient temperatures and pressures. There is no liquid discharge or gaseous exhaust. The particles become suspended in the PFC fluid, which is then filtered and reused.

The PFC fluids used in the Sonatol® process have zero ozone depletion potential, are nontoxic, nonflammable, and are generally recognized as non-hazardous materials. These liquids have a high specific gravity (s.g. 1.7), low viscosity (less than 1 cP), and a low surface tension (less than 15 dynes/cm), which facilitates wetting and penetration into holes and crevices. PFCs are an environmentally acceptable means of cleaning highly complex material configurations under the Significant New Alternatives Program of the U.S. Environmental Protection Agency.

PROCESS

The surrogate waste is placed in an empty cleaning chamber (see Figure 2). The chamber is first filled through spray jets with a dilute solution (<1 wt-%) of fluorinated surfactant and carrier liquid. The material is agitated ultrasonically and/or mechanically. The ultrasonic subsystem generates waves from 40 to 170 kHz, which maximize removal of a wide range of particle sizes, especially in the micron to nanometer range. The now-contaminated fluid is then drained from the chamber into the storage tank through a filter train. The filter train consists of a stainless steel screen, a 17 µm stainless-steel filter, and a 0.2 µm ceramic filter. The sub-micron Pu-238 particles are removed in the 0.2 µm ceramic filter. Passing the filtered wash fluid through an activated carbon fiber filter generates a rinse that is sprayed over the rotating basket of waste. The above process steps are repeated as required to achieve the requisite decontamination factor. A turbidity meter monitors the solution as it exits the cleaning chamber and allows each step to extend or be repeated as required.

TEST PLAN

The test plan included development of representative surrogate wastes, Pu-238 simulants, and methods to test process parameters to develop optimum decontamination protocols for legacy Pu-238 waste. Surrogate waste materials were developed to represent divergent Pu-238 waste streams (see Figure 3). Surrogates were contaminated with two plutonium oxide simulants. One simulant is a 800-nm diameter phosphor particles; the second consists of 300-nm diameter cerium oxide particles. The phosphor fluoresces intensively at a wavelength of 546 nm (green light) when illuminated with 365 nm ultraviolet light. The fluorescent light emitted from the samples is quantified rapidly and inexpensively by DPA. The cerium oxide used in the experiments is a chemical analogue of plutonium oxide. The waste surrogates were contaminated by spraying with dilute (~1000-ppm) suspensions of simulant powders in a volatile, low surface tension liquid. This liquid has a <1 ppm non-volatile residue. Evaporation of the liquid leaves a uniform surface contamination residue.

The individual DPA samples were analyzed with a high-resolution digital camera to provide immediate scoring of the relative DF during startup testing and parametric testing. For the formal integrated cold tests, surrogate waste samples were analyzed by to the Massachusetts Institute of Technology Neutron Research Laboratory (MITNRL) using neutron activation analysis (NAA). NAA provides identification and quantification of trace quantities of contaminants by activating stable cerium and detecting gamma ray emissions from the resulting isotope.

The effects of several process parameters were examined before carrying out full-scope, cold demonstration trials. The parameters and results included:

- a. *Bath Chemistry:* Successful particle removal only occurred in PFC solutions with at least 0.2 wt-% Krytox 157FS fluorinated surfactant. Increasing the surfactant concentration higher than 0.2 wt-% does not improve DFs.
- b. *Means of Applying Shear:* Spraying surfactant solution onto the samples resulted in minimal or no decontamination. Sonication resulted in significant particle removal at 132 kHz, 104 kHz and 40 kHz. Frequencies of 72 kHz and 170 kHz were not effective in this test environment.
- c. *Time:* There was significant removal in 5 minutes, and nearly complete removal (with favorable surfactant concentration, ultrasonic frequency, etc.) in 10 minutes for polyethylene and aluminum samples. Rubber gloves and porous materials may require longer sonication steps.
- d. *Temperature:* The effect of bath temperature on particle removal was examined from 80°F to 125°F (26°C to 53°C). While the Sonatol® solution appears to sonicate more readily at higher temperature, no significant difference in particle removal efficiency was observed within this temperature range.
- e. *Basket Design:* Basket design criteria for successful decontamination include: minimum basket weight, minimum wall thickness, basket hole size $\geq 6\text{mm}$, open area $> 70\%$, basket length less than ultrasonic transducer spacing, and full submersion in the bath liquid. Low-density objects that float in the PFC were effectively decontaminated without a basket.
- f. *Filtration Efficiency:* The 0.3 micron cerium oxide particles were quantitatively removed ($>99.9\%$) from suspension by the 0.2 μm ceramic filter used in the experiments. The turbidity of the filtrate was indistinguishable from the turbidity of fresh Sonatol® liquid.

Surrogate waste materials were tested with two integrated decontamination protocols. Protocol One employed one continuous period of ultrasonic agitation. Protocol Two added a cleaning chamber drain/fill cycle between two periods of ultrasonic agitation. No consistent difference was observed between the protocols.

RESULTS

The NAA results showed weighted average DFs of 137 to 162 for the SRS representative surrogate waste matrices and the bounding cases (>99% removal). In general, the individual samples subjected to the trials appeared as clean or cleaner than they were prior to contamination. A decontamination test conducted with a 132 kHz ultrasonic generator after a >110 Mrad dose to surrogate rubber glove debris showed 86% removal of the cerium particles from irradiated lead-lined glove samples – as compared to 97% for non-irradiated rubber gloves. Results for individual materials are:

<i>Surrogate Material</i>	<i>DF</i>	<i>Removal</i>
<i>Black polyethylene</i>	<i>284</i>	<i>99.7%</i>
<i>Alcohol towel/wipe</i>	<i>14</i>	<i>93%</i>
<i>Alcohol wipe package</i>	<i>308</i>	<i>99.7%</i>
<i>Lead lined rubber</i>	<i>39</i>	<i>97%</i>
<i>Aluminum</i>	<i>369</i>	<i>99.7%</i>
<i>HEPA filter material</i>	<i>3.5</i>	<i>78%</i>
<i>Black cardboard</i>	<i>6.5</i>	<i>87%</i>
<i>Wood</i>	<i>11</i>	<i>92%</i>
<i>Aged blue poly tarp</i>	<i>161</i>	<i>99.4%</i>
<i>Irradiated lead lined rubber</i>	<i>6.0</i>	<i>86%</i>

DISCUSSION

The results show decontamination removal effectiveness of 78% to 99.7% for samples of organic surrogates in a one-hour process cycle. The removal efficiency for inorganic (metal) items was 99.7%. The weighted average DF for the representative waste matrix proposed by SRS (see table below) is 162. The weighted average DF for SRS bounding cases “A” and “B” are 156 and 137. These matrices are 70% to 82% organic. The weighted average DFs for the organic portions of these waste surrogates are 150, 65, and 168. Longer sonication times and reduced clumping of organic materials may improve the performance results, if greater removal rates are required.

DF Values of Representative SRS Waste Streams					
Case	Content (W-%)	Cold Test DF by NAA	Weighted DF of Surrogate Waste	Weighted DF of Organic Fraction	Weighted DF of Inorganic Fraction
Representative (Avg. Drum)					
iron	10.3	369			
metal	0.3	369			
oil absorbent	7.3	3			
paper/wood	39.6	11			
rubber	0.9	29			
plastic	41.7	284	162	150	220
Bounding Case "A"					
gloves	60	29			
plastic	10	284			
metal	10	369			
can	20	369	156	65	370
Bounding Case "B"					
wipes	30	14			
oil absorbent	20	3			
cans	4	369			
glass	1	369			
plastic	40	284			
HEPA	5	3	137	168	64

Successful results are anticipated in the Phase II hot tests. The Phase I system was operated in a mock-up of the glovebox that would be used in hot testing. Therefore, hot testing procedures would follow established Phase I protocols. The level of radiation that the organic coupons were subjected to in the MIT Nuclear Reactor is much higher than the levels of radiation than are likely to be found in the legacy waste. This indicates that the Sonatol® process removes contaminants from radiation damaged materials, as will be the case in the Phase II hot testing program.

The scale-up from hot testing to production would use established production scale mechanical handling and processing equipment. A full scale system would include automated shredding of the waste, industrial flow-through ultrasonic cleaning systems, enhanced contamination control features, and assay systems to ensure that packages are compliant with transportation requirements.

CONCLUSIONS

Cold tests were performed in which representative mixtures of legacy waste, both organic and inorganic, were decontaminated. Decontamination factors in excess of 100 were achieved with a total process cycle time of 1 hour. The Sonatol® process

demonstrated a weighted DF of 150 for the organic fraction of representative SRS surrogate waste. If a drum of waste with a 15-fold excess of Pu-238/hydrogen gas were processed with a DF of 150, the resulting processed waste would theoretically then exhibit 10% of the transportation limit. This study demonstrated >99.9% capture of the contaminant on a ceramic filter. Tests with radiation aged organic material resulted in 86% contaminant removal. Furthermore, the results indicate:

1. Sonication in solutions of >0.2% fluorinated surfactant in a perfluorocarbon liquid is an effective means of removing particulate contaminants from surrogate wastes.
2. Decontamination is accomplished without dissolving the substrates being cleaned or the contaminants being removed.
3. The contamination can be collected in 100% inorganic filters.
4. The optimum particle removal was achieved at an ultrasonic frequency of 104 kHz.
5. Sonication at any frequency was more effective than spraying or immersion only.
6. Two drain/fill cycles may be better than one, but the data are inconsistent.
7. Longer sonication periods may improve results for porous materials.
8. The operations are can be conducted at near ambient conditions in a glove box.
9. Exposing contaminated organic samples to a high radiation dose (>110 Mrad) in a nuclear reactor core prior to decontamination, resulted in 86% particle removal. Since this radiation dose rate and total dose are significantly higher than likely exposures to hydrogenous matter in surrogate waste, the probability of achieving high decontamination factors with actual wastes is considered high.
10. DPA provides a rapid, inexpensive index of decontamination effectiveness.

1.0 BACKGROUND

1.1 LEGACY WASTE

The United States Department of Energy's Savannah River Site (SRS), near Aiken, South Carolina, stores Pu-238 contaminated transuranic waste. Portions of this waste cannot be shipped to the Waste Isolation Pilot Plant (WIPP) in TRUPACT-II containers due to excessive generation of hydrogen gas as a result of radiolysis of organic constituents in the waste matrix. Treatment of this waste to destroy the organics (eliminating the source of hydrogen) would allow the waste to meet the TRUPACT-II hydrogen concentration limit. The alternative is to decontaminate the organic matrix so that the Pu-238 can be concentrated in a WIPP-compliant package and the treated organic material has a hydrogen gas generation rate below the regulatory limit. The inorganic materials in the waste can also be processed through the cleaning system to facilitate contamination control during repacking operations.

The TRU waste drum contents consist of heterogeneous job control waste primarily contaminated with Pu-238 oxide. The drum is lined with a polyvinyl chloride liner. Waste is packaged in the drum in four to six layers of polyvinyl chloride bags. Drums may contain numerous waste packages. Waste contents include polyvinyl chloride, cellulose, metals, and other job control waste, and may include cans and bottles (both metal and plastic) containing hazardous liquids. The details of a typical waste drum are listed in Tables 1 and 2 of section J-1 of the Alternatives to Incineration Solicitation, PRDA No. DE-RA26-01NT40981 (1) found at www.NETL.doe.gov/business/solicit/.

The Pu-238 contamination is expected to consist of 0.2 μm particles. Submicron-sized particles generally adhere tenaciously and non-specifically to other solid surfaces, and cannot be removed by simple mechanical means. There is a large body of physical evidence that suggests that their adhesion is due to secondary valence forces between the particles and the substrate(2). The removal of unwanted fine particles from solid substrates is a problem that has been recognized for several decades (5, 6), and is of continuing importance in a number of technological areas (7, 8, 9), including nuclear decontamination.

1.2 RELEVANT EXPERIENCE

In 1987, Dr. Robert Kaiser of ESI (8) demonstrated that contacting particle-contaminated surfaces with a dilute solution of a high molecular weight fluorinated surfactant in an inert perfluorocarbon (PFC) liquid under high shear conditions resulted in very effective removal of micron or sub-micron sized particles from solid surfaces. Under the auspices of a Small Business Innovation Research Program (SBIR) (AF91-186)

sponsored by the Ballistic Missile Organization of the U.S. Air Force, ESI was able to demonstrate enhanced removal of small particles from solid surfaces of inertial guidance instruments (9). This technology is the basis of the Sonatol® process.

The perfluorinated liquid solutions used in the Sonatol® process have zero ozone depletion potential, are nontoxic, nonflammable, and are generally recognized as non-hazardous materials (See Table 1, next page). They are generally innocuous to all materials including paper, plastics, metals, metal salts and oxides, wood and cloth. Not only are they compatible with the processed items, but also they do not react irreversibly with the particles being removed. These liquids have a high specific gravity (s.g. 1.7), low viscosity (less than 1 cP), and a low surface tension (less than 15 dynes/cm), which facilitates wetting and penetration into holes and crevices. PFCs, which boil in the range of 50° to 80°C, are used to facilitate their recycling and recovery by distillation. The heat of distillation (20 cal/gram) is less than 3% of that of water, which greatly facilitates the distillation process. Under the Significant New Alternatives Program (SNAP) of the U.S. Environmental Protection Agency (EPA), these materials have been found to be an environmentally acceptable means of cleaning highly complex geometric configurations that must meet stringent particulate cleanliness standards.

Under the auspices of a U.S. Nuclear Regulatory Commission (NRC) SBIR program (1, 1), ESI was able to initially develop, and then demonstrate at a nuclear electric power generating station, the application of the Sonatol® process as a means of decontaminating sensitive equipment, such as personal computers, monitors, voltmeters, and precision micrometers. During the development program, decontamination factors (DFs) in excess of 1,000 were obtained.

In 1996, BSI received a contract to decontaminate the large coalescing filters (24" x 24" x 6") that were used to clean-up effluent gases from the Consolidated Incineration Facility at the Savannah River Site. These filters contain multiple layers of a woven Hastalloy and fiberglass filtration medium. For this project, BSI and ESI designed, constructed, and operated a Sonatol® filter decontamination system(12). The system was installed in a 44 ft. long mobile trailer that was installed at SRS for this project (see Figure 4). Over the period 1997 (when the Sonatol® system went into operation) to 2000 (when the incinerator was shut down), 344 filters were processed in the Sonatol® System. Of these, over 95% were returned to service. This is the first known successful demonstration of cleaning this type of filter for reuse.

In 1997, the Sonatol® process was adapted for the Battelle Columbus Laboratories Decommissioning Project to decontaminate 3,000 ft³ of TRU level debris down to a low-level waste (LLW) classification(13). The TRU removed from the debris was transferred

to collection filters, which will be classified as TRU waste and eventually shipped to WIPP. To facilitate characterization under WIPP's waste acceptance criteria (WAC), these filters are of specially designed 100% stainless steel construction. They are built to fit within a standard 55-gallon drum with space for four inches of shielding. The filters include several features that enhance mass loading in order to minimize the number of drums of TRU waste that will result. These include a pleated filter construction to maximize filter surface area, welded stainless steel wire construction to resist compaction of the filter medium, spring loaded check valves on the outlet and inlet allow the canister to be classified as an open container per the WIPP.

BSI also developed a decontamination system specifically for TRU level mop heads. The mop heads are cleaned in a closed-cycle agitation system with collection filters and a still. This system has successfully reclaimed TRU level mop heads for reuse during the BCLDP and avoided a problem with non-WIPP compliant waste due to the water and hydrogen content in the mop heads. Mop heads are one of the waste streams of interest in this project. We are not aware of any other process that has been successful in decontaminating TRU level mop heads for reuse and later disposal as LLW.

2.0 TECHNICAL APPROACH

BSI proposes to use the Sonatol® process to reduce the Pu-238 content of the legacy waste, and thus reduce the level of hydrogen generation sufficiently to allow transportation in TRU-PACTII containers. The Sonatol® process uses fluorinated surfactant solutions to detach micron and submicron sized particles that spontaneously adhere to substrates and transfer these particles to stable inorganic filters. Filtration removes the particles from suspension and allows the process liquid to be recycled.

Fluorinated surfactant solutions are effective media for the removal and transfer of Pu-238 particles from legacy wastes because:

1. Sonatol® solutions are inert liquids that do not chemically react with or dissolve materials in legacy wastes.
2. Sonatol® solutions are inherently safe to use. The solvents are non-toxic, non-flammable and have zero ozone depletion potential and have been accepted by the US EPA under the Significant Alternatives Program (SNAP) as suitable process liquids for demanding particle removal processes.
3. The Sonatol® process operates at low temperatures and pressures in a closed loop operation. There is no liquid discharge or gaseous exhaust.
4. The process can be scaled up to a 55-gallon batch mode in which the waste and the Sonatol® processing equipment are totally enclosed in a sealed chamber.

2.1 DECONTAMINATION SYSTEM

Based on the experience gained in the programs described in Section 1.2 above, BSI believes that the organic TRU waste in the drums at SRS will be successfully decontaminated with the Sonatol® perfluorinated surfactant solution in a cleaning chamber that allows for mechanical agitation, as well as spraying or ultrasonic agitation. The geometry and composition of legacy waste varies widely such that no single method of introducing shear forces may be well suited to the entire range of materials that will be encountered. A simple analogy can be made to domestic cleaning equipment. Most homes have a clothes washer and a dishwasher. Washing dishes in a clothes washer would be disastrous and washing clothes in a dishwasher would be futile. However, each is well adapted for its intended use. In addition, no one would seriously consider cleaning jewelry in either of these machines. An ultrasonic bath is preferred. However, for this proposal, we have designed a Sonatol system that can operate in these modes plus provide contamination control. A single machine will be able to agitate, sonicate, and/or spray clean the waste.

2.1.1 Cleaning System Design

BSI built a specially designed cleaning system with three cleaning options for this unique waste stream. This would be equivalent to having a single machine in your home that would clean dishes, clothes, or jewelry. This is readily achievable due to our experience with all three cleaning modes and our knowledge of digital control systems. The cleaning system has the ability to spray a detergent solution or a rinse solution onto contaminated items, while circulating the cleaning fluid through 0.2 μm ceramic filters. The chamber is also equipped to mechanically or ultrasonically agitate the material in the chamber in a detergent solution or a rinse solution and then circulating the fluid through 0.2 μm ceramic filters.

A programmable logic controller (PLC) will provide a menu of cleaning options for the operator. The PLC is programmed to implement a different series of cleaning steps under each option. The exact sequence and timing of steps is coded into the PLC so that the operator need only select a menu option and the cleaning system will implement a cleaning regimen. The PLC can also be used to intervene in the cleaning process and control the system manually.

2.1.2 Ceramic Filters

Ceramic filters offer high temperature stability, strength, and compactness. They are available in the form of cylinders or large diameter honeycomb monoliths that contain a large number of 2 mm square parallel passageways extending from one face to the other, as shown in Figure 5, Schematic Representation of Ceramic Membrane Monolith. This results in a very compact filter arrangement. Note that in dead-end re-circulating filtration, which is used here, there is no retentate flow.

The Sonatol® liquid filtering system consists of membrane-coated silicon carbide filter modules. These all-ceramic, solvent-inert filters are currently being evaluated as “washable HEPA [high efficiency particulate air] filters” under DOE Contract DE AC26 99FT40569 and are being evaluated at Savannah River Technology Center under the direction of Mr. Duane Adamson.

BSI expects that most of the Pu-238 oxide particles will be collected on the ceramic filters. These filters will be dry when they are removed from the process, and will be treated as TRU solid waste.

2.1.3 Cleaning Operations

As indicated in Figure 6, the material in the cleaning chamber is sequentially exposed to fluorinated surfactant solution and fluorinated rinse. The material to be processed is

loaded into a basket and placed in an empty cleaning chamber. As outlined in Step 1 of Figure 6, the chamber is first filled through spray jets from the solvent storage tank with a dilute solution (at least 0.2 wt-%) of fluorinated surfactant and carrier liquid. The material is agitated mechanically and ultrasonically and the liquid is re-circulated from the bottom of the cleaning chamber through a bank of filters and back to the spray jets in the chamber (Figure 6, step 2).

The agitation suspends small particles present on the surfaces of the items being treated. Suspended particles are removed from the liquid stream by the filter bank. After being agitated for a few minutes, the remaining suspended particles are removed by pumping the liquid in the cleaning chamber to the holding tank through the filters (Figure 6, Step 3).

The filter bank consists of a stainless steel screen, a coarse stainless steel or ceramic filter, and a 0.2 μm ceramic filter. The liquid first passes through a screen to remove debris. Then a 17-micron filter removes suspended textile and paper fibers without affecting much smaller Pu-238 oxide particles. The sub-micron Pu-238 particles are removed in the 0.2 μm ceramic filter.

The next stage of the cleaning process is to reintroduce fluorinated wash solution and repeat applying mechanical agitation and ultrasonics by repeating steps 1 to 3, as required. In each stage, the sequence of steps 1, 2 and 3 is repeated. The amount of particles removed typically decreases with each successive stage as the process reduces the number of remaining particles. The appropriate number of repetitions can be determined by experimentation or by in-line measurements of the turbidity in the fluid exiting the waste-cleaning tank.

The final step (Figure 6, Step 4) uses a carbon filter to polish the cleaning solution and create a rinse solution. The rinse solution is sprayed into the chamber to dislodge particles that may be attached to surfactant residues. This rinse liquid dissolves residual surfactant on the waste and transports attached Pu-238 particles to the filters.

2.1.4 Turbidity Measurement

The use of in-line turbidity meters combined with PLC controls allows optimization of the cleaning cycles by controlling the duration of each step according to the removal rate of particles from the waste. Using in-line monitors also provides a method to estimate the decontamination rate while the waste is being cleaned. The data can be used to terminate the cleaning early or to extend the cleaning process until a minimum value of the turbidity has been obtained. All of these options are readily programmed into the PLC. When the effectiveness of the wash solution has declined, the wash

solution can be drained from the cleaning chamber and the chamber refilled with rinse liquid.

The system incorporates an in-line turbidity meter as a means of controlling the decontamination process. This instrument effectively monitors the rate and extent of cleaning in the flow-through cleaning chamber. The turbidity in the effluent stream is evidence that the process is actively removing particles from the processed waste (i.e. it is not yet clean).

The required duration of the cleaning sequence depends on the initial concentration of particles, the desired final concentration, and the removal rate. Using the turbidity meter will allow the system to adjust the cleaning duration according to the contamination levels of the items in the chamber. Eventually, the process of comparing real time turbidity with post cleaning surveys will allow users to adjust the PLC action levels to be adjusted efficiently, based on the turbidity meter signal.

2.2 COLD SYSTEM DEMONSTRATION

The prototype decontamination system was shipped from the fabricator to ESI in Woburn, MA where system characterization and optimization trials were performed.

2.2.1. Surrogate Waste and Plutonium Oxide Simulant

Representative components of a surrogate waste matrix of the SRS waste stream were assembled, characterized, and contaminated with two plutonium oxide simulants. Waste stream components included inorganic and organic materials, rigid and deformable materials, and components of different levels of surface complexity. Examples are machined parts, such as fasteners, electrical and electronic components, instruments, sealed containers with various liquids, textile products, such as protective garments and mop heads, rubber goods, such as gloves and aprons, plastic sheeting, etc. Emphasis was placed on the processing of organic materials most likely to release hydrogen gas by photolysis.

The surrogates were contaminated with two non-radioactive submicron sized powders that simulate Pu-238 oxide, namely cerium oxide and a phosphor. The phosphor fluoresces intensively at 546 nm (green light) when illuminated with a high intensity ultraviolet (365 nm) light. To quantify these UV observations, representative samples of the contaminated material were photographed with a high-resolution digital camera under UV light, and the photographs were digitally analyzed on a personal computer, using image analysis software. The procedure counts the number and intensity of green pixels in the image. The degree of decontamination due to a procedure is obtained by

comparing the green pixel counts of photographs of an object taken before and after this procedure.

The effect of different methods of solid/liquid contacting (spray, immersion with and without ultrasonic agitation) on the rate and extent of contaminant removal from different matrix components was examined. Process kinetics were monitored by measuring the turbidity of the liquid effluent from the cleaning chamber. The post-cleaning level of contamination of the processed material was first estimated from DPA measurements and confirmed by NAA of selected parts at the MIT Nuclear Reactor Laboratory. NAA involves the identification of trace quantities of elements by first activating the stable isotope and then counting the energies of the resulting gamma rays.

2.2.2 Process Requirements/Constraints

For this system to work effectively the legacy material must be contacted by the working fluid and the contamination itself must be in particulate form on the surface of the waste. Large plastic bags would have to be cut into segments or shredded. The process is not recommended for the decontamination of soils, sweepings, resins, and degenerated material that could disintegrate. (The ceramic filters will be protected from incidental debris by a removable screen and a pre-filter with high dirt loading capacity.) Radiation levels on the collection filter could be monitored to ensure that the dose rates on the exterior of the drum packages do not exceed dose rate, gram quantity, and wattage limits.

Also, this system was designed for a limited number of production runs. The D-1 filter, which generates the rinse solution, has a nominal capacity sufficient to process 6-12 baskets. When the D-1 filter is loaded to capacity with surfactant, the system will continue to decontaminate materials, but a slight surfactant residue will be left on the items and the DF may be lowered somewhat. This constraint could be easily modified if the user desired to operate in a daily production mode. The filter lifetime could be extended to hundreds of baskets.

2.3 STATEMENT OF WORK

The principal deliverables of the Phase I Cold Demonstration are discussed below.

2.3.1 Design and Build

The first objective was to design and build a prototype Sonatol decontamination system that can process one batch of heterogeneous, Pu-238 contaminated TRU waste. The cleaning chamber was based on the existing design of a rotary basket parts washer

manufactured by ADF Systems, Ltd., Humboldt, IA. The cleaning chamber provides a combination of immersion, agitation, and spray cleaning required to remove contaminants from blind holes, internal holes, cavities and threads. BSI modified the existing design by adding ultrasonic agitation and additional vapor and contamination control features.

The design included but was not be limited to:

1. Development of process flow sheets
2. Identification, description and sizing of the major pieces of process equipment
3. Development of the bill of materials
4. Development of electrical schematic diagrams
5. Development of process logic control software
6. Documentation installation and operating procedures

Burn-in and optimization of the prototype decontamination system was performed at ESI's facilities in Woburn, MA. Quality control testing ensured that all system components operated properly, and that the system is leak free and vapor tight.

2.3.2 Demonstrate 99% Removal of Pu-238

The second objective was to demonstrate 99% removal (DF of 100) of a cerium oxide containing simulant for Pu-238 particles from the organic portion of a non-radioactive surrogate waste by washing with the perfluorinated process liquids, and the subsequent collection of the surrogate from the perfluorinated process liquids by ceramic filters. A related task is the development of optimum process parameters (cycle steps, duration, process temperatures, number of filtration passes, etc.) for each type or combination of surrogate waste tested.

The effect of different methods of solid/liquid contacting (spray, immersion with and without ultrasonic agitation) on the rate and extent of contaminant removal from different matrix components was examined. The post-cleaning level of contamination of the processed material was estimated by DPA of fluorescence emitted from Pu-238 simulants and by NAA of representative samples at the MIT Nuclear Reactor Laboratory.

Doping surrogate waste samples with simulated Pu-238 contaminant tested the second objective. The calculated mass-weighted average NAA DF was 162 for the SRS representative surrogate waste matrix, which was 82% organic. The weighted DFs for two bounding cases, which were both 70% organic, were 137 and 156. The weighted DFs for the organic-only portions of the surrogate waste matrices were 150 for the

representative case and 65 and 166 for the bounding cases. Measured DFs for individual organic materials ranged from 3.5 to 308.

2.3.3 Demonstrate 99.9% Removal of Submicron Cerium Oxide

The design includes a stainless steel pre-filter and a 0.2 μm , ceramic Pu-238 filter. Most of the Pu-238 is expected to collect in the ceramic filter. Nevertheless, both the ceramic filter and the stainless steel prefilter are 100% inorganic to preclude the possibility of generating hydrogen gas from the structure of the filters. Testing of the ceramic filter indicated that the retention fraction of the submicron cerium oxide powder was at least 99.9% for a single pass. The removal of sub-micron particles by the ceramic filter was established by the filtration of sub-micron cerium oxide suspensions from fluorinated surfactant solutions. The effective removal of particles was determined by measuring the particle concentration of the liquid effluent leaving the filtration module with a turbidity meter and comparing this value to the turbidity reading of clean Sonatol solution.

2.3.4 Fugitive Emissions

The system operates in a closed loop with no emission points. All process fluids are contained in a glovebox, which captures any leaks or fugitive emissions. The facility's ventilation and effluent control systems would further prevent fugitive emissions from the gloveboxes.

2.3.5 Designed for Hot Testing

The cold test results support further hot testing. The system design was verified to be suitable to operate in actual SRS gloveboxes at the Savannah River Technology Center. The mockup glovebox used in cold testing is a dimensional replica of one such SRS glovebox, based on as built measurements and photographs. SRS personnel have observed the operation of the prototype Sonatol system and their suggested improvements have been incorporated into the system. The modifications included relocating electronic equipment outside the glovebox boundary. As a result, a bulkhead fitting was designed and fabricated to pass control wiring and the ultrasonic power cable through the glovebox wall. An additional modification consisted of adding a heat exchanger to cool the fluid exiting the cleaning chamber. Cooling water penetrations provide supply and return water. Taken together, these modifications result in more free volume in the glovebox and a reduction in heat buildup.

The system was designed such that, when the system is in the glovebox, any single item of equipment can be detached and removed/replaced for maintenance, if required. This includes the cleaning chamber itself. Qualitative surveys of the glovebox interior

and process tubing downstream of the ceramic filter show no residues of contamination simulants at the end of the cold tests.

The signal obtained from the turbidity meter correlated well with the various steps of the decontamination process. Turbidity measured in the fluid leaving the decontamination chamber increased abruptly as the ultrasonic power was turned on and returned to background during the rinse step. The PLC is programmed to extend the ultrasonic cleaning step if turbidity readings remain elevated. This will support hot testing because the system can automatically accommodate wastes that require longer cleaning times.

2.3.6 Suggested Modifications to Prototype Hot Operations

Experience gained from cold testing has already been incorporated into the prototype system to support hot testing. These changes include relocation of electronics, elimination of physical interferences, and reduction of heat generation in the glovebox. In addition, we learned that the wall of the basket attenuates the ultrasonic waves. Under certain conditions, the basket interfered with the transfer of ultrasonic energy to the organic component of the waste. The magnitude of this interference depends on the mass and the mesh size of the basket. As a result, the basket was redesigned in order to achieve the higher DFs required. The revised basket design was verified in the round of integrated cold testing; and six redesigned baskets are being provided for hot testing.

The equipment arrangement in the glovebox and the plumbing runs were revised to facilitate installation and operations. As a result of these changes, the installation procedure has been revised and an installation videotape was prepared to facilitate installation at SRS. The mockup glovebox is also being shipped to SRS to provide a training aid for SRS personnel. The prototype Sonatol system can be installed and operated in the mockup at SRS.

The DPA test can also be used to support hot testing. When the prototype is assembled at SRS, the fluorescent test can be used to validate operator training and/or the operability of the system before actual hot tests are conducted.

2.3.7 Phase II Enhancements (Hot and Full-Scale)

During Phase II testing, longer sonication times and measures to reduce clumping of organics is recommended. Use of DPA for quality control and training is also recommended.

2.3.8 Full-Scale Enhancements

During full-scale operations, adopting a mechanical design that does not require use of baskets for organic materials may enhance the decontamination factor for organic material by delivering more of the ultrasonic energy to the material that is being cleaned. By eliminating attenuation of ultrasonic energy by the wall of the basket, the productivity and/or DF could potentially be improved. By eliminating a handling step, contamination control would also be improved.

The collection filter for the full sized system can be designed to fit in a 55-gallon drum and allow four inches of internal shielding on all sides. Adding provisions for shielding in the drum will reduce the potential for generating remote handled TRU waste. The canister can also be designed to be self-venting in order to satisfy the WIPP criterion, which prohibits sealed containers larger than four liters.

It may also be possible to reduce the surfactant concentration from 0.4 wt-% to 0.2 wt-% without a deleterious effect on the decontamination capability of the system. This reduction would result in an extension in the useful life of the activated carbon fiber adsorbent.

2.3.9 Disposition Paths for Secondary Waste (cold and hot tests)

None of the materials used in the cold tests are toxic or hazardous. Therefore all secondary solid wastes generated by cold testing have been disposed as municipal waste.

At the completion of hot testing, the prototype system components that are operated in the glovebox will be disposed as regulated radioactive waste. The cleaning fluid evaporates rapidly so that the contaminated components can be dried in the glovebox by evaporation, leaving only solid waste. There are no toxic or hazardous materials in the construction of the decontamination system itself. If the total quantity of Pu-238 that is admitted into the hot testing is comfortably less than 430 microcuries (100 nCi/g times 4,300 g total weight of ceramic filters), then no component will be classified as TRU level waste. Therefore the entire waste stream should be classified as low-level waste, which can be disposed in a regulated disposal site at SRS.

3.0 SYSTEM DESCRIPTION

3.1 SYSTEM OVERVIEW

The Sonatol® decontamination system that was designed and built under the present program is an automated, small-scale, closed-loop cleaning system. Photographs of the individual components are provided in the installation manual (Appendix G). The heart of the system is a stainless steel cleaning chamber that provides for spraying, rotation, or ultrasonic agitation of the waste to achieve decontamination.

3.2 SYSTEM CONSTRAINTS

The system was designed for operation within the specific glove box identified for hot testing at SRS. The components are individually placed in the glove box and then assembled by quick connect fittings.

3.3 SYSTEM COMPONENTS

3.3.1 Cleaning Chamber and Cleaning Baskets

The cleaning chamber is 18" (45.7 cm) long x 12" (30.5 cm) wide x 10.25" (26.7 cm) high. It contains two spray manifolds. Each contains 4 flat fan spray nozzles. The manifold inside the front (or operator side) of the chamber is used to spray recirculation liquid at high pressure. The rear manifold is used to fill the chamber with either wash solution or rinse liquid.

Twelve (12) Blackstone-Ney ultrasonic transducers that are welded to the bottom of the chamber provide ultrasonic agitation. To protect these transducers, the cleaning chamber is mounted on a 3.5" (8.9 cm) high base. While removable, the base is attached to the chamber with Velcro tape because it is easier to install the combined assembly than to place the chamber on the base inside the glove box. The weight of the cleaning chamber and base is 77.5 lbs. (35.2 kg).

The cleaning chamber also contains two parallel cylindrical shafts fitted with polyvinyl chloride covered 1.5" (3.8 cm) diameter drive rollers that are used to rotate a parts basket that fits inside the chamber at 4.5 rpm. The drive rollers are powered by a chain drive that is connected to an external and removable AC gear motor. The dimensions of the 1/15 HP motor assembly (including pulleys and belt guard) are 9" (22.9 cm) long x 7" (17.8 cm) high x 6" (15.2 cm) deep. The weight of the gear motor and protective belt guard is 18 pounds (8.2 kg).

Five cleaning baskets are provided with the system. As shown below in Figure 7, these cylindrical baskets are 4" (10.2 cm) in diameter by 10" (25.4 cm) long. The body of the basket consists of 4 mesh welded 304 stainless steel screening that is supported by two, 4" (10.2 cm) diameter by 0.75" (1.8 cm) long 304 stainless steel end rings. The screening material has 75% open area. The ends of the basket were made of 0.036" thick perforated steel sides with 3/16" holes (63% open space). The sharp edges of the end plates are edged with a circular PVC grommet. The ends are attached to the basket by 8-32 x 3/8" thumbscrews with a 5/8" knurled head. A threaded rod and washer are attached to each end plate to center the basket within the cleaning chamber. Each basket weighs 1 pound (450 grams).

Figure 7 Basket Provided with System

3.3.2 Holding Tank (T-1)

In-process liquid storage is provided by a stainless steel holding tank. This tank is 15.5" (45 cm) high x 13" (39 cm) wide x 12" (30.4 cm) deep, and has a liquid capacity of 9 gallons (34 liters). Its empty weight is 25.5 lbs. (11.6 kg).

3.3.3 Adsorption Column (D-1)

The adsorption column provides surfactant free solution for the final rinse. As described in more detail in Appendices C and D, the adsorption column consists of activated carbon felt that is wrapped over a filtration mandrel, and is housed in an aluminum alloy assembly. The assembly consists of a 10" (25.4 cm) length of 8" schedule 40 6061-aluminum alloy capped by two 10.5" (26.7) diameter, 0.500" (1.27 cm) thick 6061-aluminum alloy end-plates. The assembly is held together by six 3/8"-16 stainless steel threaded rods.

A ¼" NPT stainless cross is attached to the top of the housing. A vent valve (MV-3), to release air when the column is filled with liquid, a pressure gauge (PG-1), and a over pressure relief valve (PRV-1) set at 25 psig are attached to this cross. The outlet of the pressure relief valve is attached to the vent line going to the top of T-1. The dry weight of the ACF housing and filter is 26.5 lbs. (12 Kg).

3.3.4 Filters

Filters F-1, F-2, and F-3 are in contact with the process solution that leaves the cleaning chamber, and thus exposed to radioactive material. However, filter F-4 is in contact only with the final rinse.

- a) *Filter F-1*: This filter is a removable piece of 10-mesh screening that is placed over the bottom of the cleaning chamber. It is used to prevent objects large enough to plug the heat exchanger or interfere with pump J-1 from leaving the cleaning chamber.
- b) *Primary Filter (F-2)*: The primary filter is an all stainless steel unit that houses a 10" (24.4 cm) long x 2.5" (6.4 cm) 19 µm stainless steel filter module inside a stainless steel shell. The inlet and outlet connections are ¾" Swagelok™ compression fittings. The dry weight of F-2 and associated bracket is 16.5 pounds (7.5 kg).
- c) *Secondary Filter Assembly (F-3A & F-3B)*: The secondary filter assembly consists of two parallel 0.2 µm Al₂O₃ over SiC filter modules. Each 1" (2.54 cm) diameter by 12" (30.4 cm) long module is housed in a 1-1/2" (3.8 cm) diameter 316 stainless steel housing. The filtration area of each module is 1.2 ft² (0.1 m²). Ceramem, Inc. of Waltham makes both the filter modules and housings, MA. The purpose of these filters is to remove sub-micron particles suspended in the solution that leaves the cleaning chamber before this solution is returned to storage. Two filters were placed in parallel to reduce the pressure drop to a level that matched the output pressure of the circulation pump J-1. The dry weight of each filter and module is approximately 5.5 lbs (2.5 kg).
- d) *Rinse Filter F-4*: The output of D-1 is filtered by a 0.45 µm Durapore™ membrane filter capsule with a filtration area of 1,000 cm². Millipore Corp. of Bedford, MA makes it. Because the liquid that comes into contact with this filter was previously filtered through F-3 and through the activated carbon column, this filter is unlikely to be exposed to any radioactive material.

3.3.5 Ultrasonic Generator

A MultiSONIK Dualsweep generator manufactured by Blackstone-Ney Ultrasonics, Jamestown, NY, powers the ultrasonic transducers in the cleaning chamber. In order to minimize heat generation in the glove box, this unit is positioned outside the glove box. Its specifications are as follows:

	SPECIFICATIONS
Input voltage	208 VAC
Input power	1000 watts
Output power	720 watts
Output Frequencies	40, 72, 104 and 170 kHz
Weight	20 lb. (9.1 kg)
Dimensions	13" (33.0 cm) W x 16.5" (41.9 cm) L x 6" (15.2 cm) H

3.3.6 Turbidity Meter (TM-1)

A turbidity meter is placed on the output of Filter F-2 to monitor the circulating liquid for the presence of fine particles. The particles could be the Pu-238 simulants that were added to the surrogate waste as well as to fine particles of dust or dirt that were naturally present on the surfaces of the surrogate waste samples.

	SPECIFICATIONS
Input voltage	110 VAC
Measurement range	0.01 – 1000.0 NTU
Accuracy	±2% - ±5%
Weight (w/ bracket)	10.5 lb. (4.8 kg)
Dimensions	9" (22.9 cm) W x 9" (22.9 cm) D x 15" (38.1 cm) H

3.3.7 Heat Exchanger (C-1)

The heat exchanger is a stainless steel brazed plate heat exchanger, 8" (20.4 cm) L x 3" (7.6 cm) W x 1-11/16" (4.3 cm) H is used to cool the Sonatol® solution after the ultrasonic cycle. This allows the system to remain in thermal balance and also prevents pump J-1 from cavitating. The cooling liquid is chilled water. The heat load is about 12.5 BTU per minute of sonication. In protocol II A, the liquid is sonicated for 27 minutes per cycle (1 hour). The heat load per cycle is therefore 340 BTUs.

3.3.8 Pumps

- a) *Main Circulation Pump (J-1)*: Pump J-1 is a magnetically coupled pump. It is used to drain the cleaning chamber and cool the liquid through the heat exchanger

during recirculation. In order to prevent over pressurization of the pump, the impeller was trimmed $\frac{1}{4}$ ". The pump output curves for water and Sonatol A 70 are presented in Figure 8a. The weight of J-1 is 18 pounds.

- b) *Feed Pump (J-2)*: Pump J-2 is a magnetically coupled pump. It is used to fill the cleaning chamber with solution and for the final rinse. Because of the lower pressures associated with J-2 it was not necessary to trim the impeller. Figure 8b shows the J-2 performance curve in water and PF-5070. The weight of J-2 is 16 pounds.

Figure 8a Performance Curve for Pump J-2

Figure 8b Performance Curve for Pump J-1

3.3.9 Thermistor

A thermistor monitors the temperature of the liquid in the cleaning chamber. The temperature display is on the front of the electrical control box. The instrument range is from 0°F to 250°F.

3.3.10 Valves

a. Actuated valves:

There are four solenoid valves in the system that are controlled by the PLC and regulate the movement of cleaning fluid, and a fifth solenoid valve on the water line of the chiller. The characteristics of these valves are as follows:

Application	Process	Water Line
Type	2 way-NC	2 way NC
Body	Brass	Stainless Steel
Diaphragm	Buna N	Teflon
Size	½" NPT	½" NPT
Voltage	110 V AC 1 Φ	110 V AC 1 Φ

b. Manual Valves:

There are four manual valves in the system:

Valve No.	Type	Purpose
MV-1	Ball	P-2 isolation valve
MV-2	Vent	Purge D-1
CV-1	Needle	Limit flow to D-1
CV-2	Needle	Control flow from TM-1

c. Pressure relief /check valves:

PRV-1 is a 25-psig, pressure relief valve. Located at the top of the D-1, PRV-1 is a safety valve that will release fluid into the vent line if D-1 is over-pressured.

PCV-1 is a 1-psig-check valve. Located below CON-9, PCV-1 prevents liquid from siphoning from D-1 into the cleaning chamber when the system is idle.

3.3.11 Pressure Gauges and Sensors

a. Pressure Sensors

Five pressure sensors are used in the system to monitor pressures and liquid levels. The output pressures are relayed to the PLC and can be accessed through the PanelView operator interface.

Pressure Sensor Number	Voltage	Pressure Range	Purpose
P-1	24 in/ 5 out	0-5 psi	Monitor liquid level in cleaning chamber
P-2	24 in/ 5 out	0-5 psi	Monitor T-1 liquid level
P-3	24 in/ 5 out	0-60 psi	Monitor F-2 upstream pressure
P-4	24 in/ 5 out	0-60 psi	Monitor J-2 outlet pressure
P-5	24 in/ 5 out	0-60 psi	Monitor F-2 downstream pressure

b. Pressure Gauges

There are four pressure gauges used in the system to assist the operator at monitoring the system status. The operator can determine the liquid level in T-1 directly from PG-2 because an oz/in² is equivalent to the pressure exerted by 1 inch of Sonatol® solution.

Pressure Gauge Number	Pressure Range	Purpose
PG-1	0-15 psig	Monitor the pressure in D-1
PG-2	0-10 oz/in ²	Monitor the liquid level in T-1
PG-3	0-60 psig	Monitor upstream pressure of F-2
PG-4	0-60 psig	Monitor downstream pressure of F-2

3.3.12 Quick Disconnect Fittings

Thirteen quick disconnect fittings are used in the system, enabling it to be easily installed within the glove box. All quick disconnects are 316 Stainless steel fittings manufactured by Swagelok™. The characteristics of these fittings are given in Table 3.3.12-1

Table 3.3.12-1 Quick Disconnect Fittings used in System

Number	Size	Model	Double-Ended Shut Off	Spillage (ml)
Con 1	3/8"	SS-QF4	No	-
Con 2	1/2"	SS-QC8	Yes	3.0
Con 3	1/2"	SS-QC8	Yes	3.0
Con 4	1/2"	SS-QC8	Yes	3.0
Con 5	1/2"	SS-QC8	Yes	3.0
Con 6	1/2"	SS-QC8	Yes	3.0
Con 7	3/8"	SS-QC6	Yes	1.0
Con 8	3/8"	SS-QC6	Yes	1.0
Con 9	3/8"	SS-QC6	Yes	1.0
Con 10	1/4"	SS-QC4	Yes	0.3
Con 11	1/4"	SS-QC4	Yes	0.3
Con 12	3/8"	SS-QC6	Yes	1.0
Con 13	3/8"	SS-QF4	No	-

3.3.13 Electrical Components

All of the electrical components including the PLC are provided in a NEMA enclosure, as shown in Figure 9. The operator interface is housed in a separate enclosure.

Figure 9 – NEMA enclosure

Table 3.3.13-1 presents the power and signal connections that are made through bulkhead fittings. For additional information on the bulkhead fittings see Appendix I

3.4 UTILITIES

3.4.1 Electrical

The system requires a single-phase, 208 VAC, 20 Amp power source for operation. The ultrasonic generator requires 208 VAC power. The remaining electrical components require single phase 120 VAC.

3.4.2 Cooling Water

Cooling water is needed to remove heat build-up in the process liquid due to sonication, approximately 340 BTUs/cycle. Water inlet temperature should be 15°C (60°F) or less. One inlet and one outlet line is required. Cooling water is only needed

for 9 minutes during the normal operation cycle and is controlled by a Solenoid valve (Valve AV-5) that is open only when pump J-1 is on.

Table 3.3.13-1 Power and Signal Cables Connected through Bulkhead Fittings

Cable #	Unit	Purpose	Voltage (volts)	Wire Size (gauge)	# of Conductors	Diameter (in)	Termination Description
1	US Frequency Generator	Power	208	14	3	0.37	250V/15A twist lock on end
2	US Frequency Generator	Signal	N/A	20	8	0.25	Cable from factory
3	Turbidity Meter	Power	110	14	3	0.37	125V/15A twist lock on end
4	Turbidity Meter	Signal	(4-20mA)	18	2 (shielded)	0.25	Threaded Electrical Quick Connect on end
5	Pump J-1	Power	110	14	3	0.37	125V/15A twist lock on end
6	Pump J-2	Power	110	14	3	0.37	125V/15A twist lock on end
7	Gear Motor	Power	110	14	3	0.37	125V/15A twist lock on end
8	Pressure Transducer P-1	Signal	24in 5 out	22	4 (shielded)	0.17	Threaded Electrical Quick Connect on end
9	Pressure Transducer P-2	Signal	24in 5 out	22	4 (shielded)	0.17	Threaded Electrical Quick Connect on end
10	Pressure Transducer P-3	Signal	24in 5 out	22	4 (shielded)	0.17	Threaded Electrical Quick Connect on end
11	Pressure Transducer P-4	Signal	24in 5 out	25	4 (shielded)	0.17	Threaded Electrical Quick Connect on end
12	Pressure Transducer P-5	Signal	24in 5 out	26	4 (shielded)	0.17	Threaded Electrical Quick Connect on end
13	Solenoid Valves AV-1	Power	110	18	2 (shielded)	0.2	Connection Supplied with Valve
14	Solenoid Valves AV-2	Power	110	18	2 (shielded)	0.2	Connection Supplied with Valve
15	Solenoid Valves AV-3	Power	110	18	2 (shielded)	0.2	Connection Supplied with Valve
16	Solenoid Valves AV-4	Power	110	18	2 (shielded)	0.2	Connection Supplied with Valve
17	Solenoid Valves AV-5	Power	110	18	2 (shielded)	0.2	Connection Supplied with Valve
18	Thermistor	Signal	6 DC	26	2	0.14	Hardwired to Thermistor

3.5 AUTOMATED CONTROL SYSTEM

3.5.1 Overview

Operation of the bench scale Pu-238 decontamination system is operated by an Allen-Bradley Programmable Logic Controller (PLC), which allows the system to operate in either a preprogrammed automated mode or in a manual mode. The operator interacts with the system through an Allen-Bradley PanelView 600 touch-screen interface. The

PanelView presents the operator with a series of touch screens that allow him/her to perform all required functions. The PanelView operator interface is logically organized so that the operator can easily move horizontally or vertically within the program (as shown in Figure 10). This allows the operator to switch between manual and automatic modes and quickly check the output status of the system.

Figure 10: Flow diagram of the PanelView Touch-screen Interface

The system is also protected against unauthorized use or accidental alterations to the program steps by a two-password system. To operate the system in manual and automatic modes, the primary password needs to be entered. However, in order to make changes to the program, a secondary password must also be entered.

The manual mode is utilized to run parametric tests and insure that all components are properly functioning. The manual mode is accessed through the Maintenance Screen. In this mode, the operator can test individual component by pressing the respective button

on the touch screen panel. The manual mode is also used to prime the system with the Sonatol® solution during the Start-up Procedure.

The automatic mode is accessed through the Process Interface Screen and is used for the automated running of the system. The automatic mode is preprogrammed and only requires the operator to press the start button on the touch screen panel. The Process Interface Screen presents the operator with the system status, i.e. step number and the elapsed time. The operator can also access the output of various sensors, and determine the pressures, turbidity, and ultrasonic status.

3.6 SYSTEM LIMITS

The Sonatol decontamination system is designed to ease operation if safety limits are exceeded.

Pressure Limits

The system contains five pressure sensors. Pressure limits are as follows:

<u>SENSOR</u>	<u>FUNCTION</u>	<u>MAX. VALUE</u> <u>(psi)</u>	<u>REASON</u>
P-1.....	Monitor Liquid Level in Cleaning Chamber.....	0.35.....	Prevent Overflow
P-2.....	Monitor Liquid Level in T-1	0.65.....	Prevent Overflow
P-3.....	Monitor F-2 Upstream Pressure.....	60.0.....	Max. Sensor Reading
P-4.....	Monitor J-2 Outlet Pressure.....	30.0.....	Max. Pump Pressure
P-5.....	Monitor F-2 Downstream Pressure	60.0.....	Max. Sensor Reading

These pressure limits are integrated into the alarm levels of the system operating software given in Appendix J.

Turbidity Limits

The system operating software also establishes an upper turbidity limit of 10 NTU as differentiating between “dirty” and “clean” liquid. The system will repeat a cycle if the turbidity at the end of a procedure is higher than 10 NTU. This is equivalent to a cerium oxide concentration of 0.001 g/L above baseline (i.e. pure liquid), as noted in Figure 6 of Appendix A1.

Temperature Limits

Thermocouple TC-1 monitors but does not control the temperature of the liquid in the ultrasonic bath. The system has been operated successfully over a temperature of about 70°F (21°) to 120°F (50°C). System operation should be avoided outside this temperature range. If the temperature is too low, the process liquid will be difficult to degas, and it will not sonicate properly. If the temperature is too high, this will have an adverse effect on the adsorption of surfactant in the adsorption column.

Cautionary Note

The ultrasonic power supply should never be operated without at least one inch (2.5 cm) of liquid in the bath. Operating a dry bath is likely to damage the system.

4.0 EXPERIMENTAL PROGRAM

4.1. MATERIALS

4.1.1 *Transuranic Wastes of Concern*

The legacy wastes consist of heterogeneous job control waste primarily contaminated with plutonium-238 oxide. The drum is lined with a polyvinyl chloride liner. Waste is placed in the drum in four to six layers of polyvinyl chloride bags. Drums may contain numerous waste bags. Waste contents include polyvinyl chloride, cellulose, metals, and other job control waste, and may include cans and bottles (both metal and plastic) containing hazardous liquid. Tables 4.1.1-1 and 4.1.1-2 summarize the range of organic and inorganic materials that may be found in a typical waste drum (Ref. 21). The composition of the major components of the waste that is found in an average drum is presented in Table 4.1.1-3. This table also presents the volumetric composition of a drum for two bounding cases of interest to the DOE. Bounding case A is for a drum that primarily contains lead lined gloves and other gloves. Bounding Case B is for a drum that mainly contains organic materials, as well as hazardous materials, such as used Oil Dry adsorbent, partially full aerosol cans, and mercury thermometers.

4.1.2 *Simulated Waste Used in Study*

The various materials and components used as simulated waste in the experimental program are listed in Table 4.1.2-1. These include a broad range of both organic and inorganic materials. These materials were chosen based on the following criteria:

- a. They were representative of materials and components found in the actual contaminated wastes at SRS.
- b. They were sized to fit in the bench scale cleaning equipment developed under the present program. For example, the smallest commercially available tin can and a pocket size aerosol dispenser were used in the experiments. Similarly, 1/4" pipe fittings were primarily used because of their small size.

Materials that exhibited minimal fluorescence when exposed to "black light" were used whenever possible. For this reason, the parametric testing trials were primarily carried out with aluminum and black polyethylene coupons that did not fluoresce.

The actual composition of the materials used in the process development tests and the demonstration cold tests are presented in subsequent sections in which these specific tests are discussed.

**TABLE 4.1.2-1 - MASS CONCENTRATION OF ORGANIC MATERIAL
IN TRANSURANIC WASTE DRUMS**

Item	Physical Description	Mass Concentration (wt-%) in Transuranic Drums	
		Average	Maximum
Polyvinylchloride	Huts/Bags 8-20 mils	1-5	90
	Bottles (reagent)	1-5	50
	Pipe 1/2" - 1" diameter/3' long	0-2	90
	Tape (plastic, nylon, electrical)	0-1	5
	Hose (breathing, Nylobrade)	0-1	50
	Plastic Shoe covers	0-5	50
	Protective Clothing (Plastic Suits)	0-2	50
	Films (Plastics)	0-1	10
	Plexiglas	0-2	50
	Valves	0-5	20
	Valve Handles	0-2	10
	Lexan	0-2	50
	Gloves (Nitride, Acid)	0-2	5
	5 gallon bottles, carboy bottles	0-1	20
Polyethylene	90 mil drum liner (separate - 16 lbs/drum)		
Polystyrene	1 liter bottle	0-1	20
Polypropylene	Kim Wipes	0-1	5
	Filters	0-1	10
	Celite	0-2	5
	Kotex	0-5	50
	Cloth-Coveralls	0-10	100
	Mop Heads (Manmade & Natural Fiber)	0-3	50
	Craft Paper	0-5	70
	Masking Tape	0-1	5
	Cartons	0-2	5
	Wood (planks, HEPA Housings)	0-1	90
	Sponges	0-5	20
	Paper	0-2	5
	Celite	0-2	5
	Kotex	0-5	50
Cellulose	Cloth-Coveralls	0-10	100
	Mop Heads (Manmade & Natural Fiber)	0-3	50
	Craft Paper	0-5	70
	Masking Tape	0-1	5
	Cartons	0-2	5
	Wood (planks, HEPA Housings)	0-1	90
	Sponges	0-5	20
	Leather Gloves	0-1	10
	Ceiling Panels	0-1	90

**TABLE 4.1.2-1 - MASS CONCENTRATION OF ORGANIC MATERIAL
IN TRANSURANIC WASTE DRUMS**

Item	Physical Description	Mass Concentration (wt-%) in Transuranic Drums	
		Average	Maximum
	Oil Dry	0-5	50
	Filters (cartridges)	0-5	20
Rubber	Dry Box Gloves - Non Leaded	0-10	90
	Wet Box Gloves - Leaded	0-10	90
	Dry Box Gloves - Leaded	0-10	90
	Surgeon's Gloves	0-2	5
	Rough Grip Gloves	0-2	5
Miscellaneous**	Cleaner (BH-38)	0-1	10
	Isoclean*	0-1	10
	Scene*	0-1	10
	Magnaflux*	0-1	10
	Spray Paint (Liquid)	0-1	10
	Bleach*	0-1	10
	Oil*	0-1	10
	Calcium Oxide	0-5	10
* May be included in bottles, cans, or absorbent wiping materials (cellulose)			
** Bottles, cans or absorbent material may also contain trace quantities of Resource Conservation and Recovery Act chemicals			
Source: U.S. Dept. of Energy, National Energy Technology Laboratory, PRDA No. DE-RA26-01NT40981, Attachment A, December 21, 2000.			

Table 4.1.2-2 – Mass Concentration of Inorganic Material in Transuranic Waste Drums

Item	Physical Description	Mass Concentration (wt-%) in Transuranic Drums	
		Average	Maximum
Metals		0-20	90
Carbon Steel	Spray Paint (container)		
	Tools		
	Cans		
	Pipe		
	Tubing		
	Hardware		
	Hot Plate		
Stainless Steel	Instruments		
	Motors		
	Scales		
	Agitators		
	Valves		
	Valve Handles		
	Beakers		
	Cans		
	Pipe		
	Equipment		
Tin	Button Cans		
	Oxide Cans		
Copper	Wiring		
	Tubing		
Lead	Aprons		
	Bricks		
	Acrylic Lead		
Cadmium	Sheets	0-5	20
Platinum	Wires, Sheets, and other small structural forms	0-5	20
Ceramics	Glass	0-2	90
	Beakers	0-2	20
	Sheet Rock	0-2	90
	Miscellaneous	0-2	90
	Thermometer (Mercury)	0-1	5
Rubber	Concrete	0-5	90
	Sand (MgO ₂)	0-5	50
	HEPA Filters (Media)	0-5	50
Resins	Cation/Anion	0-5	50
Source: U.S. Dept. of Energy, National Energy Technology Laboratory, PRDA No. DE-RA26-01NT40981, Attachment A, December 21, 2000.			

TABLE 4.1.2-3 SURROGATE WASTE MATRICES PROPOSED BY SRS				
Case	Weight Kg/Drum	(wt-%) Contents	(wt-%) w/Drum	Volume Percent
Avg. Drum Representation Case				
Iron Base Alloys	2.47	10.3	4.2	
Other metals/alloys	0.06	0.3	0.1	
Oil Dry (Kitty Litter)	1.74	7.3	2.9	
Paper/Wood	9.5	39.6	16.0	
Lead Lined Gloves, gloves, gaskets	0.21	0.9	0.4	
PVC, Polyethylene	10	41.7	16.9	
Sub-total	23.98	100.0	40.4	
Liner Container Material	6.65		11.2	
Steel Container Material	28.71		48.4	
Sub-total	35.36		59.6	
Total Weight	59.34		100.0	
Bounding Case A (*)				
Lead Lined Gloves & Other Gloves				60
PVC/Poly bags & sealing tape				10
Metal pieces (fittings)				10
Paint cans (empty and closed)				20
Total				100
Bounding Case B (*)				
Organics (alcohols) on wipes				30
Oil Dry (kitty litter)				20
Aerosol Cans with Cleaner				4
Mercury Thermometer				1
Polypropylene				40
HEPA Material				5
Total				100
* For testing purposes, smaller quantities may be prorated from the data provided				
** Values for bounding cases are by volume, and do not include the drum liner and drum				
Source: V. Bhambri, SRS, December 10, 2002				

TABLE 4.1.2-4 – SURROGATE WASTE MATERIALS USED IN EXPERIMENTAL PROGRAM**Number Organic Materials**

O-1	Black Polyethylene Film, 1.5" x 1.5" x 0.006"
O-2	Aged Blue Reinforced Polyethylene Tarpaulin, 1.5" x 1.5" x 0.0045"
O-3	Clear Polyethylene Coupons from Gloves, 1.5" x 3.0" x 0.001"
O-4	Lead Lined Rubber from Gloves, 1.5" x 1.5" x 0.22
O-5	Buna N O-ring, Size 118, 7/8" ID x 3/32" width
O-6	Neoprene Coated Fabric from Gloves, Tips and Fingers, 0.054" thick
O-7	Yellow Latex Rubber from Gloves, Tips and Fingers, 0.040" thick
O-8	Nitrile Coated Polyester Apron Coupons, 1.5" x 1.5" x .014"
O-9	Electrical Tape, Black, 0.75" x 3.0" x 0.013"
O-10	Polypropylene, Black, Hex Nipples, 1/2" NPT
O-11	Polyvinyl Chloride Close Nipples, 1/2" NPT
O-12	Wood Coupons, 1.5" x 1.5" x 0.060"
O-13	Wood Popsicle Sticks, 0.75" x 0.50" x 0.083"
O-14	Alcohol Wipe Fabric, 2.0" x 2.5" x .003"
O-15	Black Napkin Paper, 1.5" x 3.0" x 0.0014"
O-16	Black Card Stock, 1.5" x 1.5" x 0.016"

Number Inorganic Materials

I-1	Aluminum Alloy 2024 Sheet Coupons, 1.5" x 1.5" x 0.022"
I-2	Brass Coupling, 1/4" NPT
I-3	Brass Square Head Plug, 1/4" NPT
I-4	1/4-20 Stainless Steel Nuts
I-5	1/4-20 x 1/2" Long Hex Head Stainless Steel Machine Screws
I-6	1/4" NPT Stainless Steel Street Elbow
I-7	Stainless Steel Reducing Bushing, 3/8" MNPT to 1/4" FNPT
I-8	Eyeglass Cleaner in Aluminum Aerosol Can, 1" Diameter x 4.5" long
I-9	(Tin Paint) Can, 1/4 pint capacity, 2-1/16" H x 2-1/8" Diameter
I-10	Alcohol Wipe Packaging (mainly Al), 1.5" x 3.0" x 0.0035"
I-11	Thermometer, Mercury in Glass, 1/4" Diameter x 6" Long
I-12	Glass Ampoule, Sealed, 3/8" Diameter x 2" Long
I-13	Vermiculite Medium Texture Bulk Sorbent
I-14	Corrugated HEPA Filter Medium (Boron Silicate with Waterproofing Binder) Coupons, 1.5" x 1.5" x 0.050"

4.1.3 Simulated Radionuclides

In the cold testing experimental program, the simulated waste was contaminated with two plutonium oxide surrogates. These were:

- a. Osram Sylvania Phosphor No. 2284C
- b. Cerium Oxide (Ce(IV)O₂) Polishing Compound (Alfa-Aesar No. 39802)

Phosphor 2284C is a fluorescent powder that is compatible with the photodigital method of surface analysis developed by ESI to support its surface cleaning activities. This method, discussed in more detail in Section 4.2.1, provides a rapid and inexpensive means of measuring the level of cleanliness, or decontamination factor, achieved in a cleaning operation. The principal physical properties of this material are given in Table 4.1.3-1

Table 4.1.3-1 Properties of Phosphor 2284C used as a Simulated Radionuclide

Chemical Formula.....	ZnSiO ₄ :Mn
Molecular Weight.....	226
Specific Gravity (H ₂ O = 1)	4.1
Emission Color	Green
Emission Peak.....	528 nm
Emission Bandwidth (50%)	40 nm

The fluorescent powder used in the present program was supplied as aqueous slurry that had a reported volume-average suspended particle size of 0.88 μm. The as-supplied material was partially agglomerated, and the material contained a significant population of smaller particles, as evidenced by the scanning electron micrograph presented as Figure 11.

Cerium oxide (Ce(IV)O₂) was also used in the experiments because it is a generally accepted chemical analogue of plutonium oxide. The particular grade of material used in the experiments is the finest particle sized material sold by Alfa-Aesar, the premium supplier of high purity inorganic chemicals in the country. This material is sold as containing particles smaller than 0.3 μm. This claim is substantiated by the scanning electron micrograph presented as Figure 11.

The simulated waste was contaminated by spraying the various items with dilute (~1000-ppm) suspensions of simulant powders in a volatile, low surface tension liquid (3M's HFE-71-IPA). This method was used in order to be able to uniformly and

reproducibly deposit the contaminants on the substrates of interest as well as to maximize the state of dispersion of the deposited powders on these surfaces. The mass of simulant applied to the surfaces of the surrogates was not determined because the errors associated with the determination of mass were potentially large. A mass balance approach would require accounting for over spray and run off of the dilute suspensions or limiting the area of the sample that could be coated. The NAA approach allowed us to measure actual cerium concentration of the samples after the carrier fluid evaporated. This approach was considered to be more accurate than calculating the mass from the volume of dilute suspension applied to each sample.

The particles in the as received aqueous suspension were transferred from the initial aqueous phase to the dispensing solvent, HFE-71-IPA, by a process of solvent exchange. In this process, the initial suspension medium, water, was replaced by isopropanol (IPA), and then the isopropanol was replaced by the HFE-71-IPA. In this manner, each sequential washing liquid is miscible with the prior suspending liquid. Experimentally, this entailed allowing the aqueous suspension to stand in a large wide mouth jar for sufficient time (days) for the particles to settle out of the supernatant liquid. The supernatant liquid is then decanted, and replaced with isopropanol. The jar is sealed and placed on a rotating mill for four hours to mix the contents. After removing the jar from the mill, it is placed on a table, and the suspended material is provided enough time to settle. The supernatant liquid is replaced by fresh isopropanol. The procedure is repeated once more with isopropanol; and then three times with HFE-71-IPA. This results in the formation of a master batch of concentrated suspension (~50,000 ppm). Aliquots of this concentrate were added to HFE-71-IPA to form the ~1200 ppm suspensions that were ultimately applied to the simulated waste. The suspensions were sonicated at 132 kHz for 1 hour when bottled. The important properties of HFE-71IPA are given below:

Table 4.1.3-2 Properties of HFE-71IPA

Methoxy-Nonafluorobutyl Ethers	95.5% by weight
Isopropanol	4.5% by weight
Boiling Point (Azeotrope), °C.....	54.8
Freeze Point, °C	-42
Liquid Density @ 25°C, g/ml	1.48
Surface Tension @ 25°C, dynes/cm.....	14.5
Vapor Pressure @ 25°C, torr	207
Heat of Vaporization @ bp, cal/g	39.5
Flashpoint.....	None
Flammability Range in Air	4.0 -16.7%
Ozone Depletion Potential.....	Zero
Exposure Guidelines, 8 hr TWA, ppm.....	750 (HFE-7100)/400 (IPA)

Figure 11 SEM of Osram Sylvania Phosphor No. 2284C Powder used as Cold Test Contaminant

Figure 11 SEM of Alfa-Aesar Submicron Sized Cerium(IV) Oxide Powder

Mixed Cerium (IV) Oxide/Phosphor Suspension: A cerium oxide master suspension was prepared adding dry powder to HFE 71-IPA to form a 1 wt-% suspension of cerium oxide powder. This suspension was then sonicated for 1 hour at 132 kHz. Aliquots of this material were then added to the ~1200 ppm phosphor suspension to make a mixed suspension that contained ~ 750 ppm ceria and 1000 ppm phosphor.

Contaminating the Waste: The suspensions are transferred to a small (8 oz.) plastic spray bottle. The components of the surrogate waste are then contaminated by spraying the suspension and letting the liquid evaporate. This results in the uniform deposition of the contaminants on the surfaces being coated.

This method of applying the contaminants was used in order to maximize the uniformity of deposition and to minimize the level of agglomeration of the contaminant powders once deposited. A characteristic of sub-micron solid particles is that they tend to agglomerate spontaneously, so that powder beds of these materials consist mainly of agglomerates whose geometry (agglomerate size distribution and packing density) are history dependent (Ref. 24). Shaking the objects in a bed of contaminant powder results in the contamination level that was due mainly to the attachment of relatively large agglomerates, which are much easier to remove than the primary particles that make them-up. The level of deposition is hard to control, and would tend to be on the high side. With this method of deposition it would be hard not to obtain high decontamination factors, even under circumstances that would result in little decontamination under hot testing conditions.

Applying the powder from suspension in HFE-71-IPA results in the deposition of well-dispersed powders. The powder suspensions in this liquid settle very slowly, less than 1 cm/hr. Based on Stokes' Law, this settling rate is characteristic of particles that have an equivalent spherical diameter of less than 1 μm . The combination of high liquid volatility and low surface tension prevents the particles from reagglomerating after deposition, and thus leaves behind a uniform powder deposit. This liquid has a very low (<1 ppm) level of non-volatile residues that could affect the surface behavior of the deposited particles.

4.1.4 Sonatol Solutions

The process liquids used in the Sonatol® process are a dilute solution (less than 1 wt-%) of a fluorinated surfactant (Krytox 157FSL) in perfluoroheptane (C_7F_{16}) as the dispersing solution and neat perfluoroheptane as a rinse. The properties of perfluoroheptane (Sonatol A70) are given in Table 4.1.4-1. The MSDS is provided in Appendix E.

Table 4.1.4-1 Comparative Properties of Sonatol® A70 and Water		
	Sonatol® A70	Water
Chemical Formula:	C7F16	H2O
Average Molecular Weight:	388	18
Boiling Point, °C	80	100
Pour Point, °C	-95	0
Vapor Pressure @ 20°C, torr	79	24
Density, g/ml	1.73	1.00
Surface tension, dyne/cm	13	72
Viscosity, cs	0.56	0.9
Specific Heat, cal/g-°C	0.25	1.00
Heat of Vaporization @ BP, cal/g	19	540
Hildebrand Solubility Parameter, (MPa ^{-0.5})	12.3	47.9
Hydrogen Bonding Capability	Very Low	Very High
Flammability	None	None
Flash Point, °C	None	None
Flammability Limits, vol-% in air	None	None
Volatile Organic Compound (VOC) Limits, ppm	NA	NA
Ozone Depletion Factor (CFC11 = 1.0)	0	0
Total Worker Exposure Limits per 8 hr day, ppm	None	None

These perfluorinated liquids have zero ozone depletion potential, are nontoxic, nonflammable, and are generally recognized as non-hazardous materials. Under the Significant New Alternatives Program (SNAP) of the U.S. Environmental Protection Agency (EPA), these materials have been found to be an environmentally acceptable means of cleaning highly complex geometric configurations that must meet stringent particulate cleanliness standards.

They are generally innocuous to all materials including paper, plastics, metals, metal salts and oxides, wood and cloth. Not only are they compatible with the processed items, but also they do not react irreversibly with the particles being removed. Perfluoroheptane has a high specific gravity (s.g. 1.7), low viscosity (less than 1 cP), and a low surface tension (less than 15 dynes/cm), which facilitates wetting and penetration into holes and crevices. Sonatol A70, which boils at 80°C and has a heat of vaporization of 20 cal/g (less than 3% of that of water), is easily recycled and recovered by distillation.

The surfactant, Krytox 157FS (E.I. Du Pont de Nemours & Co., Inc.), is a carboxylic acid terminated oligamer of hexafluoropropylene oxide with an approximate molecular weight of 2,300 Dalton. It is a dense (S.G. = 1.9), nonhazardous and nonvolatile viscous liquid.

Recently, ESI carried out some IRAD studies to examine the removal of closely sized fluorescent polystyrene beads of different sizes from aluminum coupons in perfluorinated solutions under the following experimental conditions:

Particle Size, μm	0.1, 0.26, 0.94
Carrier Liquid	Sonatul A 70
Surfactant Concentration	0, 0.1 wt-%, 0.2 wt-%, and 0.4 wt-%
Ultrasonic Bath:	Blackstone-Ney MultiSONIK™
Ultrasonic Frequency, kHz	40, 72, 104 and 170
Ultrasonic Power Density	100 watts/gal

In these experiments, maximum particle removal was obtained at 104 kHz. At this frequency, particle removal with a 0.2 wt-% surfactant solution was better than in a 0.1 wt-% surfactant solution, which in turn, was significantly better than in pure Sonatul A 70. Increasing the concentration from 0.2 wt-% to 0.4 wt-% had little effect on the level of cleaning attained. The results of these experiments are presented in Appendix D. A minimum surfactant concentration of 0.4 wt-% in the wash solution was used in the present experimental program rather than 0.2 wt-% or 0.3 wt-% in order to have a margin of safety between the operating surfactant concentration and the concentration range of 0.1 wt-% at which performance drops off.

4.2 ANALYTICAL PROCEDURE

DPA was startup testing and as a quality control procedure during integrated cold testing. NAA was used to measure the DF of process for the surrogate wastes.

4.2.1 DPA

The fluorescent properties of the phosphor particles were used to measure their concentration on the test items before and after decontamination. Trace levels of these particles are easily detected by eye, or in photographs, when the surface being examined is illuminated with ultraviolet light (364 nm). Photographs are not only useful for giving a general idea of the cleanliness of the substrate, but can also provide semi quantitative information on the levels of surface contamination. This is accomplished by digitally analyzing these images on a personal computer with image analysis software. To quantify these observations, a procedure was developed in which photographs of the substrates being processed were captured with a digital camera (Cool-Snap Pro 36 color camera) and then digitally analyzed on a personal computer with Media Cybernetics' Image Pro Express image analysis software. Figure 12 shows a picture of the camera setup.

Figure 12: Digital Camera Setup

Before discussing the method in more detail, it is useful to review how computers store and read picture images. Each picture is stored as a collection of small dots, called pixels, with each pixel having a distinct color. Each pixel is defined by some intensity of three different colors: red, green, and blue. A digital TIFF (.tif) photograph is made up of many thousands of pixels. Each pixel has a certain intensity that dictates how bright it is. Image Pro Express has the capability to break a color image down into 4096 red, blue and green channels; and give a histogram of the number of pixels of each color at intensity values ranging from 0 to 4095. A digital TIFF (.tif) photograph is made up of many thousands of pixels. Each pixel has a certain intensity that dictates how bright it is. Each color has 4096 intensities, ranging in value from 0 to 4095. It is a combination of these three colors at different intensities that produce every color seen in the picture. This allows for a combination of up to 10^{10} colors (4096^3) that can be displayed. By definition, black has a red-green-blue intensity value of 0-0-0, while white has a red-green-blue intensity value of 4095-4095-4095. A bright orange color may have red-green-blue intensity of 4000-1500-300, as an example.

The first step of analysis is to acquire an image of the blank coupon and determine the average Green Pixel Luminosity (GPL) per unit area for the area of interest. The software program has a histogram function that analyzes the area of interest and displays the intensity levels of the red, blue and green channels on the X-axis, and a count of the pixels at each intensity level on the Y-axis. The area under the curve is known as the luminosity. Our interest is in the GPL since the contaminant, 2284c Phosphor, fluoresces in the green. The GPL is then divided by the area of the coupon, in square pixels, to provide an average GPL/Area value for the coupon. Figure 13 is an example of a histogram for a blank aluminum coupon.

Figure 13: Histogram for Blank Aluminum Coupon

The next step is to contaminate the blank coupon and then acquire an image of the contaminated substrate. Figure 14 shows a picture of a contaminated aluminum coupon and its corresponding histogram.

Figure 14: Histogram for Contaminated Aluminum Coupon

The last step is to clean the coupon and then acquire an image of the processed coupon. Figure 15 presents a histogram of a processed aluminum coupon.

Appendix B provides data showing that the GPL/A values are linear over the range of area contamination loading used in this study. The process may not be linear, however, if contaminants of differing luminosities and DF values were combined in a single test. Therefore the DPA process is used in this study as a semi quantitative indicator of decontamination effectiveness and NAA is used to quantify DF values.

Figure 15. Processed Aluminum Coupon and Corresponding Histogram

The average GPL/A values for the blank, contaminated, and processed coupons can now be used to estimate the corresponding decontamination factor (DF). The DF of a cleaning operation is defined here as the ratio of the concentration of contaminant present before cleaning to the concentration of the contaminant present after cleaning, and is expressed by the following equation:

$$DF = \frac{(\text{GPL/A}_{\text{Contaminated}} - \text{GPL/A}_{\text{Blank}})}{(\text{GPL/A}_{\text{Processed}} - \text{GPL/A}_{\text{Blank}})} \quad (\text{Eq. 4.2.1})$$

An appropriate GPL/A blank value must be obtained for each type of material. In some cases the GPL/A for a processed coupon was less than the unprocessed blank itself. In these instances, the standard deviation was used as the value of the denominator in the calculation of the reported value of the DF. An error analysis test was performed for the denominator of the above equation, which is the net value of the processed aluminum and plastic coupons, to determine the range of values that one processed coupon would return in terms of the GPL/A. A processed aluminum coupon was placed on the digital analysis background, its image was recorded, and then the coupon was removed. This process was performed ten times such that ten images were captured for both aluminum and plastic.

The statistics for the ten images were:

<u>Material</u>	<u>GPL/A</u> <u>Average</u>	<u>Standard Deviation of</u> <u>DF Denominator</u>
Aluminum.....	190.1.....	4.20
Plastic	107.4.....	3.25

4.2.2 Neutron Activation Analysis:

Neutron activation analysis (NAA) is a sensitive analytical technique useful for performing both qualitative and quantitative multi-element analysis of major, minor, and trace elements in samples from almost every conceivable field of scientific or technical interest. For many elements and applications, NAA offers sensitivities that are superior to those attainable by other methods, on the order of parts per billion or better. In addition, because of its accuracy and reliability, NAA is generally recognized as the "referee method" of choice when new procedures are being developed or when other methods yield results that do not agree. (http://www.missouri.edu/~glascock/naa_over.htm)

The method is based on the detection and measurement of characteristic gamma rays emitted from radioactive isotopes produced in the sample upon irradiation with neutrons. Typically, unknown samples together with standard materials of known elemental concentrations are irradiated with thermal neutrons in a nuclear reactor. After some appropriate decay period, high-resolution gamma ray spectroscopy is performed to measure the intensity and energies of the gamma lines emitted. A comparison between specific activities induced in the standards and unknowns provides the basis for computation of the abundance of the elements of interest.

The process of measurement of the gamma ray spectra following neutron irradiation is generally performed using a resolution germanium semiconductor detector. These devices provide sufficient resolution to differentiate between most all commonly occurring gamma lines. The MITNRL uses 4 high purity germanium detectors to support its NAA activities. The series of samples were irradiated in one of the MITNRL's pneumatic tubes for 12 hrs. The total thermal neutron fluence each sample received is about 3×10^{17} n/cm². Each sample was then counted for 6 hours to obtain the gamma ray spectrum. A selected gamma ray energy peak, which is the characteristic gamma ray of the radioisotope of interest, was then integrated to obtain the total gamma ray counts.

The phosphor and cerium oxide assays were obtained by examining the following gamma ray emissions:

<u>CONTAMINANT</u>	<u>CERIUM (IV) OXIDE</u>
Target isotope (natural abundance)	Ce-140 (88.5%)
Radioactive isotope	Ce-141
Half-life, days	32.5
Gamma Ray Energy, keV	145

4.3 PARAMETRIC CLEANING TESTS

The parametric tests focused on determining the optimal conditions or parameters for specific portions of the decontamination process. Table 4.3-1 summarizes the tests.

Table 4.3-1 Parametric Test Matrix

# of Runs	Title	Basket	Bath Chemistry	Substrates	Means of Applying Shear	Ultrasonic Frequency (kHz)	Temperature
4	Spray Tests	ADF	Neat Sonatol A70	Aluminum and Plastic	Spray	N/A	Room (70°F - 85°F)
4	Spray Tests	ADF	0.4% Krytox	Aluminum and Plastic	Spray	N/A	Room (70°F - 85°F)
16	Initial Ultrasonic Tests	ADF	Neat Sonatol A70	Aluminum and Plastic	Ultrasonic	40 - 170	Varied
16	Initial Ultrasonic Tests	ADF	0.4% Krytox	Aluminum and Plastic	Ultrasonic	40 - 170	Varied
6	Baffle Tests	ADF	0.4% Krytox	Plastic	Ultrasonic	104	Varied
8	Material Interference Tests	Varied	0.4% Krytox	Plastic	Ultrasonic	104	Room (70°F - 85°F)
1	Tests with Improved Basket	Rotating Basket 2	0.4% Krytox	Mixed Waste Lot	Ultrasonic	104	Room (70°F - 85°F)

The main variables examined were the bath chemistry and temperature, means of applying shear, basket design, and coupon agitation. Tests performed in neat Sonatol A 70 showed little or no contaminant removal, while those in 0.4% Krytox solutions showed significant removal under most test conditions. Ultrasonic tests outperformed spray tests with the highest level of decontamination occurring at 104 kHz. A lighter basket design with a greater percent open area caused less ultrasonic interference and resulted in significantly better decontamination than heavier baskets with less open area. Rotation of the basket increased coupon movement and coincided with better decontamination. There was little effect of temperature over the range of 70° F (21°C) to 120°F (50°C).

The materials test clearly shows that the ADF basket material has a large effect in damping the ultrasonic wave intensity. The test also revealed that lighter materials with greater open area have less of a damping effect on the ultrasonics than heavier materials with a lower open area. The test also showed that the opening in the basket material should be greater than 6 mm (1/4").

Table 4.3-2 Effects of Materials on Ultrasonic Agitation

Material	Thickness (inch)	% Open Area	Visible Loss in Intensity		
			No Effect	Small Effect	Large Effect
Aluminum Sheet	0.016	0	X		
Aluminum Sheet	0.032	0	X		
Steel Foil	0.002	0	X		
Copper Sheet	0.060	0		X	
Perforated Steel	0.036	63		X	
Perforated Steel	0.060	51			X
2-Mesh Screen	0.047*	82.1	X		
3-Mesh Screen	0.042*	73.6	X		
4-Mesh Screen Welded	0.032*	76	X		
4-Mesh Screen	0.047*	65.9	X		
6-Mesh Screen	0.035*	62.7			X
10-Mesh Screen	0.025*	56.3			X
20-Mesh Screen	0.016*	46.2			X
60-Mesh Screen	0.0075*	30.5			X

** FOR SCREEN MATERIAL, VALUE PRESENTED IS THE WIRE DIAMETER*

To confirm the suspected correlation between ultrasonic wave interference and poor cleaning, a series of material interference tests were performed with baskets of different material. The first test was to simply place the plastic coupons inside the ultrasonic bath, without any basket. Five coupons were placed inside the cleaning chamber and sonicated at 104 kHz. After five minutes three of the coupons were removed, and after ten minutes, the remaining two coupons were removed. Removal of the basket resulted in significantly higher decontamination factors.

Table 4.3-3 Preliminary Waste Compositions

Item	Quantity	Item	Quantity
Tin Paint Can Cover	1	Stainless Steel Bushing	1
Aluminum Coupon	2	Stainless Steel Elbow	1
Black Polyethylene Coupon	2	Lined Neoprene Glove Tips	2
Rubber Apron Coupon	1	Lined Neoprene Glove Fingers	2
Clear Polyethylene Glove Tip	2	Yellow Rubber Glove Tips	2
Black Napkin Strip	2	Yellow Rubber Glove Fingers	2
Stainless Steel Union	1		

The mixed waste was sonicated at 104 kHz in rotating basket 2 for ten minutes at room temperature. Blank, contaminated, and clean images of selected items are presented in Section 4.6. In all cases, the processed items were visibly cleaner than the entering blanks. Table 4.3-4 confirms this quantitatively by presenting the GPL/A values for the blank, contaminated, and clean pieces that could be photo digitally analyzed.

Table 4.3-4 Preliminary Waste Trial Statistics

Sample	Sample	Blank GPL/A	Contaminated GPL/A	Clean GPL/A
Aluminum Coupon	1	127	1658	106
	2	147	1801	122
Black Polyethylene Coupon	1	90	668	87
	2	91	708	93
Stainless Steel Union	1	155	890	102
Stainless Steel Bushing	1	213	899	140
Stainless Steel Elbow	1	302	1957	198
Tin Paint Can Cover	1	130	1700	103
Rubber Apron Coupon	1	591	1471	498

In the above table, the GPL/A of the clean sample was lower than the GPL/A of the blank before contamination, except for the second set of black polyethylene coupons, where the difference is not significant. For most items, and especially the metal fittings, the cleaning process not only removed the fluorescent contaminants that were applied, but also any prior surface contaminant that may have existed on blank items.

Initial blank pieces, such as the tin paint can cover and the stainless steel fittings, were pulled directly from the package such that minor surface residue may have existed (i.e. oil or particulates).

4.4 FILTRATION TESTS

The removal of sub-micron sized Pu-238 particles from the surface of waste articles by sonication in Sonatol® surfactant solutions is the first step in the Sonatol® process. For the process to be successfully applied, it is also necessary to be able to remove the suspended particles from the process solutions so that these may be recycled to the cleaning chamber. Filtration of Sonatol® surfactant solutions through ceramic filters was considered to be an effective strategy for the removal and capture of detached Pu-238 particles because:

1. Inorganic compounds, including plutonium oxides, are insoluble in the Sonatol® surfactant solutions.
2. Ceramic filters are stable in a radiological environment. They do not contain hydrogen in their structure, and thus cannot generate hydrogen gas in the presence of Pu-238 particles. This is in contrast to standard filters made of polymeric materials that will generate gaseous hydrogen when subjected to alpha radiation.

Ceramem™ multichannel, alumina coated, cross-flow filters with a 0.2 µm nominal pore size were qualified for the removal step was investigated by challenging them with Sonatol® surfactant solutions in which 0.3 µm cerium (IV) oxide powder or 0.2 µm silica powder were suspended as Pu-238 simulants

As discussed in more detail in Appendix A, alumina membrane ceramic filters effectively remove 99.9% of dispersed sub-micron sized particles (cerium oxide and silica oxide) in a single pass from a fluorinated surfactant solution, as measured by turbidity.

Both Ceramem™ filters have a high soil loading capacity in a dead-end mode, and can be back flushed, if desired.

4.5 ADSORPTION TESTS

The final step in the Sonatol® PU-238 decontamination process is to rinse the processed parts with surfactant free PFC. This is to remove any radioactive contaminants that might have been trapped in dried-out surfactant film residue. This solvent is obtained by passing surfactant solution through a bed of activated carbon to remove the Krytox 157FS surfactant by adsorption.

The rate of adsorption of a polar contaminant from solution in a fluorinated liquid onto activated carbon is significantly faster for activated carbon fabrics than for granular activated carbon (GAC). The specific adsorption capacity of activated carbon fiber felt,

especially at low residence times, is much higher than the specific adsorption capacity of GAC. This is believed a consequence of the fact that a much larger percentage of the adsorbing pores are directly exposed to the flowing liquid for the felt than for the granules. In case of the granules, the mass transfer process is impeded by the need for the adsorbate to diffuse within the bulk of the granules to reach many of the adsorption sites.

The advantages of using an activated carbon felt adsorbent over GAC are:

- a. For an adsorber of a given volume, the operating capacity of the felt is higher than that of GAC by a factor of two to three.
- b. The higher the flow rate, or the shorter the available residence time, the greater the relative capacity of felt as compared to GAC.
- c. Pressure drop across felt packing is lower than that for granular packing.

Because of the limited amount of space available in a glove box, it was decided to use an activated carbon felt adsorber, rather than a granular activated carbon column, in the Sonatol® Pu-238 decontamination system.

The design and performance characteristics of the Activated Carbon Fiber (ACF) adsorption module are described in Appendix A. The activated carbon fiber module (including housing) was pressure tested to satisfy SRS safety requirements. These results are presented in Appendix C.

4.6 INTEGRATED COLD TESTS:

The detailed data log is provided in Appendix F.

4.6.1 Testing Conditions

Cold tests were designed to demonstrate the system's ability to remove contaminants from mixed waste lots. This basket used for integrated cold tests was a 4" diameter cylinder 10" in length made of 4-mesh 304 stainless welded screening that has a 75% open area. The ends were constructed from 0.036" thick perforated steel with 0.156" hole diameter and 63% open area. The end pieces were not made of mesh stock to facilitate their handling in a glove box and to eliminate sharp points that could perforate gloves used by an operator. Figure 7 shows a picture of the basket used in the integrated cold tests. In these cold tests, the test items were contaminated with both cerium oxide and 2284C Phosphor powders. Six different mixtures were processed according to three different cleaning protocols as outlined in Table 4.6.1-1.

Table 4.6.1-1 Integrated Cold Test Matrix

Run #	Test List Items	Initial Condition	Processed by Protocol	Krytox Concentration
1	1	Contaminated	Not Processed	N/A
2	2	Contaminated	Not Processed	N/A
3	3	Contaminated	Not Processed	N/A
4	1	Clean	1	0.40
5	2	Clean	1	0.54
6	3	Clean	1	0.65
7	1	Contaminated	1	1.13
8	2	Contaminated	1	1.19
9	3	Contaminated	1	1.09
10	1	Contaminated	2	1.38
11	2	Contaminated	2	1.55
12	3	Contaminated	2	1.58
13	4	Contaminated	2a	0.45
14	5	Contaminated	2a	0.52
15	6	Contaminated	2a	0.49

The items in runs 1 to 3 were contaminated and not processed. These items were used as “contaminated” samples for analysis. Items in runs 4 to 6 were processed blanks. The items in these runs were “as-received” items that had been processed to remove any surface contamination. These items were used as “blank” samples for analysis. The items in runs 7 to 15 were contaminated and then processed according to the protocols outlined in Table 4.6.1-1. The effectiveness of the process was obtained by analyzing these “cleaned” items.

The mixtures used in these tests are listed in Tables 4.6.1-2 and 4.6.1-3.

Table 4.6.1-2 lists the number of coupons of each item in each of the mixtures. Table 4.6.1-3 presents the weights of these components in the mixture, including the weight percent of organics in each mixture.

Mixture 1 is a representation of an average drum. Mixtures 2 and 3 are representations of different bounding conditions. Mixtures 4, 5, and 6 are variations of the previous three. Aluminum and plastic coupons were used as telltale markers in each mixture since quantitative photodigital analysis could not be performed on every item in a mixture due to fluorescence interference.

Samples of representative items were sent to the MITNRL for NAA, as outlined in Table 4.6.1-4, to confirm the results of the photodigital analysis performed at ESI.

Table 4.6.1-2 List of Test Items

		Mixture No.					
		1	2	3	4	5	6
Number	Organic Materials	Items per Mixture					
O-1	Black Polyethylene Film, 1.5" x1.5" x 0.006"	6	6	6	6	6	6
O-2	Aged Blue Reinforced Polyethylene Tarpaulin, 1.5" x 1.5" x 0.0045"						3
O-3	Clear Polyethylene Coupons from Gloves, 1.5" x 3.0" x 0.001"	2					
O-4	Lead Lined Rubber from Gloves, 1.5" x1.5" x 0.22	3	3		3		3
O-5	Buna N O-ring, Size 118, 7/8" ID x 3/32" width	2			2		
O-6	Neoprene Coated Fabric from Gloves, Tips and Fingers, 0.054" thick		3				
O-7	Yellow Latex Rubber from Gloves, Tips and Fingers, 0.040" thick	3					
O-8	Nitrile Coated Polyester Apron Coupons, 1.5" x 1.5" x .014"					3	
O-9	Electrical Tape, Black, 0.75" x 3.0" x 0.013"		2			2	
O-10	Polypropylene, Black, Hex Nipples, 1/2" NPT			3			3
O-11	Polyvinyl Chloride Close Nipples, 1/2" NPT	3					
O-12	Wood Coupons, 1.5" x 1.5" x 0.060"	3			3		
O-13	Wood Popsicle Sticks, 0.75" x 0.50" x 0.083"				3		
O-14	Alcohol Wipe Fabric, 2.0" x 2.5" x .003"			8			8
O-15	Black Napkin Paper, 1.5" x 3.0" x 0.0014"	2					
O-16	Black Card Stock, 1.5" x 1.5" x 0.016"					3	

Number	Inorganic Materials						
I-1	Aluminum Alloy 2024 Sheet Coupons, 1.5" x 1.5" x 0.022"	3	3	3	3	3	3
I-2	Brass Coupling, 1/4" NPT		1			1	
I-3	Brass Square Head Plug, 1/4" NPT		1			1	
I-4	1/4-20 Stainless Steel Nuts	10			10		
I-5	1/4-20 x 1/2" Long Hex Head Stainless Steel Machine Screws	10			10		
I-6	1/4" NPT Stainless Steel Street Elbow		1			1	
I-7	Stainless Steel Reducing Bushing, 3/8" MNPT to 1/4" FNPT		1			1	
I-8	Eyeglass Cleaner in Aluminum Aerosol Can, 1" Diameter x 4.5" long			1			1
I-9	(Tin Paint) Can, 1/4 pint capacity, 2-1/16" H x 2-1/8" Diameter		1			1	
I-10	Alcohol Wipe Packaging (mainly Al), 1.5" x 3.0" x 0.0035"			2			2
I-11	Thermometer, Mercury in Glass, 1/4" Diameter x 6" Long			1			
I-12	Glass Ampoule, Sealed, 3/8" Diameter x 2" Long			1			1
I-13	Vermiculite Medium Texture Bulk Sorbent						
I-14	Corrugated HEPA Filter Medium (Boron Silicate with			4			4
	WaterproofingBinder) Coupons, 1.5" x 1.5" x 0.050"						

Table 4.6.1-3 Weights of Test Items

		Mixture No.					
		1	2	3	4	5	6
Number	Organic Materials	Weight of Items (g)					
O-1	Black Polyethylene Film, 1.5" x1.5" x 0.006"	1.1	1.1	1.1	1.1	1.1	1.1
O-2	Aged Blue Reinforced Polyethylene Tarp, 1.5" x 1.5" x 0.0045"						0.5
O-3	Clear Polyethylene Coupons from Gloves, 1.5" x 3.0" x 0.001"	0.4					
O-4	Lead Lined Rubber from Gloves, 1.5" x 1.5" x 0.22	11.3	11.3		11.3		11.3
O-5	Buna N O-ring, Size 118, 7/8" ID x 3/32" width	0.9			0.9		
O-6	Neoprene Coated Glove Fabric, Tips and Fingers, 0.054" thick		19.3				
O-7	Yellow Latex Rubber from Gloves, Tips and Fingers, 0.040" thick	8.7					
O-8	Nitrile Coated Polyester Apron Coupons, 1.5" x 1.5" x .014"					2.3	
O-9	Electrical Tape, Black, 0.75" x 3.0" x 0.013"		1.1			1.1	
O-10	Polypropylene, Black, Hex Nipples, 1/2" NPT			24.5			24.5
O-11	Polyvinyl Chloride Close Nipples, 1/2" NPT	19.6					
O-12	Wood Coupons, 1.5" x 1.5" x 0.060"	4.5			4.5		
O-13	Wood Popsicle Sticks, 0.75" x 0.50" x 0.083"				0.6		
O-14	Alcohol Wipe Fabric, 2.0" x 2.5" x .003"			1.0			1.0
O-15	Black Napkin Paper, 1.5" x 3.0" x 0.0014"	0.2					
O-16	Black Card Stock, 1.5" x 1.5" x 0.016"					1.3	
Total Weight of Organic Materials (g)		46.7	32.8	26.6	18.4	5.8	37.9
Percent Weight of Organic Materials (%)		34%	8%	42%	17%	3%	62%
Number	Inorganic Materials	Weight of Items (g)					
I-1	Aluminum Alloy 2024 Sheet Coupons, 1.5" x 1.5" x 0.022"	6.4	6.4	6.4	6.4	6.4	6.4
I-2	Brass Coupling, 1/4" NPT		32.2			32.2	
I-3	Brass Square Head Plug, 1/4" NPT		17.5			17.5	
I-4	1/4-20 Stainless Steel Nuts	31.9			31.9		
I-5	1/4-20 x 1/2" Long Hex Head Stainless Steel Machine Screws	52.2			52.2		
I-6	1/4" NPT Stainless Steel Street Elbow		51.1			51.1	
I-7	Stainless Steel Reducing Bushing, 3/8" MNPT to 1/4" FNPT		14.9			14.9	
I-8	Eyeglass Cleaner in Aluminum Aerosol Can, 1" Diameter x 4.5"			14.5			14.5
I-9	(Tin Paint) Can, 1/4 pint capacity, 2-1/16" H x 2-1/8" Diameter		40.5			40.5	
I-10	Alcohol Wipe Packaging (mainly Al), 1.5" x 3.0" x 0.0035"			0.4			0.4
I-11	Thermometer, Mercury in Glass, 1/4" Diameter x 6" Long			13.0			
I-12	Glass Ampoule, Sealed, 3/8" Diameter x 2" Long			1.6			1.6
I-13	Vermiculite Medium Texture Bulk Sorbent						
I-14	Corrugated HEPA Filter Medium (Boron Silicate with Waterproofing Binder) Coupons, 1.5" x 1.5" x 0.050"			0.5			0.5
Total Weight of Inorganic Materials (g)		90.5	162.6	36.4	90.5	162.6	23.3
Percent Weight of Inorganic Materials (%)		66%	92%	58%	83%	97%	38%
Total Weight of All Materials (g)		137.1	176.1	62.9	108.9	168.4	61.7

Table 4.6.1-4 Samples Sent to MITNRL for NAA

	SAMPLE	BLANK	CONTAMINATED	PROCESSED BY PROTOCOL 1	PROCESSED BY PROTOCOL 2	PROCESSED BY PROTOCOL 2A
List 1	Nut	1	1	2	2	
	Wood	1	1	2	2	
	Lead Lined Rubber	1	1	2	2	
	Black Polyethylene	1	1	2	2	
	Aluminum	1	1	2	2	
List 2	Electrical Tape	1	1	2	2	
	Lined Neoprene	1	1	2	2	
	Lead Lined Rubber			2	2	
	Black Polyethylene			2	2	
	Aluminum			2	2	
List 3	IPA Towel	1	1	2	2	
	Towel Pack	1	1	2	2	
	HEPA Filter	1	1	2	2	
	Black Polyethylene			2	2	
	Aluminum			2	2	
List 4	Nut		1			2
	Wood		1			2
	Lead Lined Rubber		1			2
	Black Polyethylene		1			2
	Aluminum		1			2
	Blue Tarp	1	1			2
List 5	Electrical Tape		1			2
	Black Polyethylene					2
	Aluminum					2
	Rubber Apron	1	1			2
	Black Card Stock	1	1			2
List 6	IPA Towel		1			2
	Towel Pack		1			2
	HEPA Filter		1			2
	Black Polyethylene					2
	Aluminum					2
	Lead Lined Rubber					2

The various compositions of surrogate waste materials were tested with the three decontamination protocols listed in Tables 4.6.1-5, 4.6.1-6, and 4.6.1-7

Table 4.6.1-5 Protocol 1

Protocol 1			
Step No	Step	Step Time	Total Process
		min	Time, min
0	Load Basket in Chamber	5.00	5.00
1	Fill to 0.06 psi	1.17	6.17
2	Spray Part a	1.00	7.17
	Spray part b	1.00	8.17
3	Fill basket to 0.21 psig	2.00	10.17
4	Sonicate @ 40 kHz	10.00	20.17
	Sonicate @ 72 kHz	2.50	22.67
	Sonicate @ 104 kHz	12.50	35.17
	Sonicate @ 170 kHz	2.50	37.67
5	Circulate a	10.00	47.67
	Circulate b	1.00	48.67
6	Drain	2.67	51.34
7	Fill with rinse	4.17	55.50
8	Drain	1.50	57.00
9	Drip and Unload	2.00	59.00

Table 4.6.1-6 Protocol 2

PROTOCOL 2			
Step No	Step	Step Time	Total Process
		min	Time, min
0	Load Basket in Chamber	5.00	5.00
1	Fill to 0.06 psi	1.17	6.17
2	Spray Part a	1.00	7.17
	Spray part b	1.00	8.17
3	Fill basket to 0.21 psig	2.00	10.17
4	Sonicate @ 40 kHz	5.00	15.17
	Sonicate @ 72 kHz	0.00	15.17
	Sonicate @ 104 kHz	5.00	20.17
	Sonicate @ 170 kHz	0.00	20.17
5	Circulate a	0.00	20.17
	Circulate b	0.00	20.17
6	Drain	2.67	22.84
3	Fill basket to 0.21 psig	2.00	24.84
4	Sonicate @ 40 kHz	5.00	29.84
	Sonicate @ 72 kHz	0.50	30.34
	Sonicate @ 104 kHz	10.00	40.34
	Sonicate @ 170 kHz	0.50	40.84
5	Circulate a	8.00	48.84
	Circulate b	0.83	49.67
6	Drain	2.67	52.34
7	Fill with rinse	4.17	56.50
8	Drain	1.50	58.00
9	Drip and Unload	2.00	60.00

Table 4.6.1-7 Protocol 2a

PROTOCOL 2A			
Step No	Step	Step Time	Total Process
		min	Time, min
0	Load basket in chamber	3.00	3.00
1	Fill to 0.06 psi	1.17	4.17
2	Spray part a	0.50	4.67
	Spray part b	0.50	5.17
3	Fill basket to 0.21 psig	2.00	7.17
4	Sonicate @ 40 kHz	5.00	12.17
	Sonicate @ 72 kHz	0.50	12.67
	Sonicate @ 104 kHz	7.50	20.17
	Sonicate @ 170 kHz	0.50	20.67
5	Circulate a	3.00	23.67
	Circulate b	1.00	24.67
6	Drain	2.75	27.42
	<i>Return to Step 1</i>		
1	Fill to 0.06 psi	1.17	28.58
2	Spray part a	0.50	29.08
	Spray part b	0.50	29.58
3	Fill basket to 0.21 psig	3.17	32.75
4	Sonicate @ 40 kHz	5.00	37.75
	Sonicate @ 72 kHz	0.50	38.25
	Sonicate @ 104 kHz	7.50	45.75
	Sonicate @ 170 kHz	0.50	46.25
5	Circulate a	3.00	49.25
	Circulate b	1.00	50.25
6	Drain	2.75	53.00
	<i>Final Rinse</i>		
7	Fill with rinse	4.00	57.00
8	Drain	1.25	58.25
9	Drip and unload	2.00	60.25

4.6.2 Plastic and Aluminum Results Based on Photodigital Analysis

DPA results were obtained for plastic and aluminum coupons. These data are presented in Tables 4.6.2-1 and 4.6.2-2, respectively. The DPA assay measures removal of contamination from flat surfaces only. Three-dimensional samples are not suitable for assay by DPA. This factor may introduce bias into estimates of removal of contamination from surfaces. These biases could result in overestimating the DF when porous surfaces, cracks or crevices are present. The DPA might also underestimate DFs for three-dimensional items because the flat organic test samples might clump together in the cleaning chamber, whereas actual waste materials tend to tumble freely. The nature of the DPA process, however, is well suited to parametric and quality control testing because the parametric and quality control results are referenced to other DPA results for consistency.

The reported DFs are based exclusively on NAA analysis. DPA was used to set up the system to ensure that expensive NAA analyses would yield useful data. In addition, the NAA results are based exclusively on activation of cerium oxide powder, which is a chemical analogue of plutonium oxide. The DPA phosphor is not suitable for NAA testing because the phosphor is not chemically similar to plutonium oxide and because the only element suitable for NAA, zinc, is present as a trace element many of the surrogate waste materials. The zinc that is naturally present in the surrogate materials produces a large and variable background signal that confounds attempts to measure the gamma emissions from the applied contaminant.

Table 4.6.2-1. Average DFs for Plastic DPA Runs

Protocol	Blank GPL/A	Contaminated GPL/A	Clean GPL/A	DPA Decon Factor
Protocol 1, 3 Runs	95	405	106	28
Protocol 2, 3 Runs	95	359	99	72
Protocol 2a, 3 Runs	89	503	92	127

Table 4.6.2-2. Average DFs of Aluminum DPA Runs

List - Protocol	Blank GPL/A	Contaminated GPL/A	Clean GPL/A	Estimated Decon Factor
Protocol 1 3 Runs	113	805	132	36
Protocol 2 3 Runs	110	707	112	142
Protocol 2a 3 Runs	158	915	151	182

* Those results marked with asterisks used the propagated standard deviation of the blank and clean coupons as the denominator in the estimated decon factor equation.

In each of these DPA runs, six (6) plastic and three (3) aluminum coupons were processed and analyzed. On average, higher decontamination factors were obtained with Protocol 2a than with Protocol 1 or 2. This may be attributed to the intermediate drain introduced between sonication cycles. In protocol 2a, the final cleaning occurs with less contamination in the processing liquid.

Figures 12 to 13 provide representative images of selected blank, contaminated, and clean items from the cold tests for qualitative evaluation. The procedure for obtaining quantitative results is described in Appendix B. DF values are provided only for plastic and aluminum due to the limitations of the DPA process.

Blank **Contaminated** **Clean** **Aluminum Coupons** **Black Polyethylene (Plastic) Coupons** **Black Cardstock***Figure 12. Images of Items Processed in Cold Tests - 1*

Figure 13. Images of Items Processed in Cold Tests - 2

Figure 14. Images of Items Processed in Cold Tests – 3

As Received **Contaminated** **Clean** **Brass Coupling** **Stainless Steel Elbow** **Stainless Steel Bushing** **Brass Square Nut***Figure 15. Images of Items Processed in Cold Tests – 4*

4.6.3 Results Based on Neutron Activation Analysis

The results based on neutron activation analysis are presented in Tables 4.6.3-1 to -4.

The NAA results show that substrates with smooth surfaces (aluminum, black polyethylene, blue tarp) can be effectively decontaminated with decon factors greater than 100. These results, in general, substantiate the results obtained by PDA.

Somewhat lower decontamination factors were obtained for both lead-lined rubber and rubber apron material. For these rubber goods, decon factors ranged from 19 to 39. These decon factors may be lower than those obtained with other smooth substrates because rubber is a very good mechanical damper, so that the cavitation induced agitation from ultrasonic waves has less amplitude, thereby retarding the rate of particle removal.

The wood, cardboard, HEPA filter material, and towel/wipe results reported in Table 4.6.3-4 exhibit DFs ranging from 3.5 to 14. These materials are porous. The poorer decontamination observed is attributed to entrapment of contaminant particles in the pore structure. It is likely that most of the contaminant particles are removed from the test piece surfaces within the pores, but because the convective flow rate to the bulk fluid in the tank is very low during the short ultrasonic agitation, only a small fraction of the particles originally present in the pores will be able to escape into the bulk liquid during the decontamination period. In this case, higher decon values will be obtained if the duration of the ultrasonic agitation is increased.

The summary results for individual materials, presented as an average of Protocol 2 and Protocol 2A data, are as follows:

<i>Surrogate Material</i>	<i>DF</i>	<i>Removal</i>
<i>Black polyethylene</i>	<i>284</i>	<i>99.7%</i>
<i>Alcohol towel/wipe</i>	<i>14</i>	<i>93%</i>
<i>Alcohol wipe package</i>	<i>308</i>	<i>99.7%</i>
<i>Lead lined rubber</i>	<i>39</i>	<i>97%</i>
<i>Aluminum</i>	<i>369</i>	<i>99.7%</i>
<i>HEPA filter material</i>	<i>3.5</i>	<i>78%</i>
<i>Black cardboard</i>	<i>6.5</i>	<i>87%</i>
<i>Wood</i>	<i>11</i>	<i>92%</i>
<i>Aged blue poly tarp</i>	<i>161</i>	<i>99.4%</i>
<i>Irradiated lead lined rubber</i>	<i>6.0</i>	<i>86%</i>

Widely variable results were obtained with electrical tape, with DFs ranging from less than 2 to more than 200. Inconsistent DFs are attributed to the adhesion of the contaminant particles to

the adhesive of the fresh tape, with high DFs being obtained when the contaminant particles were deposited on the nonadhesive side of the tape. The effect of aging and embrittlement of the tape was not examined. However, it is anticipated that higher DFs would be obtained with aged tape than with fresh tape.

Lined neoprene glove material was the only material tested that was not effectively decontaminated. For this material, a decon factor of only 2 or 3 was obtained. These results are believed to be due to the unusual structure and geometry of the glove material. The lining is a fine hair like brush. The geometry of the fingertips prevents liquid flow. The compliance of the neoprene shell results in adsorption of the applied ultrasonic energy. This combination of geometry and structure evidently results in a very effective particle trap.

Applying the above NAA DFs to the mixtures used in integrated cold testing, the following weighted DFs are calculated for these six mixtures. Figure 16 shows details of the calculation.

MIXTURE	ORGANIC WEIGHTED DF	INORGANIC WEIGHTED DF	TOTAL WEIGHTED DF
1	140	370	290
2	34	370	310
3	270	360	330
4	45	370	310
5	63	370	360
6	200	360	260

For the representative surrogate waste profile and the two bounding cases (Table 4.1.2-3) developed by SRS, the following overall DFs can be calculated:

SURROGATE WASTE PROFILE	ORGANIC WEIGHTED DF	INORGANIC WEIGHTED DF	TOTAL WEIGHTED DF
Representative	150	220	162
Bounding Case A	65	370	156
Bounding Case B	168	64	137

Figure 16

Fig. 16 Weighted DF of Mixtures Used in Integrated Cold Tests

Material Type	Mixture Number						Mixture Number					
	1	2	3	4	5	6	1	2	3	4	5	6
	Composition of Mixture - Organics						Mass Weighted DF of Mixture Component - Organics					
	Weight (grams)						Weighted DF (grams)					
Black poly sheet	1.1	1.1	1.1	1.1	1.1	1.1	3.1E+02	3.1E+02	3.1E+02	3.1E+02	3.1E+02	3.1E+02
Aged blue tarp						0.5	161					
Clear poly sheet	0.4						284	1.1E+02				
Pb lined rubber	11.3	11.3		11.3		11.3	39	4.4E+02	4.4E+02	4.4E+02		4.4E+02
Rubber o-ring	0.9			0.9			19	1.7E+01		1.7E+01		
Neoprene		19.3					19	3.7E+02				
Liatex	8.7						19	1.7E+02				
Nitrile					2.3		19				4.4E+01	
Electrical tape		1.1			1.1		3	3.8E+00			3.8E+00	
Black poly pipe			24.5			24.5	284		7.0E+03			7.0E+03
PVC hardw are	19.6						284	5.6E+03				
Wood block	4.5			4.5			11	5.0E+01		5.0E+01		
Wood stick				0.6			11			6.6E+00		
IPA tow elette			1			1	14		1.4E+01			1.4E+01
Paper napkin	0.2						6	1.3E+00				
Cardboard					1.3		6				8.4E+00	
ORG. WT. (grams)	46.7	32.8	26.6	18.4	5.8	38.4	Org. DF	1.4E+02	3.4E+01	2.7E+02	4.5E+01	6.3E+01
ORG. WT. (%)	34%	17%	42%	17%	3%	62%						
Material	Mixture Number						Mixture Number					
	1	2	3	4	5	6	1	2	3	4	5	6
	Composition of Inorganics						Mass Weighted DF of Mixture Component - Inorganics					
	Weight (grams)						Weighted DF (grams)					
Al sheet coupons	6.4	6.4	6.4	6.4	6.4	6.4	369	2.4E+03	2.4E+03	2.4E+03	2.4E+03	2.4E+03
Brass Coupling		32.2			32.2		369		1.2E+04		1.2E+04	
Brass plug		17.5			17.5		369		6.5E+03		6.5E+03	
SS nuts	31.9			31.9			369	1.2E+04		1.2E+04		
SS screws	52.2			52.2			369	1.9E+04		1.9E+04		
SS elbow		51.1			51.1		369		1.9E+04		1.9E+04	
SS bushing		14.9			14.9		369	5.5E+03		5.5E+03		
Al aerosol can			14.5			14.5	369		5.3E+03		5.3E+03	
Tin can		40.5			40.5		369		1.5E+04		1.5E+04	
IPA packaging (Al foil)			0.4			0.4	369		1.5E+02		1.5E+02	
Glass thermometer			13				369		4.8E+03			
Glass ampoule, sealed			1.6			1.6	369		5.9E+02			5.9E+02
Vermiculite							369					
HEPA filter			0.5			0.5	3		1.7E+00			1.7E+00
INORG. WT. (grams)	90.5	163	36.4	90.5	163	23.4	Inorg. DF	3.7E+02	3.7E+02	3.6E+02	3.7E+02	3.6E+02
INORG. WT. (%)	66%	83%	58%	83%	97%	38%						
TOTAL WT. (g)	137	195	63	109	168	61.8	Total / DF	2.9E+02	3.1E+02	3.3E+02	3.1E+02	2.6E+02

Table 4.6.3-2 Aluminum NAA Results

Aluminum	Cerium
Blank 1	1372
Blank 2	1521
Blank 3	1375
Blank 4	1302
Average Blank	1393
Standard Deviation of Blank	92
Contaminated (List 1 - 3)	270127
Contaminated (List 1 - 3)	273311
Contaminated (List 1 - 3)	295728
Contaminated (List 1 - 3)	283098
Average (List 1 - 3) Contaminated	280566
Standard Deviation of Contaminated	11517
Contaminated (List 4 - 6)	449003
Contaminated (List 4 - 6)	384943
Average (List 4 - 6) Contaminated	416973
Standard Deviation of Contaminated	45297
Average (All) Contaminated	326035

Aluminum				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
1	1	1	1845	617
1	1	2	2680	217
1	1	Average	2263	321
2	1	1	2126	381
2	1	2	1679	974
2	1	Average	1903	547
3	1	1	1750	781
3	1	2	1730	827
3	1	Average	1740	803
All	1	Average	1968	485
1	2	1	2342	294
1	2	2	2873	189
1	2	Average	2608	230
2	2	1	2734	208
2	2	2	2420	272
2	2	Average	2577	236
3	2	1	2564	238
3	2	2	1791	701
3	2	Average	2178	356
All	2	Average	2454	263
4	2a	1	2408	409
4	2a	2	2716	314
4	2a	Average	2562	355
5	2a	1	2538	363
5	2a	2	1896	825
5	2a	Average	2217	504
6	2a	1	1530	3022
6	2a	2	1472	5227
6	2a	Average	1501	3830
All	2a	Average	2093	593
All	2 & 2a	Average	2274	369
All	All	Average	2172	417

Table 4.6.3-1 Polyethylene NAA Results

Black Polyethylene	Cerium
Blank 1	217
Blank 2	215
Blank 3	195
Blank 4	242
Average Blank	217.25
	Cerium
Contaminated (List 1-3)	211080
Contaminated (List 1-3)	212715
Contaminated (List 1-3)	233552
Contaminated (List 1-3)	212281
Average (List 1-3)	
Contaminated	217407
Contaminated (List 4 - 6)	209902
Average (All)	
Contaminated	215906

Black Polyethylene				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
1	1	1	570	616
2	1	1	712	439
2	1	2	1808	137
2	1	Average	1260	208
3	1	1	2803	84
3	1	2	872	332
3	1	Average	1838	134
All	1	Average	1353	191
1	2	1	3831	60
1	2	2	338	1799
1	2	3	1450	176
1	2	Average	1873	131
2	2	1	400	1188
2	2	2	671	479
2	2	3	2654	89
2	2	Average	1242	212
3	2	1	223	37772
3	2	2	267	4366
3	2	3	891	322
3	2	4	152	-3329
3	2	Average	383	1308
All	2	Average	1088	250
4	2a	1	1479	166
5	2a	1	416	1055
5	2a	2	1258	201
5	2a	Average	837	338
6	2a	1	400	1147
6	2a	2	220	76249
6	2a	Average	310	2261
All	2a	Average	755	390
All	2&2a	Average	977	284
All	All	Average	1071	253

Table 4.6.3-3 Lead Lined Rubber

Lead-Lined Rubber	Cerium
Blank 1	18365
Blank 2	19233
Average of Blank	18799
Standard Deviation of Blank	614
Contaminated (List 1-2)	154459
Contaminated (List 1-2)	173943
Average (List 1- 2)	
Contaminated	164201
Standard Deviation of Contaminated	13777
Contaminated (List 4, 6)	209902
Average (All)	
Contaminated	179435

Lead-Lined Rubber				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
1	1	1	23497	31
1	1	2	24460	26
1	1	Average	23979	28
2	1	1	23122	34
2	1	2	23026	34
2	1	Average	23074	34
All	1	Average	23526	31
1	2	1	23854	29
1	2	2	20949	68
1	2	Average	22402	40
2	2	1	21669	51
2	2	2	21466	55
2	2	Average	21568	53
All	2	Average	21985	46
4	2a	1	21970	60
4	2a	2	22263	55
4	2a	Average	22116.5	58
6	2a	1	28332	20
6	2a	2	26823	24
6	2a	Average	27578	22
All	2a	Average	24847	32
All	2&2a	Average	23416	39
All	All	Average	23453	35

Table 4.6.3-4 Other Substrates

IPA Towelette	Cerium
Blank 1	144
Contaminated (List 3)	216341
Contaminated (List 3)	214111
Average (List 3)	
Contaminated	215226
Contaminated (List 6)	480453
Average (All)	
Contaminated	303635

IPA Towelette				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
3	1	1	16983	13
3	1	2	27245	8
3	1	Average	22114	10
3	2	1	9317	23
3	2	2	16211	13
3	2	Average	12764	17
6	2a	1	33510	14
6	2a	2	29533	16
6	2a	Average	31521.5	15
All	2&2a	Average	22143	14
All	All	Average	22133	14

Electrical Tape	Cerium
Blank 1	4405
Contaminated (List 2)	250869
Contaminated (List 5)	411238
Contaminated (All)	
Average	331053

Electrical Tape				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
2	1	1	2.16E+05	1
2	1	2	1.78E+05	1
2	1	Average	1.97E+05	1
2	2	1	3.69E+04	8
2	2	2	3.07E+04	9
2	2	Average	3.38E+04	8
5	2a	1	5.52E+03	364
5	2a	2	6.82E+03	168
5	2a	Average	6.17E+03	230
All	2&2a	Average	N/A	N/A
All	All	Average	79053	4

IPA Towel Package	Cerium
Blank 1	1573
Contaminated (List 3)	205513
Contaminated (List 6)	775555
Average (All) Contaminated	490534

IPA Towel Package				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
3	1	1	1645	2833
3	1	2	2093	392
3	1	Average	1869	689
3	2	1	1915	596
3	2	2	2760	172
3	2	Average	2338	267
6	2a	1	4173	298
6	2a	2	3787	350
6	2a	Average	3980	322
All	2&2a	Average	3159	308
All	All	Average	2729	423

HEPA Filter	Cerium
Blank 1	205
Contaminated (List 3)	404234
Contaminated (List 6)	816057
Average (All)	
Contaminated	610146

HEPA Filter				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
3	1	1	109517	4
3	1	2	103601	4
3	1	Average	106559	4
3	2	1	81628	5
3	2	2	96238	4
3	2	Average	88933	5
6	2a	1	267996	3
6	2a	2	256084	3
6	2a	Average	262040	3
All	2&2a	Average	175487	4
All	All	Average	152511	4

Black Cardstock	Cerium
Blank 1	3310
Contaminated (List 5)	641194

Black Cardstock				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
5	2a	1	78324	9
5	2a	2	125274	5
5	2a	Average	101799	6

Rubber Apron	Cerium
Blank 1	13463
Contaminated (List 5)	459072

Rubber Apron				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
5	2a	1	40174	17
5	2a	2	33686	22
5	2a	Average	36930	19

Lined Neoprene	Cerium
Blank 1	436
Blank 2	562
Average Blank (1-2)	499
Contaminated (List 2)	246012
Contaminated (List 2)	211296
Average (All)	
Contaminated	228654

Lined Neoprene				
List	Protocol	Sample	Cerium	Decon Factor Cerium
2	1	1	104926	2
2	1	2	97709	2
2	1	Average	101318	2
2	2	1	91481	3
2	2	2	88141	3
2	2	Average	89811	3
2	All	Average	95564	2

Wood	Cerium
Blank 1	58
Blank 2	69
Average Blank	63.5
Contaminated (List 5)	501746

Wood				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
5	2a	1	42635	12
5	2a	2	48180	10
5	2a	Average	45408	11

Blue Tarp	Cerium
Blank 1	123
Contaminated (List 4)	353933

Blue Tarp				
Mixture	Protocol	Sample	Cerium	Decon Factor Cerium
4	2a	1	1560	246
4	2a	2	3072	120
4	2a	Average	2316	161

5.0 DECONTAMINATION OF IRRADIATED SAMPLES

In order to address the capability of the Sonatol® process to remove Pu-238 particles embedded in plastic and or rubber substrates, a small number of 1 cm diameter lead lined rubber coupons were first contaminated with cerium oxide and phosphor powders, and then subjected to a radiation dose in excess of 110 Mrad in the MIT Nuclear Reactor. These contaminated samples were then assayed by neutron activation analysis, subjected to a decontamination cycle, and assayed again by neutron activation analysis. Since samples were radioactive, the decontamination experiment was performed at the MITNRL. The vials were suspended in a 5-gallon Crest 132 kHz ultrasonic bath operated at 500 watts. The test coupons were subjected to the following decontamination cycle:

1. Immersion and sonication for 7.5 minutes in 20 ml of Sonatol A 70/ 0.4-% Krytox 157FSL solution in a 40 ml glass vial.
2. Transfer of the coupon to a new 40 ml vial that contained 20 ml of fresh Sonatol A 70/ 0.4-% Krytox 157FSL solution, and sonication for an additional 7.5 minutes.
3. Transfer of the coupon to a new 40 ml vial that contained 20 ml of fresh Sonatol A 70 and sonication for 3.5 minutes to rinse.

The results of these experiments are presented in Table 4.6.4-1. Approximately 80% of the deposited cerium oxide was removed. This corresponds to a decontamination factor of about 5. The removal of cerium from irradiated lead lined samples was significantly lower than for unirradiated samples (Table 4.6.3-3), showing that the cerium particles may be somewhat embedded in the irradiated rubber. Further tests would be necessary to determine if increasing the ultrasonic power or adding suitable agents to the bath can increase the DF. Nevertheless, an 86% removal rate is a positive result, especially given the extremely high radiation dose (>110 Mrad).

Table 4.6.4-1 Irradiated Lead Lined Rubber Results

Irradiated Lead Lined Rubber				
Mixture	Cerium Blank	Contaminated	Clean	Decon Factor
1	24999	266829	73711	5
2	22132	219038	73244	4
3	30310	212385	69426	5
4	26047	216457	49969	8
Average	25872	228677	66588	5

6.0 SYSTEM SPECIFICATIONS ---

6.1 MATERIALS LIST

The only materials used in the process are the perfluorinated Sonatol solvent and the fluorinated surfactant. MSDSs are provided in the Appendices.

6.2 HARDWARE COMPONENTS

A bill of materials is provided in Appendix H.

6.3 OPERATING PROCEDURES

6.3.1 PanelView Startup Procedure

A PanelView startup procedure is provided in Appendix J.

6.3.2 System Start-up

A system startup procedure is provided in Appendix K

6.3.3 System Operations

An operations checklist and operating steps are provided in Appendix L

6.3.4.1 ACF Housing Replacement

After 6 complete runs through the Activated Carbon Filter with 0.4% surfactant solution, it is advisable that the filter be replaced before breakthrough of surfactant occurs. To replace the filter, the ACF housing must be removed. First, break Con 7 and 13. Then connect Con 12 to the Con 12a drain tube and open MV-2 in order to drain the contents of the ACF housing into the cleaning chamber. After it has completely drained, remove the threaded rod legs. Then break the 1/4" swage elbow on the right of the ACF top. Now tilt the ACF housing towards the glove box window. This will allow enough room to break the 3/4" SS swage nut that connects the elbow at the top of the ACF to the mandrel. Now the ACF Housing can be removed and replaced with the new Housing. After the new ACF housing is in place and all the connections have been made it is ready to be filled with liquid (see 3.6.2).

6.3.2 Replacement of Filter F-2

When the pressure differential between pressure sensors P-5 and P-3 reaches 15 psig the system will stop operating. At this point, filter F-2 should be replaced. This is accomplished by breaking quick disconnect fittings Con 2, 3, 4, and 10, and removing the F-2 assembly from its bracket. The assembly should be held over the (preferably empty) cleaning chamber so that when the Swagelok end fittings are loosened, the liquid hold-up in the filter drains in the chamber. The drained filter is capped by screwing on $\frac{3}{4}$ " Swagelok caps. The used filter capsule can now be removed safely from the glove box. It is replaced with a new filter capsule, which is installed by reversing the above steps

6.3.3 Replacement of F-3 Filter Assembly

If the F-3 filters become clogged they must be removed from the system and replaced. While it is physically possible to replace the individual filter modules, BSI recommends replacing the whole F-3 assembly because of concerns about the relatively high level of Pu-238 contaminants that will have collected on these modules.

First, break Con 4 and Con 5 connections to isolate the F-3 apparatus. Then, with a wrench, break the $\frac{1}{2}$ " stainless steel (SS) swage nuts after the two F-3s join at the downstream end. Some liquid will escape until it is capped with a $\frac{1}{2}$ " SS swage nut plug.

The next step is to remove the T-1 support leg (the left one) that is in front of the assembly. First, back the top nuts off the threaded rod. *If T-1 has been drained*, lift the T-1 platform with one hand while removing the leg with the other. Otherwise, move the lower connecting nut down the threaded rod and then lift the leg up. With one hand against tank T-1 (to prevent it from tipping), move the filter into the open workspace in front of the left-hand gloves. Replace the support leg under the tank.

Turn the apparatus upside down so that the legs are sticking straight up. Then break the $\frac{1}{2}$ " SS swage nut between the filters and the AV-2 Solenoid valve and quickly cap the filters with a $\frac{1}{2}$ " SS swage plug. The F-3 filters are now sealed and can be removed from the system. Now the new F-3 apparatus can be brought into the glove box and the swage connections made. First connect the line between the Solenoid valve (AV-2) and the underside of the apparatus. Then, temporarily remove the T-1 support leg and slide the F-3 assembly into position. Replace the threaded rod leg for T-1. Make the connection with line 4 and reconnect Con 4 and 5. The F-3 filter assembly is now operational.

6.3.4 Operating Maintenance

The major maintenance procedures are the replacement of the activated carbon column when it becomes saturated with Krytox 157FS surfactant or other polar contaminants that are soluble in Sonatol® 70, and the replacement of filters F-2 and F-3.

Table 6.3.4-1 is a list of the spare parts and consumables provided with the system.

Table 6.3.4-1 Spare Parts and Consumables Provided with System

Equipment	Part Description	Part No	Supplier	Number	Units
Spares Parts					
Item No					
D-1	ACF Adsorption Chamber		ESI	1	each
F-4	Millipak 200	MPL20CB3	Millipore	1	each
J-1	Impeller, Turned Down	MD0521-trimmed	Maltz Sales	1	each
J-1	EPDM O-ring	MD-049	Maltz Sales	2	each
J-1	Front Casing	MD0599	Maltz Sales	1	each
J-2	Impeller for WMD 30	MD0062	Maltz Sales	1	each
J-2	EPDM O-ring	325700008	Maltz Sales	2	each
J-2	Front Casing	MD0313	Maltz Sales	1	each
TM-1	Turbidity Meter	2003	HF Scientific	1	each
	Cylindrical Baskets		Leo Eng.	5	each
	Polyethylene Utility Jug, 1 gal, 4/pk	4266T61	McMaster Carr	3	pack
Consumables					
	Polyethylene Utility Jug, 1 gal, 4/pk	4266T61	McMaster Carr	8	pack
	Sonatol A70 Process Liquid (25 gal)	5 each 5 gal pails	ESI	350	lbs
	Krytox 157FSL Surfactant		DuPont	2	lbs

6.3.5 Decommissioning

The decommissioning checklist is as follows:

- Run the system empty in the automatic mode for at least three cycles.
- After the completion of the last cycle, break Con 9.
- Attach drain line 9a to the female part of Con 9.
- Now operate the system in manual mode
- Drain contents of T-1 through D-1 (by opening AV-3 and turning on J-2) into drain hole or floor of glovebox. Passing liquid through D-1 minimizes deposition of nonvolatile surfactant on the floor of the glove box. Sonatol A 70 will evaporate from floor of glove box.
- Drain D-1 by connecting drain tube 12a to Con 12 and opening MV-2.
- Disconnect D-1 as described in 3.8.1

- h. Disconnect and cap Filter F-2 as described in 3.8.2
- i. Disconnect and cap Filter F-3 Assembly as described in 3.8.3
- j. Disconnect all signal and power connections.
- k. Disconnect all remaining quick disconnect fittings.
- l. Remove equipment from glove box in the reverse of the order of installation.

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8.0 APPENDICES

APPENDIX A FILTRATION TESTS*November 21, 2001*

To: R. Kaiser

From: J. Antkowiak

SUBJECT: J177 - TESTING OF CERAMEM™ FILTERS FOR REMOVAL OF SUSPENDED PARTICLES**INTRODUCTION**

The goal of this project is to remove Plutonium-238 (Pu-238) particles from transuranic mixed waste by agitating the waste in a fluorinated surfactant solution, and then passing this liquid through 0.2 µm ceramic filters to remove the suspended Pu-238. ESI investigated the use of Ceramem™ multichannel, alumina coated, cross-flow filters with a 0.2 µm nominal pore size for the removal step by challenging them with Cerium (IV) Oxide and Silica powder suspensions as Pu-238 simulants

The purpose of the testing was to establish:

- a. Pressure drop as a function of clean liquid flow rate.
- b. Particle retention and capacity of a 0.2 µm cordierite filter challenged with well dispersed suspensions of Cerium (IV) Oxide in a 0.3 wt-% solution of a fluorinated surfactant, Krytox 157 FSL [DuPont], in either perfluorohexane (3M's PF-5060) or perfluoroheptane (3M's PF-5070), and
- c. Particle retention of 0.2 µm cordierite and silicon carbide filters challenged with well dispersed suspensions of closely sized 0.2 µm silica particles in a 0.3 wt-% solution of a fluorinated surfactant, Krytox 157 FSL [DuPont], in perfluorohexane (3M's PF-5060).

EXPERIMENTAL**System Description**

For the present pressure-drop and capacity test, the filter was placed horizontally with the outlet (retentate) stream valved off, so that the filter was operated in a dead-end mode. A suspension of cerium oxide in fluorinated surfactant solution is pumped through the filter and the filtrate is collected beneath the filter from the permeate channel. The filter has a surface area of 1.2 ft². A schematic of the filter system is shown in *Figure 1*.

Figure 1 - Schematic of Ceramem™ Filter System

A Model 132 Micropump® drives the suspension through the filter. Valve V1 on the rotameter controls the flow rate through the system. A picture of the system is shown as Figure 2.

FIGURE 2 - FILTER SYSTEM

FILTER CHALLENGES

ESI examined two different filter challenges:

- Cerium (IV) oxide (CeO_2) [Alfa Aesar, Stock# 39802]. The cerium oxide has a listed particle size of less than $0.3\ \mu\text{m}$.¹ *Figure 3* is an SEM photo of the dry cerium oxide particles. This powder is polydisperse, with individual particles ranging in size from 0.1 to $1\ \mu\text{m}$. The individual cerium oxide particles also are not uniform in shape. *Figure 5* is a photomicrograph (light microscope) of a cerium oxide suspension in 5070. This photomicrograph highlights agglomerates rather than individual particles.
- Silicon oxide. (SiO_2) [Geltec Inc., Product Code S0201, 0.2 micron]. The claimed particle size for the SiO_2 powder is $0.2\ \mu\text{m}$. *Figure 4* is an SEM photo of the dry silicon oxide particles. This powder consists of monodisperse spheres, with individual particles being nearly all $0.2\ \mu\text{m}$ in diameter.

SUSPENSIONS

The suspensions of cerium oxide or silica oxide that were used to challenge the filters were prepared as follows:

- The cerium oxide suspensions were prepared by weighing out 0.5 to 3 grams of cerium oxide (depending on the feed particle concentration desired) and adding 100 ml of fluorinated surfactant solution. The jar was shaken and then sonicated for 20 minutes. 50 ml of this suspension was diluted to 4 liters of feed suspension.
- The silica suspensions were made in a similar fashion. 1.25 g of silica powder was ground with a mortar and pestle to eliminate large agglomerates. The ground silica powder was added to 100 ml of fluorinated surfactant solution. Upon sonication, larger agglomerates, not in suspension, floated to the top and were removed. Those particles were ground again and added back to the 100 ml suspension. Any particles on the surface during sonication were removed. The resulting 100 ml suspension was diluted to 4 liters of feed suspension.

PARTICLE CONCENTRATION ANALYSIS

The particle concentration in the challenge suspensions and in the filtrates was determined by measuring the turbidity of these liquids. Turbidity is a standard means of establishing drinking water quality, since the turbidity of a liquid suspension is due to the scattering of light

by the suspended particles (1,2). For a given suspended powder in a given liquid, the turbidity of a suspension is directly proportional to the concentration of particles in the suspension.

Turbidity measurements were made with a HF Scientific Micro 100 Turbidimeter. The calibration curves presented in *Figure 6* were obtained by adding known amounts of

Figure 3 – SEM picture of Cerium Oxide

FIGURE 4 – SEM PICTURE OF SILICA

FIGURE 5 - PHOTOMICROGRAPHS OF CERIUM OXIDE PARTICLES (200x) AND CALIBRATION SLIDE

Figure 6 - Particle Concentration in PF-5070 vs Turbidity

either cerium oxide or silica to a given volume well filtered fluorinated surfactant solution. Neat, filtered (through Gelman Acrodisc 0.2 μm membrane filters) fluorinated surfactant solution has a turbidity of 0.46 to 0.49 NTU (National Turbidity Units).

RESULTS AND DISCUSSION

PRESSURE DROP AS A FUNCTION OF LIQUID FLOW RATE

The effect of the flow rate of the neat, filtered fluorinated surfactant solution on the pressure drop across the filters is summarized in Table 1.. A 0.22 μm Millipore Millipak™ 200 GL CB filter was used to prefilter the liquid going to the ceramic filter.

TABLE 1 – FILTER PRESSURE DROP VERSUS FLOW RATE

Cordierite			<u>SILICON CARBIDE</u>		
Rotameter Flow Rate		Pressure Drop	Rotameter Flow Rate		Pressure Drop
Reading	(mL / min)	(psig)	Reading	(mL / min)	(psig)
20	114	3.2	20	114	1.0
40	354	6.1	40	354	2.0
60	594	11.0	60	594	3.0
80	834	15.3	80	834	4.2
87	918	16.8	90	954	5.0

Further testing was performed at a system operating flow rate of 500 mL/min. At this flow rate, the pressure drop across the silicon carbide filter was about 3 psig, which was 8 psi lower than that across the cordierite filter. According to Ceramem, the silicon carbide filter is more recent development than the cordierite filter. The lower pressure drop across the silicon carbide filter is due to improvements in their fabrication process, such as the ability to apply a thinner alumina coating.

FILTER CAPACITY

Initial testing was performed with an alumina-coated cordierite filter that ESI had in stock. This filter quantitatively removed cerium oxide particles suspended in challenge suspensions. The filtrates from the pressure-drop test runs described below had turbidity measurements averaging 0.48. Photographs of a representative feed solution and of a filtrate are presented in *Figures 7 and 8*.

Filter capacity was established by challenging the filter with cerium oxide suspensions and observing the pressure drop increase across the filter as a function of time (or of loading). The suspensions were pumped through the system at 500 mL/min. The cerium oxide filter challenge suspensions ranged in concentration from 0.075 g/L to 0.38 g/L. The concentration of cerium oxide was increased with time as long as no increase in pressure drop was observed. . The test ended when the pressure drop increased 12 psig from the initial, clean-fluid pressure drop (from 11 to 23 psig).

Figure 7 – Challenge Suspension

Figure 8 - Permeate

The pressure drop across the filter as a function of cerium oxide loading is given in *Figure 9*. At this pressure drop, a total of 114.1 g of cerium oxide was filtered by the system, which corresponds to a loading of 95 g/ft² (882 g/m²). A large increase in the pressure drop was observed at a loading of 95 g, which corresponds to a loading of 79 g/ft² (734 g/m²).

After concluding the pressure-drop test, ESI wanted to establish whether or not flushing cerium oxide from the filter would decrease the pressure drop. The filter was first flushed with normal cross-flow through the filter, and then backflushed through the permeate channel. The filter was flushed to determine whether the pressure-drop would decrease with removal of cerium oxide from the filter.

The first flush was performed by capping the filtrate channel, opening the capped retentate channel and pumping clean solvent through the filter. 9.6 g of cerium oxide was recovered in 618 mL of fluorinated surfactant solution. The second flush was performed by reversing the flow of liquid in the system. The

fluorinated surfactant solution was pumped through the filtrate channel and flushed out the retentate outlet channel. The normal input channel was capped. 47.5 g of cerium oxide was flushed out in 800 mL of liquid, suggesting that back flushing is a more efficient method of flushing.

In all, 57.1 g (50.1 % of total) of cerium oxide were recovered from the filter through flushing. Upon challenging the cleaned filter with 0.38 g/L cerium oxide suspension the pressure drop across the filter was 18 psig. In subsequent runs when the filter was challenged with silica suspensions, the pressure drop was observed to drop further to 12 psig.

Figure 9 - Pressure-Drop vs. Cerium Oxide Loading

An additional 30 g of cerium oxide was recovered from the cordierite filter and filter housing when the cordierite filter was removed from the housing. Total cerium oxide recovery was thus 87.1 g, or 76%.

SILICA RETENTION

Because the cerium oxide suspension had a fairly broad particle size distribution, and did not have a high concentration of particles smaller than 0.3 μm (as claimed by the supplier), it was decided to challenge the test filters with suspensions of the nearly monodisperse, 0.2 μm silica powder.

Cordierite Filter

Proceeding the pressure-drop / capacity test, the filter was challenged with silica suspensions had a particle concentration of 0.017 g/L. In these tests, the filtrate was periodically sampled, and the turbidity of these sequential filtrate samples measured. In the first run, turbidity measurements of the filtrate ranged from 1.12 at the start of the run to 0.49 at the end; corresponding to a particle concentration of 5.7×10^{-5} g/L (at the start) to clean liquid at the end. Similar results were observed for a second run. As the filter was challenged with more silica in subsequent runs, the filtrates were not turbid. These results indicate that some silica oxide was initially breaking through the filter, probably due to the presence of flaws in the alumina coating; but, as the filter was challenged with more silica, the flaws in the filter became plugged, which prevented further particle leakage.

SILICON CARBIDE FILTER

After performing the clean liquid pressure drop tests, the silicon carbide filter was challenged with silica suspensions that had a particle concentration of 0.035 g/L. Turbidity measurements were taken on filtrate samples of the initial runs to detect any particle breakthrough. Samples were taken at 1min, 2min 30s, 4min, 5min 30s, and 7min from the start of a run. For all three runs, each sample had a turbidity 0.48 or below, suggesting that the liquid was clean, and that there was no breakthrough of silica particles. A picture of a filtrate sample is shown in *Figure 10* compares the clarity of such a filtrate sample to those of the silica and cerium oxide challenge suspensions.

The silica carbide filter is a newer, and obviously superior Ceramem product than the cordierite filter, in that it exhibits lower pressure drop and better retention.

Figure 10 – Cerium oxide (left), Silica (middle), and filtered fluorinated surfactant solution (right)

CONCLUSIONS

The 0.2 μm Ceramem™ filters effectively removed dispersed sub-micron sized particles (cerium oxide and silica oxide) from a fluorinated surfactant solution, as measured by turbidity.

The silicon carbide filter is superior in performance in that it exhibits lower pressure drop and better retention than the cordierite filter.

The Ceramem™ filters have a high soil loading capacity in a dead-end mode, and can be backflushed, if desired.

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INTEROFFICE MEMORANDUM

TO: DR. KAISER
FROM: JOSHUA JONDRO
SUBJECT: FULL SCALE ACF TESTING
DATE: JUNE 14, 2002

Abstract

This experiment established the effective lifetime of an activated carbon fiber felt adsorption column in which the ends are covered with an impermeable plastic film is able to effectively remove Krytox 157 surfactant from PF-5070. It was shown that the column effectively removed 90% of the Krytox 157 from five-adsorber volumes (40 L) of 0.4% Krytox 157FS solution, pumped at one adsorber volume every hour. Without these end sheets of polyethylene, the effective lifetime is reduced to approximately two adsorber volumes (16 L).

Introduction

The purpose of these experiments was to carry out full scale testing of the activated carbon fiber (ACF) felt adsorption column used in the Sonatol Pu-238 Decontamination System. A second purpose of this experiment was to verify the results of the small scale ACF adsorption on the same scale as will be used in this system. The efficiency of the adsorber design and effective adsorber lifetime are the most important information that is derived from the data collected. The effective lifetime of the filter is defined as the time until the column stops removing greater than 90% of the Krytox pumped.

Experimental

The following equipment is used in this experiment:

- Sonatol Pu-238 Adsorber
- Shimadzu UV-1201 UV-VIS Spectrophotometer
- Thermo Separations Products Spectra 100 Variable Wavelength Detector: UV Range (AUFS = 0.1), Wavelength = 210 nm
- PC equipped with UV.exe to record absorbance data output from wavelength detector
- Micropump Model Gear Pump
- Gilmont 51106 Stainless Steel/Glass Ball Rotameter
- Millipore 90 mm Filter Holder with 5 μ m Filter
- Sonatol Pu-238 Decontamination System Adsorber D-1
- Pressure Gauges 0-15, 0-30, 0-60 psi
- Swagelok Fittings and Valves

ACF Adsorption Column

The first component of the ACF adsorption column is the ACF cloth wrapped mandrel. The mandrel is composed of a section of 1" diameter porous metal pipe welded to two sections of 1" diameter pipe on either end. With one end plugged as seen in Figure 1, and the other end welded to the 0.75", which penetrates the column cover, pipe the mandrel is complete. ACF cloth is wrapped and secured around the porous section as described by J. Jondro (see J. Jondro memo 4/15/02).

Figure 1 Mandrels around which the ACF cloth is wrapped

The ACF adsorption housing is composed of 10" section of 8" schedule 40 aluminum pipe and end plates of 0.5" x 10.5" diameter aluminum plate. As seen in Figure 2, the 0.25" inlet tube penetrates the cover near the edge of the column and brings the fluid to the bottom of the column. The ACF cloth wrapped mandrel penetrates the top plate of the filter at the center. The fluid must flow through the ACF cloth to gain access to the mandrel and exit the adsorber.

Found in Figure 3 is the variation on this adsorption column setup. The difference between these two setups is that in the second there is a impermeable polyethylene sheet covering both ends. This guards against any of the fluid bypassing the ACF cloth. Without this plastic the fluid could flow through the ends of the carbon and pass through only a small portion of the carbon, exhausting the removal capacity of that small portion quickly.

Figure 2 ACF adsorption column

Figure 3 ACF adsorption column equipped with plastic ends

Experimental Setup

Figure 4 and Figure 5 are the process flow diagram (PFD) and a photograph of this experimental setup, respectively. Following is a description of the fluid flow through the experimental setup.

Fluid, drawn from the tank T-1 by the pump J-1, is first pumped through a rotameter F-1, then a throttling valve, V-1, used to control the flow rate of the fluid through the ACF adsorber, and a pressure gauge, PG-1, for pressure drop measurements. On the top of the ACF adsorber are two three-way valves, V-2 and V-3, plumbed so fluid can bypass the adsorber. The adsorber effluent is located in the center of the cover. From here, the fluid flows passed another pressure gauge, PG-2, and through a 90mm diameter, 5 μ m filter, F-1. A final pressure gauge, P-3, allows the operator to determine whether there is a plugged filter and the fluid is divided between flowing into a drum and flowing through the online wavelength detector. A needle valve maintains a flow rate of approximately 1 ml/min through the wavelength detector.

Figure 4 Process flow diagram for the experimental setup

Figure 5 Photograph of experimental setup used during the experiment

Material Preparation & Characterization

The initial step in this experiment was to verify that all fluid used is very clean using the ultraviolet light absorbance as a reference. The Krytox solution is created by adding 0.4 wt-% Krytox to drums of neat PF-5070 and mixing them very well. This is difficult due to the weight of the drums. Agitate the drums well as possible, and allow diffusion to occur for as long as possible.

A calibration curve is created that correlates Krytox concentration to UV absorbance on the Shimadzu Spectrophotometer between 0 and 1 wt-% Krytox in PF-5070. Using this calibration curve verify that the concentration of the 0.4 wt-% Krytox is in fact within ± 0.05 wt-% limits.

Experimental Procedure

Once setup, flow neat (i.e. surfactant free) fluid through the bypass to baseline the wavelength detector so once there is sufficient flow through the wavelength detector and the absorbance value is stable, zero the instrument. If at any point the wavelength detector is shut off, this step must be repeated to ensure consistent results.

After filling the packed adsorber column with neat PF-5070, start pumping 0.4 wt-% Krytox at intervals of approximately one adsorber volume. Initially, the absorbance recorded by the wavelength detector will drop below zero. This is due to the removal of UV absorbing contaminants. Allow for one hour of residence time in before pumping one adsorber volume of the 0.4 wt-% Krytox solution through the column. One hour from the start of the pumping cycle, pump one adsorber volume again. Continue to repeat the pumping intervals until the ACF cloth column no longer removes greater than 90% of the Krytox in solution.

The exhausted column is then exchanged for a new ACF cloth column. Once in the ACF cloth column is in place, run the experiment again in the same manner

Results

- The results are presented as Figure 6 through Figure 12.
- Figure 6 Rotameter Calibration
- Figure 7 Shimadzu spectrophotometer calibration
- Figure 8 Correlation between the wavelength detector and the spectrophotometer
- Figure 9 Trial 1: Comparison between the mass of Krytox captured by the ACF adsorber versus the amount pumped (Mass ACF Cloth = 597 g, Flow Rate = 1.3 l/min)
- Figure 10 Trial 1: Percent removal of Krytox (Mass ACF Cloth = 597 g, Flow Rate = 1.3 l/min)
- Figure 11 Trial 2: Comparison between the mass of Krytox captured by the ACF adsorber versus the amount pumped (Mass ACF Cloth = 521 g, Flow Rate = 500 ml/min)
- Figure 12 Trial 2 : Percent removal of Krytox (Mass ACF Cloth = 521 g, Flow Rate = 500 ml/min)

Figure 6 Rotameter Calibration

Figure 7 Shimadzu spectrophotometer calibration

Figure 8 Correlation between the wavelength detector and the spectrophotometer

Figure 9 Trial 1: Comparison between the mass of Krytox captured by the ACF adsorber versus the amount pumped (Mass ACF Cloth = 597 g, Flow Rate = 1.3 l/min)

Figure 10 Trial 1: Percent removal of Krytox (Mass ACF Cloth = 597 g, Flow Rate = 1.3 l/min)

Figure 11 Trial 2: Comparison between the mass of Krytox captured by the ACF adsorber versus the amount pumped (Mass ACF Cloth = 521 g, Flow Rate = 500 ml/min)

Figure 12 Trial 2 : Percent removal of Krytox (Mass ACF Cloth = 521 g, Flow Rate = 500 ml/min)

Discussion

Verification of Analytical Method

The initial step in deriving information from this data is to establish calibration curves. Two calibration curves are included among the figures above. Figure 6 is the calibration curve for the rotameter so flow rate of fluid through the carbon adsorption column is known and constant. Figure 7 is the Shimadzu spectrophotometer calibration, correlating UV absorption to the Krytox concentration in wt-%.

The Shimadzu spectrophotometer is a batch style instrument, but the data recording occurs in real time via the wavelength detector connected to the computer. It is because of this that Figure 8 is included. Figure 8 is a correlation between the wavelength detector UV absorbance and the spectrophotometer UV absorbance. This correlation is created by taking samples while running the full scale testing and comparing the wavelength detector measurement to that of the spectrophotometer. Combining the equation derived from this curve and the equation derived from Figure 7 yields an equation correlating the absorbance as detected by the wavelength detector to the concentration of Krytox in solution.

The above provides a method of analyzing the removal of Krytox from the PF-5070. All absorbance data for the effluent of the adsorber are translated into a Krytox concentration at that point in time. Integrating over all of these times allows the Krytox removal and, therefore, the carbon loading to be calculated.

Adsorption Results

Figure 9 through Figure 12 are the graphs for two separate trials that took place between June 3 - 11, 2002. The column used in the first trial consisted only of rolled activated carbon felt over a mandrel. The column used in the second trial differed from the one used in the first trial in that its ends were sealed with a polyethylene film.

The liquid flow rate was 1.3 l/min in the first test. In this test, the values of PG-1, PG-2 and PG-3 were respectively 23 to 26 psi, 18 to 19 psi, and 6.2 to 7 psi, over the length of the run. Most of the pressure drop, about 12 psi, was across the 5 μ m Millipore filter. The pressure drop across the adsorber column, was 6 ± 1 psi. For the second run, the liquid flow rate was reduced to 500 ml/min, resulting in significant reductions in pressure drop. The pressure drops across the Millipore filter and the adsorption column were approximately 2 ± 1 psi, and 1 ± 1 psi, respectively. The pressure drop readings were comparable to the reading uncertainty of the pressure gauges.

The results of the first test, depicted in Figure 9 and Figure 10, indicated bypassing. As seen in Figure 9, the two curves diverge quickly and by as little as two adsorber volumes, there is a significant difference between the mass of Krytox captured and that pumped. Figure 10 confirms this concept when it shows that 100% capture occurs for less than one adsorber volume of pumped fluid. Apparent capture of greater than 100% occurs because the carbon is also removing other UV absorbing material from the neat PF-5070.

The above analysis predicted that this column had removed approximately 338 g of Krytox. The mass of the ACF cloth column was recorded before and after the trial. It was found that the column weighed 1383 g on June 13, 1357 g on June 14, and 1314 g on June 21 (added by rk). These values correspond to weight gains of 478 g, 462 g, and 419 g. This indicates that some solvent remains in the carbon, and is still evaporating.

Because of these results, what was occurring inside the adsorber was re-examined. Short-circuiting is presumed to be the cause of the poor results of the first test. The mandrel is vertical in the adsorber with the ACF cloth wrapped around it. Originally, the thought was that the fluid would flow radially through the cloth to the mandrel since most of the pressure drop is through the mandrel. It appears as though this may not be the case. The shortest path, with the least pressure drop, is from the top and bottom of the wrap near the mandrel to the porous core of the mandrel. This would allow some liquid to bypass the carbon and result in seepage of adsorbate. To eliminate this problem, a sheet of polyethylene was placed over the top and bottom of the wrapped ACF cloth (cut from a one-gallon Ziploc[®] bag). These sheets were secured by simply tucking the edges of the plastic sheet into the mesh wrapped around the cloth, and by placing some tape near the center hole of the top piece to protect it against ripping. A sheet of foam was placed over the top of the wrapped carbon before putting the mandrel through the adsorber cover to apply some pressure and ensure that the top sheet of plastic did not come off.

With these modifications, the results show little or no bypassing with an overall recovery of 97.5% and carbon loading of 0.5 g Krytox/g carbon at the end of its effective life of five (5) cycles. Figure 11 and Figure 12 are the data plots for this experimental trial 2 that took place on June 6 - 11, 2002. Notice in Figure 12 that the percent removal of the column is over 100% for two cycles, meaning that the fluid was further cleaned beyond simply removing the surfactant Krytox.

During this experiment, eight cycles of fluid were passed through the adsorber, resulting in the removal of 97.6% of the Krytox pumped (0.8-g Krytox/g carbon). Two of these cycles, the sixth and seventh, the fluid was in contact with the bed for an extended period of time, overnight or over the weekend, respectively, before carrying out the run. This increase in residence time accounts for the increase in removal of Krytox from that fluid. As seen on the final cycle of the experiment, the carbon removed less Krytox in spite of the long residence time, indicating exhaustion of the carbon. These data indicate that if the effective lifetime criterion were relaxed, effective column life would be increased significantly.

These results were confirmed in two ways. The first method was by taking batch samples of the average effluent from each pumping interval and finding the Krytox concentration using the spectrophotometer. Calculating the mass of Krytox in each batch and integrating yields a value of 96.7% for the amount of Krytox captured. This value differs by less than 1% from the value found using the wavelength detector. Secondly, the carbon column was weighed before and after this trial and found to have gained 517 g in mass. As PF-5070 continues to

evaporate, the weight of the column will become closer to the 425 g expected from the results of the above analysis.

The Millipore filter that was downstream of the adsorption column was examined and weighed after each run. The weight gain for the filter used in the first run was 0.0346 g, and 0.0147 g for the second run. The filter from the first run was a deeper gray than the one from the second run. Microscopic examination of the filter from the second run indicated the presence of small filamentary particles, among others. These results indicate some slight loss of particles smaller than the nominal mandrel pore size from the filter bed.

Conclusion

The important points learned from this experiment regarding the use of an ACF cloth adsorption column are:

Fluid bypassing the ACF cloth in the adsorption column is a problem that can be eliminated by placing an impermeable sheet of polyethylene over the top and bottom of the wrapped ACF cloth column.

With these two sheets of plastic in place, the effective lifetime of the ACF cloth adsorption column is five (5) cycles consisting of pumping one adsorber volume of fluid at one-hour interval (40 L).

Operated under these conditions, the ACF cloth adsorption column will have collected 0.5 g Krytox/g carbon during its effective life (removing approximately 97.5% of the Krytox pumped through it).

APPENDIX B CARBON ADSORPTION TESTS

April 9, 2002

To: R. Kaiser
From: J. Jondro
Subject: Phosphor Calibration Curve

INTRODUCTION:

The focus of the current experimentation is to obtain a calibration curve to correlate the luminosity of fluorescent phosphor particles under UV illumination deposited on various substrates.

The following substrates were examined:

- *Aluminum*
- *Stainless steel*
- *Quartz (microscope slides)*
- *Cardboard (black, brown, and white)*

- *HEPA filter material*
- *Plastic (polypropylene, polyethylene –(Gladlock™ bag), polystyrene –(Petri dish)*
- *Rubber*
- *Wood*

The most detailed information was obtained with the aluminum substrate.

EXPERIMENTAL:

Materials

1. Test coupons: Initial tests were performed with a set of three - 1.5 inch square aluminum coupons. Subsequent tests with other materials were performed on substrates of similar geometry, unless otherwise noted.
2. Fluorescent contaminant: Osram Sylvania Phosphor 2284C SMSSCX suspended in HFE-7100/IPA Azeotrope (HFE-71-IPA), at concentrations of 0.1 wt-% and 0.5 wt-%.

Equipment

1. Camera: Coolsnap Pro Color Camera (Media Cybernetics) with UV haze filter.
2. Illumination: Sylvania 100W Par 38 Mercury Lamp
3. Data Capture and Analysis: Pentium III Personal Computer with Image-Pro Express (IPE) Imaging Software

PROCEDURE

Aluminum Coupons: The initial tests were performed with buffed 1.5-inch square coupons of 5051 aluminum alloy. Three coupons were used to obtain the measurements in triplicate. The test coupons were first cleaned very thoroughly to eliminate contaminants (including dust) and placed in a Petri dish to ensure that they stay clean. A picture of the clean coupon was taken and the image data captured on the computer, using the method described in Appendix A of this memorandum, to establish the illumination intensity of a blank coupon, i.e. at zero contamination.

The test coupons were then contaminated by adding 10 µl aliquots of the 0.1 wt-% phosphor suspension. After the suspending liquid had evaporated, a picture of the contaminated coupon was taken, and the same process was followed as was for the blank. Once the data from the imaging software was saved, an additional 10µl of suspension was added to each of the coupons, and the above process repeated. This same procedure was followed to gather data at 30, 40, 50, 70 and 80µl of the 0.1 wt-% suspension.

The test coupons were then cleaned and the contamination procedure was repeated with the addition of 15 µl, and subsequent additions of 10µl to get data at 25 µl and 35 µl of the 0.1 wt-% suspension. The objective was to obtain a multitude of data points at the low concentrations in order to determine the lower detection limit (LDL) of the procedure.

Data at higher surface concentration levels were obtained by adding aliquots of a 0.5 wt-% phosphor suspension. Pictures were taken at accumulated deposits of 20, 30, 40, 50, 60, 70, 80, 100, 150, 200, 250, 350, 400, 475, 550, 625, 700, and 800 μl .

Other Substrates: A procedure similar to the one described above was used to establish the illumination vs. phosphor concentration curve on other substrates, with some specific differences, as noted below:

a. Camera Exposure time: Because of background signal, the same exposure time could not be used on all substrates. The exposure time used was the time that gave maximum contaminant signal and minimum background signal.

b. Number of samples: The biggest difference between the process followed for the aluminum calibration and that of the other substrates was that all contaminated samples for the other substrates were saved for further analyses. This means that continued addition of contaminant could not be done and that there were more than three samples. There were six samples for each substrate between 0 - 2 g/m^2 .

c. Sample Geometry: Where possible, 1.5-inch square coupons were used. In some instances, the substrates could not be made in these dimensions. For these substrates, the formulae used in data analysis were corrected for the change in surface area.

RESULTS:

The luminosity versus concentration results for the various substrate materials are presented in the following Figures:

- Figure 13: Aluminum
- Figure 14: Stainless Steel Foil
- Figure 15: Quartz Microscope Slide
- Figure 16: Black Rubber
- Figure 17: Polystyrene
- Figure 18: Polyethylene
- Figure 19: Natural Polypropylene
- Figure 20: Black Cardboard
- Figure 21: Brown Cardboard
- Figure 22: White Cardboard
- Figure 23: HEPA Filter Material
- Figure 24: Wood

A set of photographs is presented in Appendix B for each substrate that was examined. Each set consists of photographs of the blank, the substrate at a low contamination level, and the substrate at a high contamination level.

Figure 13 Calibration curve for the luminosity of phosphor particles on an aluminum

Figure 14 Calibration curve for the luminosity of phosphor particles on stainless steel foil

Figure 15 Calibration curve for the luminosity of phosphor particles on a quartz microscope slide

Figure 16 Calibration curve for the luminosity of phosphor particles on a black rubber *Figure 17 Calibration curve for the luminosity of phosphor particles on polystyrene*

Figure 18 Calibration curve for the luminosity of phosphor particles on polyethylene

Figure 19 Calibration curve for the luminosity of phosphor particles on natural polypropylene

Figure 20 Calibration curve for the luminosity of phosphor particles on black cardboard

Figure 21 Calibration curve for the luminosity of phosphor particles on brown cardboard

Figure 22 Calibration curve for the luminosity of phosphor particles on white cardboard

Figure 23 Calibration curve for the luminosity of phosphor particles on white HEPA filter material

Figure 24 Calibration curve for the luminosity of phosphor particles on wood

DISCUSSION OF RESULTS:

The results on specific substrates are first discussed, followed by a comparison of the results obtained on the various substrates examined.

Aluminum Coupon (Figure 13)

The luminosity of phosphor particles on an aluminum coupon was studied in detail.

As can be in Figure 13, there were many data points taken at the low surface concentrations to determine the lower detection limit which is approximately 0.028 g/m² of contaminant. The luminosity is linear with concentration up to a level of approximately 1.9 g/m², and then starts to trail off with higher concentrations, indicating an approach to saturation.

There are multiple points at the lower concentrations that were repeated in an attempt to find out if sonication was beneficial. It turned out that sonication was beneficial because there was a slight increase in luminosity in the repeated points as seen in Figure 25. There may have been some phosphor particles that were becoming conglomerated, which was alleviated by use of the ultrasonic bath.

Figure B-1 includes the photographs of the aluminum coupons used in developing the calibration curve.

Figure 25 Portion of aluminum calibration curve showing the importance of sonication

Stainless Steel (Figure 14)

The next metallic substrate investigated was stainless steel. The substrates were cut from a thin sheet of steel into 1.5 inch square sizes and attached to a piece of cardboard the same size. One problem that was encountered while depositing on the stainless steel was that the suspension would channel toward any wrinkles or, even worse, roll off the substrate all together. All attempts were made to avoid this.

Stainless steel did create a higher background signal than the aluminum coupons did and, therefore, the exposure time of the camera was decreased to 500ms. Actual pictures of the stainless steel substrates are presented as Figure B-2.

Quartz Microscope Slides (Figure 15)

The calibration curve created using quartz microscope slides as the substrate has a high R^2 value, as seen in Figure 15. By having a haze filter on the camera, reflections of UV light were eliminated, so that this substrate was not a source of fluorescence, although it was transparent. Instead of having to worry about the background signal, the black backdrop of the photographic booth became a concern. Any dust on the black backdrop would show through and fluoresce so care was taken to remove all dust prior to taking photographs.

The size of the quartz slide and the exposure time used for this substrate were different than that used for the aluminum coupons. The size was 1" x 1.5" and the exposure time used was 500ms. Photographs of the quartz slides have been included as Figure B-3.

Black Rubber (Figure 16)

Black rubber is an interesting substrate in that the substrate is green under UV illumination as seen in Figure B-4. A reduction in the exposure time to 500ms allowed for the calibration curve located in Figure 16 to be created. The background signal created by the black rubber didn't interfere with the signal originating from the phosphor particles.

Polystyrene (Figure 17)

All of the substrates studied thus far were stored in polystyrene Petri dishes. For this calibration curve the Petri dish itself was contaminated and photographed to create the calibration curve found in Figure 17.

Like the quartz slides, this substrate is transparent so modifications were required. Again, the dust was cleared from the photographic booth where the pictures were taken and the exposure time was modified. For this substrate, the exposure time used was 250ms. Also, the area of the bottom of the Petri dish (3.14 in²) was used to create the calibration curve. Photographs of the polystyrene substrates are included in Figure B-5.

Polyethylene (Figure 18)

In order to explore the fluorescence of phosphor particles on polyethylene, 3" diameter circles were cut from a Gladlock™ storage bag. These circles were then secured to the aluminum coupons used for the calibration above so that they would be rigid enough to hold the

particles. Then the substrates were contaminated and photographed to create the calibration curve found in Figure 18. Actual photographs of the substrate are contained in Figure B-6. The exposure time used to photograph the polyethylene was 250ms.

Natural Polypropylene (Figure 19)

Natural polypropylene was a good substrate, yet it still had a background signal that can be seen in Figure B-7. Decreasing the exposure time alleviated this problem by decreasing the background signal. The signal from the fluorescing particles was then recorded without a problem. This created the calibration curve found in Figure 19.

Black Cardboard (Figure 20)

Like aluminum, phosphor particles on black cardboard produced a linear calibration curve as seen in Figure 20. The substrate is not a source of any major fluorescent signal, which would inhibit the determination of surface particle concentration. This fact aided in making the black cardboard calibration curve the one with the best curve fit. To see exactly how easily phosphor particles can be seen on this substrate refer to Figure B-8.

Brown Cardboard (Figure 21)

The best fit line for phosphor particles fluorescing on brown cardboard is very poor as seen in Figure 21. Notice that the slope of the best-fit line is also negative. This is because the substrate itself fluoresces. Because of the increase in signal from the coupon, the exposure time of the camera was decreased. For this particular substrate, the exposure time used was 100ms.

As the surface concentration of the phosphor particles increase, the particles are actually decreasing, or blocking, some of the background signal produced by the substrate. This is evident on the photograph contained in Figure B-9. As is seen in these photographs, deciphering between contaminant and the signal produced by the substrate is very difficult. That makes this a poor substrate to determine surface concentration using luminosity.

White Cardboard (Figure 22)

The results for a white cardboard substrate are very similar to that of brown cardboard in that the particles are actually blocking the signal produced by the substrate itself. The background signal produced by the white cardboard is even greater than that of the brown cardboard. Because of this, the exposure time was reduced even further to 25ms. Photographs of white cardboard illuminated by a UV lamp are included in Figure B-10.

Like brown cardboard, white cardboard also has a negatively sloped calibration curve. The substrate contains so much fluorescent material (commercial whitener) that it overwhelms the fluorescence of the phosphor particles. This makes white cardboard a poor substrate to do this type of analysis.

HEPA Filter Material (Figure 23)

The HEPA filter material poses another problem due to its geometry. Not only does the material that makes the HEPA filters fluoresce, but also the geometry contributes to the

problem. Notice in the photographs of the HEPA filter material in Figure B-11 that there are two vertical blue bands. These bands are the troughs. The signal resulting from the background in these troughs is great and any phosphor deposited here may be lost in the background signal. The calibration curve for the HEPA filter substrate is found in Figure 11. As seen, the best-fit curve for the data is very poor, yet removal of the first and third points greatly improves the fit. This comparison is found in Table 1.

Table 1 Comparison of calibration with and without first and third data points

	<u>Slope</u>	<u>Intercept</u>	<u>R²</u>
1st and 3rd Point Included	1.687x10 ⁷	1.620x10 ⁸	0.3139
1st and 3rd Point Excluded	2.295x10 ⁷	1.566x10 ⁸	0.8690

Wood (Figure 24)

Wood is another substrate that has a high background signal evident in Figure 12. Therefore, it is a poor substrate for determining surface concentration using luminosity. Because of the background signal originating in the substrate the exposure time was reduced to 500ms.

The wood sample came from a piece of flat clear pine molding and had the dimensions of 1.75" x 1.125" so that they would fit into a Petri dish. These changes in surface were incorporated in calculating the surface concentration of contaminant.

Comparison of Substrates

Table 2 presents the results of the calibration curves created for each of the substrates. They are in order of decreasing values of R², from the best curve fit to the worst. It is to be noted that only for the aluminum coupons were more than six data points acquired. R² values of 0.89 or above were obtained for eight of the twelve materials examined. For these materials, luminosity provides a reasonably accurate value of the surface concentration of the contaminant.

For each material examined other than aluminum, calibration curves were created based on all the data points obtained and also based only on the data points below a surface concentration level of 1.8 g/m², based on the data presented in Figure 1 that clearly indicate a nonlinear response of luminosity to concentration at values higher than 1.8 g/m². For the aluminum, an equivalent data set was obtained by dropping the highest value in the base set from the correlation. For the substrates that had the highest value of R² in the base correlation, the omission of this data point didn't affect the curve fit data significantly. The curves for which the omission of this point resulted in a significant change in the R² value, had very scattered data.

Table 2 Results of Phosphor Calibration

Substrate	Exposure Time, s	Number of Data Points	Slope	Intercept	R2	Number of Data Points	Slope	Intercept	R2
Black Cardboard	1	6	1.547×10^8	3.554×10^7	0.9972	5	1.539×10^8	3.583×10^7	0.994
Aluminum Coupon	1	31	2.085×10^8	1.309×10^7	0.9899	30	2.171×10^8	1.141×10^7	0.9923
Rubber	0.5	6	1.292×10^8	1.128×10^8	0.9847	5	1.288×10^8	1.129×10^8	0.968
Polystyrene	0.25	6	2.270×10^8	1.770×10^8	0.9542	5	2.719×10^8	1.613×10^8	0.9829
Quartz Slide	0.5	6	8.513×10^7	1.631×10^7	0.9491	5	8.807×10^7	1.469×10^7	0.9057
Polyethylene	0.25	6	1.128×10^8	4.827×10^7	0.9382	5	1.407×10^8	3.805×10^7	0.9847
Polypropylene	0.25	6	4.872×10^7	1.516×10^8	0.9087	5	5.557×10^7	1.488×10^8	0.9164
SS Sheet	1	6	1.826×10^8	5.983×10^7	0.8932	5	2.347×10^8	4.070×10^7	0.9436
HEPA Filter (Excluding 1st & 3rd Points)	0.1	4	2.295×10^8	1.566×10^8	0.869	3	1.383×10^7	1.613×10^8	0.9655
Wood	0.1	6	8.697×10^7	6.722×10^7	0.6771	5	2.491×10^7	9.294×10^7	0.6375
White Cardboard	0.025	6	-9.025×10^7	3.133×10^8	0.5317	5	-9.474×10^7	3.15×10^8	0.3718
Brown Cardboard	0.1	6	-7.194×10^7	2.11×10^8	0.4641	5	-8.576×10^7	2.161×10^8	0.3741
HEPA Filter (Including 1st & 3rd Points)	0.1	6	1.687×10^7	1.62×10^8	0.3139	5	3.563×10^6	1.669×10^8	0.01275

The background signal is a very important factor in establishing a calibration curve. A high background signal from the substrate makes deciphering between the luminosity of the phosphor particle and the luminosity of the substrate more difficult. To maximize the contaminant signal, the exposure time was adjusted to each substrate. The larger the background signal the further the exposure time had to be reduced. The variation in exposure time was not significant for materials that had the highest values of R², ranging from 0.25 to 1 seconds, at most. With pre-selected materials, effective photographs can be acquired with a constant exposure time of 0.5 seconds.

This doesn't mean that because a substrate fluoresces then it makes a poor substrate. Rubber for example, fluoresces green under UV illumination yet the correlation between surface contamination and luminosity is very good. The only way to determine whether a substrate is compatible with this method of determining contaminant concentration is to create a calibration curve.

Contaminated substrates can still be qualitatively evaluated at contamination levels much below the detection limit of the calibration. Figure 14, which is a photograph of an aluminum coupon contaminated with 0.0069 g/m² of phosphor particles. This contamination level is well below the detection limit of 0.028 g/m², based on the total area of the coupon.

Figure 26 Aluminum coupon contaminated with 0.0069 g/m²

CONCLUSIONS:

1. Not all substrates are compatible with the method of fluorescent particle analysis.
2. The suitability of a substrate depends on its optical characteristics, not its composition.
3. The method works best with surfaces that do not fluoresce under UV illumination.
4. The method is not reliable with substrates that fluoresce under UV illumination.

Appendix A - Methods for Image Capture and Analysis**Methods for Image Capture:**

- Place it on the stand under the UV black light controlled by a voltage regulator at 120 +/- 1V.
- Ensure the Cool Snap Pro camera is powered and then open the Image Pro software.
- Select "Acquire", then click "Video/Digital"
- Select the "Setup" tab, and then click the "Configure" button.
- Click "Manual Balance" button, and use the following settings:
Red: 2 Green: 2.5 Blue: 2
- Then click "OK" to close the "Manual Balance" window, and then again to close the "Configure" window.
- Then, select the "Preview" tab, and ensure the exposure time is set at "1 second" when photographing aluminum coupons.
- Make sure there are no lights on, and all stray light has been minimized.
- Then click "Snap", to photograph the image.
- Immediately a "Save File As" window will appear. Save the file with a brief file name in an appropriate folder.
- If analysis of more than one coupon is required, snap all pictures before performing analysis of the images.
- Remove contaminated coupons from under UV light before image analysis.

Methods for Image Analysis:

- Open the image desired for analysis.
- Select an Area of Interest (AOI) around the visible contaminant residue on the coupon. More often than not, the "Irregular AOI" function will be needed in combination with the "Add" function to capture all contaminated regions.
- Once all desired regions have been selected, select the "Histogram" function.
- Click the "Report", and scroll to "Statistics", then go to "File" and "Save Statistics to file", save under a data file on the desktop. This step will save Pixel Intensity Data to a temporary data file on the desktop.
- Then click "Report", and scroll to "Range/Area". Move the scaling on the histogram to the min and max values so that the entire area is incorporated, i.e. the output will say: X1:0, X2:4095, Area: "area", %: 100. These numbers means we have selected the "area" from pixel intensities 0 through 4095 which encompasses 100% of the area of the selected AOI. Now click "File", scroll to "Append Statistics to File" and add to the temporary data file on the desktop.
- Data can then be moved to a permanent master file, and the temporary file can be written over continuously.

Appendix B: Photographs of Substrates Evaluated.

a. Blank

b. 0.069 g/m²

c. 0.86 g/m²

Figure B-1 Stainless Steel Coupon

a. Blank

b. 0.069 g/m²

c. 0.86 g/m²

Figure B-2 Stainless Steel Coupon

a. Blank

b. 0.068 g/m²

c. 0.86 g/m²

Figure B-3 Quartz Microscope Slide

a. Blank

b. 0.069 g/m² c. 0.86 g/m²

Figure B-4 Black Rubber Coupon

a. Blank

b. 0.074 g/m² c. 0.86 g/m²

Figure B-5 Polystyrene Petri Dish

a. Blank

b. 0.069 g/m² c. 0.86 g/m²

Figure B-6 Polyethylene Coupon

a. Blank

b. 0.12 g/m² c. 1.2 g/m²
Figure B-7 Natural Polypropylene Coupon

a. Blank

b. 0.069 g/m² c. 0.86 g/m²
Figure B-8 Black Cardboard Coupons

a. Blank

b. 0.069 g/m² c. 0.86 g/m²
Figure B-9 Brown Cardboard Coupons

a. Blank

b. 0.069 g/m² c. 0.86 g/m²

Figure B-10 White Cardboard Coupon

a. Blank

b. 0.069 g/m² c. 0.86 g/m²

Figure B-11 HEPA Filter Coupons

a. Blank

b. 0.078 g/m² c. 0.97 g/m²

Figure B-12 Wood Coupon

APPENDIX C PRESSURE TESTING OF ADSORPTION VESSEL**MEMORANDUM**

TO: DR. KAISER
FROM: J. DESROSIERS
SUBJECT: ACF HOUSING PRESSURE TEST
DATE: 7-03-02

In order for the ACF housing to be accepted by SRS, it is necessary to demonstrate that the vessel will withstand a pressure equal to 1.5 times the maximum pressure it could be exposed to while in service. Since the highest possible pressure it could be exposed to is the output pressure of J-2 when dead-headed, or 29 psig, it was necessary to demonstrate that the vessel would contain fluids at a pressure of 1.5 times 29 psig, or 43.5 psig.

This was done by filling the vessel with water, and connecting the vessel to a source of pressurized nitrogen, as shown in Figure 1. The vessel was connected to a nitrogen cylinder through 1/4" polyethylene tubing. The vessel was pressurized by adjusting the regulator on the nitrogen cylinder to approximately 50 psig, with valve V-1 open and valve V-2 closed. At that point, the pressure gauge at the top of the vessel being tested read 46 psig. A recently purchased 0-60 psig class B (2% accuracy) pressure gauge, was used in this experiment. The gauge was not calibrated prior to the experiment. A picture of the vessel under test is given in Figure 2, with a close-up of the pressure gauge in Figure 3.

Valve V-1 was then closed to isolate the vessel from the nitrogen cylinder. After one hour, there was no change in the level of pressure in the isolated portion of the system. This indicates clearly that the pressure vessel can withstand an applied pressure of 46 psig without any noticeable effects, including leaking. The test was ended by closing the pressure regulator on the nitrogen cylinder, and opening valves V 1 and V-2 to release pressure.

Figure 1 - Experimental Arrangement - Pressure Testing of Adsorption Vessel

Figure 2: Adsorption vessel
being pressure tested.

Figure 3: Close-up of vessel showing pressure gauge At test pressure of 46 psig.

APPENDIX D PRIVATE COMMUNICATION

The information contained in this Appendix was produced by Entropic Systems, Inc. independently of the project described in this report. This Appendix is included in order to provide relevant background information.

MEMORANDUM

To: Dr. Robert Kaiser
CC: Adam Kulczyk
From: Josh Grilly
Re: Effect of Processing Conditions on Particle Removal by Sonication,
Report No: ESI IRAD Report 02-02
Date: September 23, 2002

I. PURPOSE

The purpose of these experiments was to determine the effect of ultrasonic frequency on the removal of particles in perfluoroheptane-surfactant solutions by means of sonication.

II. MATERIALS*Ultrasonic Bath*

Tests were conducted in a 5 gallon Blackstone-Ney ultrasonic bath, powered by a MultiSONIK™ Ultrasonic generator, capable of output at 40 kHz, 72 kHz, 104 kHz, and 170 kHz. The ultrasonic power density was 100 watts/gallon.

Substrate

Aluminum coupons cut into 1.5" squares were used as the test substrates.

Particle Contamination

Duke Scientific fluorescent microspheres of sizes 0.10 μm , 0.25 μm , and 0.94 μm were used as test particles. These particles fluoresce green under ultraviolet (UV) light.

Solvent

PF-5070 was used as a solvent, with Krytox surfactant concentrations of 0.0%, 0.1%, 0.2%, and 0.4%.

III. PROCEDURE*Particle Preparation*

The particles come suspended as 1% solutions in an aqueous solution. These solutions were further diluted with isopropyl alcohol. These dilute solutions were added drop wise to the test surface and then allowed to evaporate. The resulting surface coverage was approximately 0.1 g/m² – 0.2 g/m². Table 1 shows the surface coverage in percent and g/m² as a function of particle size, for 50 μL of particle suspension.

Table 3: Particle Surface Coverage

Particle Size (μm)	Surface Coverage (%)	Surface Coverage (g/m^2)
0.94	2.5%	0.163
0.25	5.2%	0.090
0.10	24.5%	0.172

Coupon Preparation

Sheets of 0.020" thick 2025 aluminum alloy were cut into 1.5" squares. The coupons were then scoured with abrasive to remove any luster, and cleaned with acetone. A picture of the uncontaminated coupon was taken under the UV light. The coupon was then contaminated with 50 μL of the appropriate particle suspension, and another picture was taken under the UV light.

Cleaning Procedure

After the coupons were contaminated, they were placed in jars containing approximately 50 mL of solvent / surfactant solution. The coupons were sonicated for ten minutes, and then the coupons were removed and placed in fresh solvent / surfactant solution. The coupons were then sonicated for three minutes, which essentially served as a rinse step to prevent redeposition of particles. Finally, a picture was taken of the cleaned coupon under the UV light.

Analytical Procedure

The method of determining residual contamination on the coupons involves taking digital pictures under an ultraviolet light. Image Pro Express software was used to quantify the average intensity of the green pixel illumination. The average intensity is reported as a value ranging from 0 to 4095. Pictures of the coupon were taken before contamination, after contamination, and after cleaning. The percent particle removal, R , is calculated by the following equation, where the values are in green pixel intensity:

$$R = 1 - \frac{\text{Cleaned} - \text{Uncontaminated}}{\text{Contaminated} - \text{Uncontaminated}}$$

IV. RESULTS

The experimental results are presented in Table 2.

Figures 1-3 represent uncontaminated, contaminated, and cleaned coupons, respectively, for a sample run with 0.10 μm particles. The pictures were all taken under a UV light. Figures 4-6 present similar data for 0.25 μm particles, and

Figures 7-9 for 0.94 μm particles. These pictures represent data obtained at 104 kHz and with 0.2 wt-% Krytox.

Table 4: Summary of Results –[(10 + 3) Minutes of Sonication]

Krytox Conc., wt-%	Particle Size (μm)	Ultrasonic Frequency (kHz)	% Removed
0	0.10	40	44.0%
0	0.25	40	62.4%
0	0.94	40	74.8%
0.1	0.10	40	54.5%
0.1	0.25	40	72.3%
0.1	0.94	40	95.1%
0.2	0.10	40	59.2%
0.2	0.25	40	72.8%
0.2	0.94	40	97.3%
0.4	0.10	40	69.8%
0.4	0.25	40	82.1%
0.4	0.94	40	95.6%
0	0.10	72	68.1%
0	0.25	72	77.6%
0	0.94	72	95.6%
0.1	0.10	72	65.7%
0.1	0.25	72	86.0%
0.1	0.94	72	93.3%
0.2	0.10	72	88.8%
0.2	0.25	72	94.1%
0.2	0.94	72	98.4%
0.4	0.10	72	80.5%
0.4	0.25	72	92.3%
0.4	0.94	72	98.2%
0	0.10	104	74.9%
0	0.25	104	85.8%
0	0.94	104	98.2%
0.1	0.10	104	85.9%
0.1	0.25	104	93.9%
0.1	0.94	104	98.5%
0.2	0.10	104	90.9%
0.2	0.25	104	95.2%
0.2	0.94	104	99.0%
0.4	0.10	104	90.4%
0.4	0.25	104	95.5%
0.4	0.94	104	98.4%
0	0.10	170	65.4%
0	0.25	170	74.6%
0	0.94	170	97.2%
0.1	0.10	170	74.0%
0.1	0.25	170	86.5%
0.1	0.94	170	96.8%
0.2	0.10	170	90.1%
0.2	0.25	170	93.3%
0.2	0.94	170	98.0%
0.4	0.10	170	92.5%
0.4	0.25	170	95.7%
0.4	0.94	170	97.8%

Figure 1
0.10 μm
Uncontaminated

Figure 2
0.10 μm
Contaminated

Figure 3
0.10 μm
Cleaned

Figure 4
0.25 μm
Uncontaminated

Figure 5
0.25 μm
Contaminated

Figure 6
0.25 μm
Cleaned

Figure 7
0.94 μm
Uncontaminated

Figure 8
0.94 μm
Contaminated

Figure 9
0.94 μm
Cleaned

V. DISCUSSION

The effects of the following three parameters on particle removal were investigated:

- particle size,
- ultrasonic frequency, and
- surfactant concentration.

Effect of Particle Size

For every set of frequency / surfactant concentration conditions, increasing the particle size resulted in increased particle removal.

Effect of Ultrasonic Frequency

Ultrasonic frequency had a significant effect on particle removal. An increase in the ultrasonic frequency generally enhanced the particle removal, to a point. Particle removal was significantly enhanced as the frequency was increased from 40 kHz to 72 kHz. A further increase was noted by increasing the frequency to 104 kHz. A further increase to 170 kHz resulted in a slight decline (as compared to 104 kHz) in particle removal. For particles between 0.1 μm and 1.0 μm , 104 kHz appeared to be the most effective frequency to use for particle removal.

Effect of Surfactant Concentration

The addition of surfactant to the solvent resulted in a noticeable improvement to particle removal up to a concentration of about 0.2 wt-%. A further increase in concentration to 0.4 wt-% resulted in little further improvement.

VI. CONCLUSIONS

- The larger the size of the particles, the easier they are to remove
- For particles in the 0.1 μm to 1.0 μm size range, the highest level of removal is obtained by sonicating at 104 kHz
- The addition of a small amount of surfactant (i.e. 0.1 wt-%) to the solvent dramatically increases particle removal. The optimal level of surfactant concentration appears to be 0.2 wt-%.

APPENDIX E MSDS

Sonatol A70
Material Safety Data Sheet

SECTION I - MANUFACTURER INFORMATION**Entropic Systems, Inc.**

34-C Holton Street
 Woburn, MA 01801

Emergency Telephone Number: Chem Trec 800-424-9300

Telephone Number for Information: 617-938-7588

Date Prepared: October 9, 1995

SECTION II - INGREDIENTS/IDENTIFY INFORMATION

Ingredients (Specific Chemical Identity; Common Names(s)):

	<u>CAS No.</u>	<u>%</u>
Perfluoro Carbon Compound	86508-42-1	100
(Primarily compounds with 7 carbons)		

OSHA PEL: None

ACGIH TLV: None

Other Recommended Limits: None

NOTE: For industrial use only. Not intended for use as a medical device or drug.

SECTION III - PHYSICAL/CHEMICAL CHARACTERISTICS

Boiling Point: 80° C (Typical)
 Specific Gravity: ca. 1.700
 Water = 1
 Vapor Pressure: ca. 79.0000 mmHg
 Calc. @ R.T.
 Melting Point: N/D
 Vapor Density: ca. 13.40
 Air = 1
 Calc. @ R.T.
 Evaporation Rate: >1.00 Butyl
 Acetate = 1
 Solubility in Water: nil
 Percent Volatile: ca. 99.7%
 Viscosity: ca. 0.5 centistokes @ R.T.
 Appearance and Odor: Colorless, odorless liquid

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

Flash Point: None (Setaflash CC) **Flammable Limits:** Not Applicable

LEL: Not Applicable **UEL:** Not Applicable

Extinguishing Media: Not Flammable

Special Fire Fighting Procedures: Self-contained breathing apparatus and full encapsulated suit impervious to HF.

Unusual Fire & Explosion Hazards: Toxic by-products may form upon decomposition.

NFPA-Hazard-Codes: Health 1 Fire 0 Reactivity 0

Unusual Reaction Hazard: none

SECTION V - REACTIVITY DATA:

Unstable	
Stable	X

Incompatibility (Materials to Avoid): Finely divided active metals, alkali and alkaline earth metals, in their reduced state.

Hazardous Decomposition or By-products: Thermal decomposition may produce trace amounts of HF and in some cases PFIB. Trace decomposition at 200°C and increased decomposition with increased surface temperatures.

Hazardous Polymerization:

May Occur	
Will Not Occur	X

SECTION VI - HEALTH HAZARD DATA

Eye Contact: Product is not expected to produce significant eye irritation. After product has been in use, contaminants may be introduced that may cause eye irritation: signs/symptoms include redness, swelling, pain, and tearing.

Skin Contact: No adverse health effects are expected from skin contact. After product has been in use, contaminants may be introduced that may cause skin irritation: signs/symptoms include redness, swelling, and itching.

Inhalation: The hazards associated with vapors of product are expected to be low. Above the boiling point small amounts of toxic decomposition products which may include hydrogen fluoride (HF) and perfluoroisobutylene (PFIB) may occur. Hydrogen fluoride (HF) has in ACGIH threshold limit value of 3 ppm of fluoride as a ceiling limit and an OSA PEL 3 ppm of fluoride as an eight hour time-weighted average and a 6 ppm of fluoride as a Short Term Exposure Limit. Perfluoroisobutylene (PFIB) has a recommended exposure guideline of 0.01 parts per million parts of air as a ceiling value.

If Swallowed: Ingestion is not a likely route of exposure to this product. No adverse health effects are expected from swallowing. Contaminants may evolve during use of the product which can be toxic if ingested.

SECTION VII - PRECAUTIONARY INFORMATION

Eye Protection: Avoid eye contact with vapor, spray, or mist. Wear safety glasses with side shields.

Skin Protection: Avoid prolonged or repeated skin contact. No skin protection required for room temperature usage.

Ventilation Protection: Use with appropriate local exhaust ventilation. Local exhaust recommended for temperatures at boiling point or greater.

Respiratory Protection: Avoid inhalation of thermal decomposition products. If decomposition occurs, in the absence of adequate ventilation, an air supplied respirator should be worn.

Prevention of Accidental Ingestion: Not applicable.

Recommended Storage: Not determined.

Fire and Explosion Avoidance: Not determined

Other Precautionary Information: Not expected to present a hazard when used with good industrial hygiene practices under the following conditions. Use only in areas with sufficient local exhaust ventilation to maintain airborne concentrations at recognized health and safety levels. Avoid prolonged breathing of vapors. Do not breathe thermal decomposition products. Do not smoke when using the product. Local exhaust ventilation with a minimum capture velocity of 50 linear feet per minute should be provided for applications at or above the boiling point. If interfering air currents are present, minimum capture velocity should be at least 100 linear feet per minute.

Source of Exposure Limit Data: NONE — None Established

SECTION VIII - ENVIRONMENTAL INFORMATION

Spill Response: Observe precautions from other sections. Cover with inorganic absorbent material. Collect spilled material. Place in a closed container.

Recommended Disposal: To reclaim or return, contact your sales representative.
U.S. EPA Hazardous Waste No.: None

Environmental Data: COD=Nil; BOD20=Nil; 96-Hr. LC50 Fathead Minnow (Pimephales promelas)=>1000 mg/1 (immiscible mixture); U.S. Clean Water Act, Section 307, Toxic Pollutants=None; Toxicity to waste treatment microorganisms = No inhibition of respiration rate at 1000 mg/1.

SARA Hazard Class: Fire Hazard: No
Pressure: No
Reactivity: No
Acute: Yes
Chronic: No

SECTION VIII - SUGGESTED FIRST AID

Eye Contact: Immediately flush with large amounts of water. Call a physician.

Skin Contact: Wash affected area with soap and water.

Inhalation: If exposed to decomposition products, remove person to fresh air. Call a physician.

If Swallowed: Call a physician or Poison Control Center with detailed description. Include possible material that contaminated liquid during use.

APPENDIX F DATA LOG

1) List of Trials with Parameters

Date - Week of	Run	Substrate	Substrate Evaluated	Chemistry	Means of Shear	Bath	Frequency (kHz)	Duty	Time (min.)	Temp	Basket	Baffles	Liquid Level	Rotation	Comments
7/7/02	1	Aluminum	Aluminum	Neat Sonatol	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/7/02	2	PE	PE	Neat Sonatol	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/7/02	3	Aluminum & PE	Aluminum	Neat Sonatol	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/7/02	4	Aluminum & PE	PE	Neat Sonatol	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/7/02	5	Aluminum	Aluminum	.4% Krytox	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/7/02	6	PE	PE	.4% Krytox	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/7/02	7	Aluminum & PE	Aluminum	.4% Krytox	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/7/02	8	Aluminum & PE	PE	.4% Krytox	Spray	Multi	N/A	N/A	5	room	ADF	none	normal	yes	
7/15/02	9	Aluminum	Aluminum	Neat Sonatol	US	Multi	40	100	5	room	ADF	none	normal	yes	
7/15/02	10	Aluminum	Aluminum	Neat Sonatol	US	Multi	72	100	5	room	ADF	none	normal	yes	
7/15/02	11	Aluminum	Aluminum	Neat Sonatol	US	Multi	104	100	5	room	ADF	none	normal	yes	
7/15/02	12	Aluminum	Aluminum	Neat Sonatol	US	Multi	170	100	5	room	ADF	none	normal	yes	
7/15/02	13	Aluminum	Aluminum	Neat Sonatol	US	Multi	40	100	5	high	ADF	none	normal	yes	
7/15/02	14	Aluminum	Aluminum	Neat Sonatol	US	Multi	72	100	5	high	ADF	none	normal	yes	
7/15/02	15	Aluminum	Aluminum	Neat Sonatol	US	Multi	104	100	5	high	ADF	none	normal	yes	
7/15/02	16	Aluminum	Aluminum	Neat Sonatol	US	Multi	170	100	5	high	ADF	none	normal	yes	
7/15/02	17	Aluminum	Aluminum	Neat Sonatol	US	Multi	40	35	5	high	ADF	none	normal	yes	
7/15/02	18	Aluminum	Aluminum	Neat Sonatol	US	Multi	72	35	5	high	ADF	none	normal	yes	
7/15/02	19	Aluminum	Aluminum	Neat Sonatol	US	Multi	104	35	5	high	ADF	none	normal	yes	
7/15/02	20	Aluminum	Aluminum	Neat Sonatol	US	Multi	170	35	5	high	ADF	none	normal	yes	
7/15/02	21	PE	PE	Neat Sonatol	US	Multi	40	100	5	room	ADF	none	normal	yes	
7/15/02	22	PE	PE	Neat Sonatol	US	Multi	72	100	5	room	ADF	none	normal	yes	
7/15/02	23	PE	PE	Neat Sonatol	US	Multi	104	100	5	room	ADF	none	normal	yes	
7/15/02	24	PE	PE	Neat Sonatol	US	Multi	170	100	5	room	ADF	none	normal	yes	
7/15/02	25	PE	PE	Neat Sonatol	US	Multi	40	100	5	high	ADF	none	normal	yes	
7/15/02	26	PE	PE	Neat Sonatol	US	Multi	72	100	5	high	ADF	none	normal	yes	
7/15/02	27	PE	PE	Neat Sonatol	US	Multi	104	100	5	high	ADF	none	normal	yes	
7/15/02	28	PE	PE	Neat Sonatol	US	Multi	170	100	5	high	ADF	none	normal	yes	
7/15/02	29	PE	PE	Neat Sonatol	US	Multi	40	35	5	high	ADF	none	normal	yes	

Separation and Extraction of Plutonium in Mixed Waste

7/15/02	30	PE	PE	Neat Sonatol	US	Multi	72	35	5	high	ADF	none	normal	yes	
7/15/02	31	PE	PE	Neat Sonatol	US	Multi	104	35	5	high	ADF	none	normal	yes	
7/15/02	32	PE	PE	Neat Sonatol	US	Multi	170	35	5	high	ADF	none	normal	yes	
7/22/02	33	Aluminum	Aluminum	.4% Krytox	US	Multi	40	100	5	room	ADF	none	normal	yes	
7/22/02	34	Aluminum	Aluminum	.4% Krytox	US	Multi	72	100	5	room	ADF	none	normal	yes	
7/22/02	35	Aluminum	Aluminum	.4% Krytox	US	Multi	104	100	5	room	ADF	none	normal	yes	
7/22/02	36	Aluminum	Aluminum	.4% Krytox	US	Multi	170	100	5	room	ADF	none	normal	yes	
7/22/02	37	Aluminum	Aluminum	.4% Krytox	US	Multi	40	100	5	high	ADF	none	normal	yes	
7/22/02	38	Aluminum	Aluminum	.4% Krytox	US	Multi	72	100	5	high	ADF	none	normal	yes	
7/22/02	39	Aluminum	Aluminum	.4% Krytox	US	Multi	104	100	5	high	ADF	none	normal	yes	
7/22/02	40	Aluminum	Aluminum	.4% Krytox	US	Multi	170	100	5	high	ADF	none	normal	yes	
7/22/02	41	Aluminum	Aluminum	.4% Krytox	US	Multi	40	35	5	high	ADF	none	normal	yes	
7/22/02	42	Aluminum	Aluminum	.4% Krytox	US	Multi	72	35	5	high	ADF	none	normal	yes	
7/22/02	43	Aluminum	Aluminum	.4% Krytox	US	Multi	104	35	5	high	ADF	none	normal	yes	
7/22/02	44	Aluminum	Aluminum	.4% Krytox	US	Multi	170	35	5	high	ADF	none	normal	yes	
7/22/02	45	PE	PE	.4% Krytox	US	Multi	40	100	5	room	ADF	none	normal	yes	
7/22/02	46	PE	PE	.4% Krytox	US	Multi	72	100	5	room	ADF	none	normal	yes	
7/22/02	47	PE	PE	.4% Krytox	US	Multi	104	100	5	room	ADF	none	normal	yes	
7/22/02	48	PE	PE	.4% Krytox	US	Multi	170	100	5	room	ADF	none	normal	yes	
7/22/02	49	PE	PE	.4% Krytox	US	Multi	40	100	5	high	ADF	none	normal	yes	
7/22/02	50	PE	PE	.4% Krytox	US	Multi	72	100	5	high	ADF	none	normal	yes	
7/22/02	51	PE	PE	.4% Krytox	US	Multi	104	100	5	high	ADF	none	normal	yes	
7/22/02	52	PE	PE	.4% Krytox	US	Multi	170	100	5	high	ADF	none	normal	yes	
7/22/02	53	PE	PE	.4% Krytox	US	Multi	40	35	5	high	ADF	none	normal	yes	
7/22/02	54	PE	PE	.4% Krytox	US	Multi	72	35	5	high	ADF	none	normal	yes	
7/22/02	55	PE	PE	.4% Krytox	US	Multi	104	35	5	high	ADF	none	normal	yes	
7/22/02	56	PE	PE	.4% Krytox	US	Multi	170	35	5	high	ADF	none	normal	yes	
7/22/02	57	PE	PE	.4% Krytox	US	Multi	104	100	5	high	ADF	none	normal	yes	
7/22/02	58	PE	PE	.4% Krytox	US	Multi	104	100	5	high	ADF	screen	normal	yes	
7/29/02	59	PE	PE	.4% Krytox	US	Multi	104	100	5	high	ADF	non porous	high	yes	
7/29/02	60	PE	PE	.4% Krytox	US	Multi	104	100	5	high	ADF	non porous	normal	yes	
7/29/02	61	PE	PE	.4% Krytox	US	Multi	104	100	5	room	ADF	non porous	normal	yes	
7/29/02	62	PE	PE	.4% Krytox	US	Multi	104	100	5	high	ADF	non porous	low	yes	

Separation and Extraction of Plutonium in Mixed Waste

7/29/02	63	PE	PE	.4% Krytox	US	Multi	104	100	5	room	none	N/A	normal	N/A	
7/29/02	64	PE	PE	.4% Krytox	US	Crest	132	100	5	room	none	N/A	1 inch	N/A	
7/29/02	65	PE	PE	.4% Krytox	US	Multi	104	100	10	room	none	N/A	normal	N/A	
7/29/02	66	PE	PE	.4% Krytox	US	Crest	132	100	10	room	none	N/A	1 inch	N/A	
7/29/02	67	PE	PE	.4% Krytox	US	Multi	104	100	5	room	ADF	none	normal	yes	
8/5/02	68	PE	PE	.4% Krytox	US	Multi	104	100	5	room	Crest 1 (perf. steel)	N/A	normal	N/A	
8/5/02	69	PE	PE	.4% Krytox	US	Multi	104	100	5	room	Crest 2 (4-mesh)	N/A	normal	N/A	
8/5/02	70	PE	PE	.4% Krytox	US	Multi	104	100	10	room	Crest 2 (4-mesh)	N/A	normal	N/A	
8/5/02	71	PE	PE	.4% Krytox	US	Multi	104	100	5	room	Rotating 2 (4-mesh)	none	normal	no	
8/5/02	72	PE	PE	.4% Krytox	US	Multi	104	100	10	room	Rotating 2 (4-mesh)	none	normal	no	
8/5/02	73	PE	PE	.4% Krytox	US	Multi	104	100	10	room	Rotating 2 (4-mesh)	none	normal	yes	*Added Haze -2 Filter
8/5/02	74	PE	PE	.4% Krytox	US	Multi	104	100	10	room	ADF	none	normal	no	*Blackstone Ney Generator Arrives
8/5/02	75	PE	PE	.4% Krytox	US	Single	104	100	10	room	ADF	none	normal	no	
8/5/02	76	PE	PE	.4% Krytox	US	Single	104	100	10	room	Rotating 1 (perf. steel)	none	normal	no	
8/5/02	77	PE	PE	.4% Krytox	US	Single	104	100	10	room	Rotating 1 (perf. steel)	none	normal	yes	
8/12/02	78	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	104	100	10	room	Rotating 2 (4-mesh)	none	normal	yes	
8/12/02	79	Kitty Litter	Kitty Litter	.4% Krytox	US	Multi	104	100	10	room	1000 mL Glass Beaker	none	normal	N/A	
8/12/02	80	Kitty Litter	Kitty Litter	.4% Krytox	US	Multi	104	100	10	room	Crest 1 (perf. steel)	none	normal	N/A	
8/12/02	81	Vermiculite	Vermiculite	.4% Krytox	US	Multi	104	100	10	room	1000 mL Glass Beaker	none	normal	N/A	

Separation and Extraction of Plutonium in Mixed Waste

8/12/02	82	Vermiculite Mixed Waste	Vermiculite Mixed Waste	.4% Krytox	US	Multi	104 Complete Cycle	100	10	room	Rotating 1 (perf. steel) Final Basket	none	normal	N/A	
8/19/02	1B	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	2B	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	3B	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	1D	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	2D	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	3D	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	1E	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	2E	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/19/02	3E	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/26/02	1F	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/26/02	2F	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	
8/26/02	3F	Mixed Waste	Mixed Waste	.4% Krytox	US	Multi	Complete Cycle	100	60	room	Final Basket	none	normal	yes	

2) List of Summary Trial Results

Date - Week of	Run	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Decon Factor
7/7/02	1	150	1502	1301	1.2
7/7/02	2	150	235	248	0.9
7/7/02	3	150	1799	1462	1.3
7/7/02	4	150	408	385	1.1
7/7/02	5	159	1873	981	2.1
7/7/02	6	102	1671	825	2.2
7/7/02	7	154	1950	867	2.5
7/7/02	8	102	1661	671	2.7
7/15/02	9	160	424	245	3.1
7/15/02	10	160	403	217	4.3
7/15/02	11	160	468	187	11.4
7/15/02	12	160	574	409	1.7
7/15/02	13	160	469	323	1.9
7/15/02	14	160	574	291	3.2
7/15/02	15	160	503	232	4.8
7/15/02	16	160	634	377	2.2
7/15/02	17	160	561	368	1.9
7/15/02	18	160	615	283	3.7
7/15/02	19	160	641	261	4.8
7/15/02	20	160	618	399	1.9
7/15/02	21	102	472	480	1.0
7/15/02	22	102	443	442	1.0
7/15/02	23	102	448	428	1.1
7/15/02	24	102	470	447	1.1
7/15/02	25	102	473	439	1.1
7/15/02	26	102	488	418	1.2
7/15/02	27	102	473	432	1.1
7/15/02	28	102	480	440	1.1
7/15/02	29	102	394	393	1.0
7/15/02	30	102	428	404	1.1
7/15/02	31	102	453	416	1.1
7/15/02	32	102	396	385	1.0
7/22/02	33	172	1838	268	17.5
7/22/02	34	158	1730	207	31.7
7/22/02	35	155	1660	188	46.5
7/22/02	36	168	1519	244	17.7
7/22/02	37	171	1545	304	10.3
7/22/02	38	145	1622	206	24.3
7/22/02	39	159	2461	187	81.4
7/22/02	40	154	2805	196	62.7
7/22/02	41	172	2691	302	19.5
7/22/02	42	158	2531	199	57.6
7/22/02	43	155	2393	180	91.5
7/22/02	44	168	2140	205	52.8
7/22/02	45	102	701	426	1.9

7/22/02	46	102	797	497	1.8
7/22/02	47	102	770	180	8.5
7/22/02	48	102	732	470	1.7
7/22/02	49	102	816	446	2.1
7/22/02	50	102	832	454	2.1
7/22/02	51	102	828	257	4.7
7/22/02	52	102	755	483	1.7
7/22/02	53	102	701	482	1.6
7/22/02	54	102	877	481	2.0
7/22/02	55	102	904	294	4.2
7/22/02	56	102	852	574	1.6
7/29/02	57	118	719	195	7.8
7/29/02	58	118	703	272	3.8
7/29/02	59	118	747	205	7.2
7/29/02	60	118	645	186	7.8
7/29/02	61	118	604	267	3.3
7/29/02	62	118	601	295	2.7
8/5/02	63	122	548	145	18.7
8/5/02	64	122	639	137	35.4
8/5/02	65	122	589	145	20.3
8/5/02	66	122	639	129	72.0
8/5/02	67	122	622	399	1.8
8/5/02	68	122	540	153	13.5
8/5/02	69	122	624	148	19.6
8/5/02	70	122	716	148	23.3
8/5/02	71	90	662	100	53.4
8/5/02	72	90	662	98	65.5
8/5/02	73	99	730	109	60.8
8/5/02	74	98	784	352	2.7
8/5/02	75	98	727	382	2.2
8/5/02	76	102	971	127	34.1
8/5/02	77	102	943	127	33.6

3) List of Detailed Trial Results

Run 1	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 6	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	150	1492	1286	1	102	1414	643
2	150	1537	1376	2	102	1580	623
3	150	1605	1285	3	102	1668	637
4	150	1709	1497	4	102	1371	691
5	150	1166	1063	5	102	1735	888
AVG	150	1502	1301	6	102	1769	854
STD	0	205	159	7	102	1861	1110
Run 2	Blank GPL/A	Cont. GPL/A	Clean GPL/A	8	102	1805	999
1	150	231	254	9	102	1697	936
2	150	224	243	10	102	1810	865
3	150	245	246	AVG	102	1671	825
4	150	241	249	STD	0	167	169
5	150	237	247	Run 7	Blank GPL/A	Cont. GPL/A	Clean GPL/A
AVG	150	235	248	1	121	1553	706
STD	0	8	4	2	125	1998	765
Run 3	Blank GPL/A	Cont. GPL/A	Clean GPL/A	3	180	1881	739
1	150	2218	1851	4	157	1890	749
2	150	1761	1473	5	156	1725	917
3	150	1628	1272	6	140	1617	805
4	150	1841	1476	7	169	2103	748
5	150	1550	1235	8	152	2032	909
AVG	150	1799	1462	9	139	2378	1179
STD	0	260	245	10	203	2318	1151
Run 4	Blank GPL/A	Cont. GPL/A	Clean GPL/A	AVG	154	1950	867
1	150	372	403	STD	25	275	172
2	150	473	404	Run 8	Blank GPL/A	Cont. GPL/A	Clean GPL/A
3	150	401	346	1	102	1600.5	838.1
4	150	455	439	2	102	1460.7	894.4
5	150	339	332	3	102	1738.9	994.6
AVG	150	408	385	4	102	1766.0	619.3
STD	0	56	44	5	102	1644.4	562.1
Run 5	Blank GPL/A	Cont. GPL/A	Clean GPL/A	6	102	1386.7	754.2
1	113	1465	713	7	102	1734.4	767.5
2	152	1862	1122	8	102	1802.0	308.5
3	183	1948	940	9	102	1724.1	558.5
4	198	2133	984	10	102	1755.4	413.2
5	158	1308	750	AVG	102	1661	671
6	188	1938	985	STD	0	139	217
7	214	2231	1059	Run 9	Blank GPL/A	Cont. GPL/A	Clean GPL/A
8	118	2156	1092	1	160	458.0	257.7
9	135	2110	1197	2	160	443.7	250.0
10	133	1576	963	3	160	367.6	202.8
AVG	159	1873	981	4	160	387.6	334.6
STD	35	319	153	5	160	465.4	177.8
				AVG	160	424	245
				STD	0	44	60

Run 10	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 16	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	160	495	200	1	160	771.8	317.8
2	160	359	172	2	160	602.1	397.6
3	160	353	182	3	160	639.8	347.5
4	160	441	278	4	160	556.2	391.9
5	160	366	252	5	160	600.2	429.9
AVG	160	403	217	AVG	160	634	377
STD	0	63	46	STD	0	83	44
Run 11	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 17	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	160	319	161	1	160	565	336
2	160	605	210	2	160	441	324
3	160	496	201	3	160	596	332
4	160	440	148	4	160	586	360
5	160	479	215	5	160	620	489
AVG	160	468	187	AVG	160	561	368
STD	0	103	30	STD	0	70	68
Run 12	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 18	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	160	542	304	1	160	655.2	281.9
2	160	580	308	2	160	660.7	186.0
3	160	607	689	3	160	526.2	252.8
4	160	311	306	4	160	535.5	392.4
5	160	827	437	5	160	697.5	300.4
AVG	160	574	409	AVG	160	615	283
STD	0	184	166	STD	0	79	75
Run 13	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 19	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	160	371	262	1	160	774	249
2	160	450	246	2	160	666	335
3	160	436	558	3	160	438	197
4	160	396	318	4	160	722	242
5	160	690	230	5	160	602	281
AVG	160	469	323	AVG	160	641	261
STD	0	128	135	STD	0	130	51
Run 14	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 20	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	160	454	214	1	160	610	380
2	160	530	353	2	160	644	429
3	160	595	219	3	160	549	341
4	160	624	297	4	160	680	339
5	160	669	373	5	160	607	507
AVG	160	574	291	AVG	160	618	399
STD	0	84	74	STD	0	49	71
Run 15	Blank GPL/A	Cont. GPL/A	Clean GPL/A				
1	160	452	244				
2	160	551	224				
3	160	443	209				
4	160	531	221				
5	160	537	259				
AVG	160	503	232				
STD	0	51	20				

Run 21	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 25	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102	443.6	464.0	1	102	503	408
2	102	524.8	458.1	2	102	447	424
3	102	506.8	499.1	3	102	495	433
4	102	474.9	511.0	4	102	493	466
5	102	462.7	524.3	5	102	426	463
6	102	436.2	489.1	AVG	102	473	439
7	102	499.2	462.2	STD	0	34	25
8	102	440.5	496.7	Run 26	Blank GPL/A	Cont. GPL/A	Clean GPL/A
9	102	446.7	422.4	1	102	403	499
10	102	480.9	474.3	2	102	518	375
AVG	102	472	480	3	102	575	368
STD	0	31	30	4	102	488	428
Run 22	Blank GPL/A	Cont. GPL/A	Clean GPL/A	5	102	456	421
1	102	474	438	AVG	102	488	418
2	102	441	432	STD	0	65	52
3	102	433	435	Run 27	Blank GPL/A	Cont. GPL/A	Clean GPL/A
4	102	450	407	1	102	495	241
5	102	452	426	2	102	519	494
6	102	428	498	3	102	384	503
7	102	446	419	4	102	469	394
8	102	418	459	5	102	497	528
9	102	415	445	AVG	102	473	432
10	102	468	458	STD	0	53	118
AVG	102	443	442	Run 28	Blank GPL/A	Cont. GPL/A	Clean GPL/A
STD	0	20	25	1	102	461.2	465.3
Run 23	Blank GPL/A	Cont. GPL/A	Clean GPL/A	2	102	491.9	473.8
1	102	442	465	3	102	528.5	332.4
2	102	416	372	4	102	478.7	428.1
3	102	481	431	5	102	437.4	498.4
4	102	470	481	AVG	102	480	440
5	102	433	393	STD	0	34	65
AVG	102	448	428	Run 29	Blank GPL/A	Cont. GPL/A	Clean GPL/A
STD	0	27	46	1	102	423	361
Run 24	Blank GPL/A	Cont. GPL/A	Clean GPL/A	2	102	375	428
1	102	498	416	3	102	369	363
2	102	447	479	4	102	418	404
3	102	452	421	5	102	386	411
4	102	444	478	AVG	102	394	393
5	102	506	440	STD	0	25	30
AVG	102	470	447				
STD	0	30	30				

Run 30	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 34	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102	432.4	376.2	1	160	1948	188
2	102	442.2	395.6	2	137	1369	180
3	102	435.7	413.2	3	130	1767	161
4	102	418.6	413.4	4	198	1807	240
5	102	410.5	421.9	5	131	1690	170
AVG	102	428	404	6	190	1809	268
STD	0	13	18	7	183	1618	215
Run 31	Blank GPL/A	Cont. GPL/A	Clean GPL/A	8	130	1764	198
1	102	443	420	9	171	1697	217
2	102	438	413	10	149	1830	238
3	102	450	444	AVG	158	1730	207
4	102	447	413	STD	26	156	34
5	102	485	393	Run 35	Blank GPL/A	Cont. GPL/A	Clean GPL/A
AVG	102	453	416	1	198	2003	223
STD	0	19	19	2	140	1425	164
Run 32	Blank GPL/A	Cont. GPL/A	Clean GPL/A	3	188	1704	218
1	102	393	422	4	145	1616	176
2	102	307	394	5	138	1885	160
3	102	370	309	6	148	1611	180
4	102	457	453	7	144	1424	168
5	102	454	346	8	185	1725	228
AVG	102	396	385	9	124	1674	148
STD	0	63	58	10	143	1530	211
Run 33	Blank GPL/A	Cont. GPL/A	Clean GPL/A	AVG	155	1660	188
1	157	1551	256	STD	25	185	29
2	210	2063	304	Run 36	Blank GPL/A	Cont. GPL/A	Clean GPL/A
3	159	2022	250	1	145	1401	229
4	170	1841	273	2	183	1316	227
5	169	2126	283	3	165	1581	256
6	178	1751	289	4	147	1688	224
7	125	1368	212	5	161	1610	224
8	179	1993	295	6	175	1751	267
9	192	1974	285	7	167	1590	247
10	186	1698	229	8	147	1182	219
AVG	172	1838	268	9	204	1546	288
STD	23	245	30	10	183	1528	259
				AVG	168	1519	244
				STD	19	173	23

Run 37	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 40	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	184	1658	296	1	121	2236	151
2	158	1596	294	2	125	2931	160
3	171	1195	304	3	180	3024	215
4	150	1587	252	4	157	2817	191
5	207	1786	363	5	156	2437	179
6	172	1567	313	6	140	2224	178
7	172	1796	360	7	169	2919	214
8	163	1330	284	8	152	3124	218
9	158	1247	252	9	139	3350	189
10	174	1692	324	10	203	2993	271
AVG	171	1545	304	AVG	154	2805	196
STD	16	215	38	STD	25	381	35
Run 38	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 41	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	117	1603	177	1	157	2167	321
2	154	1852	191	2	210	2624	311
3	155	1152	223	3	159	2907	255
4	123	1642	183	4	170	2979	326
5	181	1826	260	5	169	2995	301
6	161	1665	223	6	178	2199	305
7	127	1734	168	7	125	2075	234
8	128	1670	187	8	179	2716	331
9	118	1645	186	9	192	3099	323
10	184	1429	259	10	186	3150	309
AVG	145	1622	206	AVG	172	2691	302
STD	26	203	34	STD	23	408	32
Run 39	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 42	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	113	2058	152	1	160	2704	194
2	152	2418	177	2	137	1782	182
3	183	2586	198	3	130	2370	154
4	198	2660	222	4	198	2852	214
5	158	2022	199	5	131	2942	149
6	188	2352	244	6	190	2513	253
7	214	2863	238	7	183	2056	242
8	118	3039	138	8	130	2410	179
9	135	2545	170	9	171	2720	209
10	133	2069	137	10	149	2962	216
AVG	159	2461	187	AVG	158	2531	199
STD	35	346	39	STD	26	387	34
Run 43	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 44	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	198	2615	197	1	145	2107	205
2	140	1853	179	2	183	1974	218
3	188	2332	205	3	165	2589	197
4	145	2713	187	4	147	2661	202
5	138	2556	162	5	161	2436	170
6	148	2071	190	6	175	2289	199
7	144	2139	183	7	167	1830	219
8	185	2487	179	8	147	1606	177
9	124	2656	145	9	204	1898	254
10	143	2511	171	10	183	2011	209
AVG	155	2393	180	AVG	168	2140	205
STD	25	285	17	STD	19	344	23

Run 45	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 49	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	647	507	1	102.0	783	471
2	102.0	657	346	2	102.0	914	290
3	102.0	672	207	3	102.0	740	616
4	102.0	677	546	4	102.0	834	450
5	102.0	698	636	5	102.0	836	322
6	102.0	734	491	6	102.0	842	520
7	102.0	760	532	7	102.0	882	571
8	102.0	679	367	8	102.0	891	378
9	102.0	790	378	9	102.0	793	369
10	102.0	696	248	10	102.0	648	470
AVG	102	701	426	AVG	102	816	446
STD	0	46	138	STD	0	79	106
Run 46	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 50	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	678	539	1	102.0	815	433
2	102.0	755	475	2	102.0	675	446
3	102.0	683	503	3	102.0	855	258
4	102.0	835	190	4	102.0	796	530
5	102.0	765	541	5	102.0	915	569
6	102.0	796	611	6	102.0	935	686
7	102.0	823	510	7	102.0	899	164
8	102.0	859	743	8	102.0	894	524
9	102.0	924	653	9	102.0	858	235
10	102.0	856	201	10	102.0	677	693
AVG	102	797	497	AVG	102	832	454
STD	0	78	178	STD	0	93	184
Run 47	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 51	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	692	256	1	102.0	639	411
2	102.0	743	165	2	102.0	830	237
3	102.0	726	283	3	102.0	700	250
4	102.0	817	155	4	102.0	711	334
5	102.0	826	205	5	102.0	819	178
6	102.0	812	173	6	102.0	839	296
7	102.0	761	131	7	102.0	868	343
8	102.0	780	140	8	102.0	984	183
9	102.0	783	159	9	102.0	941	168
10	102.0	756	134	10	102.0	953	169
AVG	102	770	180	AVG	102	828	257
STD	0	43	52	STD	0	116	86
Run 48	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 52	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	669	219	1	102.0	832	297
2	102.0	713	566	2	102.0	763	163
3	102.0	695	545	3	102.0	802	581
4	102.0	810	551	4	102.0	787	656
5	102.0	801	565	5	102.0	760	610
6	102.0	732	494	6	102.0	762	656
7	102.0	702	492	7	102.0	765	464
8	102.0	729	495	8	102.0	722	588
9	102.0	681	501	9	102.0	642	620
10	102.0	789	267	10	102.0	716	199
AVG	102	732	470	AVG	102	755	483
STD	0	51	124	STD	0	53	193

Run 53	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 57	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	647	194	1	118	650	260
2	102.0	657	364	2	118	680	192
3	102.0	672	645	3	118	688	220
4	102.0	677	490	4	118	737	156
5	102.0	698	512	5	118	791	167
6	102.0	734	498	6	118	738	176
7	102.0	760	516	7	118	667	203
8	102.0	679	566	8	118	827	169
9	102.0	790	578	9	118	698	220
10	102.0	696	460	10	118	715	188
AVG	102	701	482	AVG	118	719	195
STD	0	46	126	STD	0	56	32
Run 54	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 58	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	891	631	1	118	556	320
2	102.0	1038	713	2	118	667	393
3	102.0	995	651	3	118	865	233
4	102.0	1019	301	4	118	771	309
5	102.0	879	394	5	118	751	190
6	102.0	698	240	6	118	754	438
7	102.0	900	386	7	118	682	180
8	102.0	833	696	8	118	643	205
9	102.0	694	274	9	118	616	200
10	102.0	827	521	10	118	721	250
AVG	102	877	481	AVG	118	703	272
STD	0	121	184	STD	0	88	90
Run 55	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 59	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	915	232	1	118	695	210
2	102.0	988	141	2	118	626	173
3	102.0	968	263	3	118	696	193
4	102.0	928	493	4	118	734	188
5	102.0	954	267	5	118	667	251
6	102.0	873	658	6	118	747	234
7	102.0	789	189	7	118	855	199
8	102.0	846	212	8	118	1000	193
9	102.0	930	213	9	118	807	209
10	102.0	853	275	10	118	642	199
AVG	102	904	294	AVG	118	747	205
STD	0	63	158	STD	0	114	23
Run 56	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 60	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	102.0	717	476	1	118	664	198
2	102.0	880	382	2	118	637	197
3	102.0	587	507	3	118	690	153
4	102.0	795	736	4	118	596	164
5	102.0	900	648	5	118	722	178
6	102.0	725	545	6	118	570	188
7	102.0	966	461	7	118	633	176
8	102.0	1038	730	8	118	613	242
9	102.0	979	496	9	118	659	190
10	102.0	934	760	10	118	667	173
AVG	102	852	574	AVG	118	645	186
STD	0	142	134	STD	0	45	24

Run 61	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	118	567	225
2	118	574	193
3	118	605	316
4	118	669	221
5	118	618	346
6	118	705	313
7	118	510	171
8	118	592	341
9	118	634	223
10	118	570	322
AVG	118	604	267
STD	0	56	67

Run 62	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	118	531	301
2	118	584	341
3	118	591	293
4	118	483	284
5	118	690	367
6	118	566	176
7	118	534	267
8	118	731	272
9	118	572	309
10	118	728	338
AVG	118	601	295
STD	0	86	53

Run 63 & 65	Blank GPL/A	Cont. GPL/A	Clean GPL/A 5min	Clean GPL/A 10 min
1	122	803	149	140
2	122	587	140	150
3	122	484	146	
4	122	520		
5	122	548		
6	122	590		
AVG	122	589	145	145
STD	0	112	5	7

Run 64 & 66	Blank GPL/A	Cont. GPL/A	Clean GPL/A 5min	Clean GPL/A 10 min
1	122	729	132	129
2	122	571	144	130
3	122	608	134	
4	122	695		
5	122	622		
6	122	609		
AVG	122	639	137	129
STD	0	60	6	1

Run 67	Blank GPL/A	Cont. GPL/A	Clean GPL/A	
1	122	680	473	
2	122	574	562	
3	122	597	381	
4	122	579	406	
5	122	678	175	
AVG	122	622	399	
STD	0	53	144	
Run 68	Blank GPL/A	Cont. GPL/A	Clean GPL/A	
1	122	532	149	
2	122	498	149	
3	122	495	144	
4	122	618	171	
5	122	554	153	
AVG	122	540	153	
STD	0	50	11	
Run 69	Blank GPL/A	Cont. GPL/A	Clean GPL/A	
1	122	680	147	
2	122	636	143	
3	122	605	140	
4	122	580	153	
5	122	620	155	
AVG	122	624	148	
STD	0	37	7	
Run 70	Blank GPL/A	Cont. GPL/A	Clean GPL/A	
1	122	639	149	
2	122	729	144	
3	122	803	152	
4	122	679	148	
5	122	728	144	
AVG	122	716	148	
STD	0	62	3	
Run 71 & 72	Blank GPL/A	Cont. GPL/A	Clean GPL/A 5min	Clean GPL/A 10 min
1	89.6	694	96	100
2	89.6	631	100	98
3	89.6	730	105	97.34181
4	89.6	604		
5	89.6	631		
6	89.6	682		
AVG	90	662	100	98
STD	0	48	4	1

Run 73	Blank GPL/A	Cont. GPL/A	Clean GPL/A	Run 77	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	99.1	709	103	1	101.6	616	117
2	99.1	724	108	2	101.6	776	118
3	99.1	749	112	3	101.6	840	126
4	99.1	671	112	4	101.6	1083	130
5	99.1	809	115	5	101.6	1093	124
6	99.1	721	107	6	101.6	1076	132
7	99.1	793	110	7	101.6	1165	133
8	99.1	705	107	8	101.6	826	129
9	99.1	647	107	9	101.6	1151	121
10	99.1	768	113	10	101.6	806	138
AVG	99	730	109	AVG	102	943	127
STD	0	48	4	STD	0	182	6

Run 74	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	97.6	665	315
2	97.6	576	507
3	97.6	568	552
4	97.6	566	622
5	97.6	589	178
6	97.6	997	222
7	97.6	930	374
8	97.6	1042	253
9	97.6	876	169
10	97.6	1031	322
AVG	98	784	352
STD	0	198	152

Run 75	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	97.6	445	187
2	97.6	651	276
3	97.6	551	519
4	97.6	478	602
5	97.6	925	371
6	97.6	802	434
7	97.6	885	441
8	97.6	939	419
9	97.6	692	314
10	97.6	899	260
AVG	98	727	382
STD	0	180	120

Run 76	Blank GPL/A	Cont. GPL/A	Clean GPL/A
1	101.6	869	132
2	101.6	1011	140
3	101.6	828	130
4	101.6	1175	120
5	101.6	863	121
6	101.6	995	127
7	101.6	1050	120
8	101.6	983	122
9	101.6	1175	128
10	101.6	762	131
AVG	102	971	127
STD	0	134	6

8/12/2002

Run 78

Mixed Waste Component	Sample	Blank GPL/A	Contaminated GPL/A	Clean GPL/A	Decon Factor	Clean - Blank	Decon Factor
Aluminum Coupon	1	127	1658	106	-71.2	-21	364
	2	147	1801	122	-66.0	-25	394
Black Polyethylene Coupon	1	90	668	87	-204.2	-3	178
	2	91	708	93	258.3	2	190
Stainless Steel Union	1	155	890	102	-13.8		
Stainless Steel Bushing	1	213	899	140	-9.4		
Stainless Steel Elbow	1	302	1957	198	-15.9		
Tin Paint Can Cover	1	130	1700	103	-57.2		
Rubber Apron Coupon	1	591	1471	498	-9.5		

* the error on each blank and clean aluminum coupon value is +_ 3

* the error on each blank and clean plastic coupon value is +_ 2.3

Aluminum		* the error on each blank and clean aluminum coupon value is +_ 3			
		4.20			
Protocol 1					
List 1					
sample	blank	contam .	clean	clean-blank	D.F
1	114	766	119	5	125
2	118	620	136	18	28
3	111	509	136	24	16
AVG	115	632	130	16	33
STDev	3	129	10		
Protocol 2					
List 1					
sample	blank	contam .	clean	clean-blank	D.F
1	123	824	118	-5	167
2	118	735	96	-22	147
3	123	682	91	-32	133
AVG	121	747	101	-20	149
STDev	3	72	15		
Protocol 2a					
List 4					
sample	blank	contam .	clean	clean-blank	D.F
1	151	1119	168	17	57
2	151	1263	149	-2	265
3	151	1147	148	-3	237
AVG	151	1176	155	4	244
STDev	0	76	11		
Plastic		* the error on each blank and clean plastic coupon value is +_ 2.3			
		3.25			
Protocol 1					
List 1					
sample	blank	contam .	clean	clean-blank	D.F
1	90	234	103	13	11
2	93	353	109	15	17
3	97	319	113	15	14

Separation and Extraction of Plutonium in Mixed Waste

4	95	402	109	14	21
5	96	292	109	13	15
6	95	425	106	11	30
AVG	94	337	108	14	18
STDev	4	61	5		
Protocol 2					
List 1					
sample	blank	contam .	clean	clean-blank	D.F
1	89	401	93	4	76
2	90	326	97	7	34
3	96	421	95	-1	100
4	97	394	97	0	91
5	93	353	98	6	45
6	98	339	102	3	69
AVG	94	372	97	3	85
STDev	4	50	2		
Protocol 2a					
List 4					
sample	blank	contam .	clean	clean-blank	D.F
1	94	631	86	-8	165
2	94	591	83	-11	153
3	94	633	87	-7	166
4	94	567	95	1	145
5	94	577	90	-4	149
AVG	94	600	89	-5	156
STDev	0	23	2		

Aluminum					
Protocol 1					
List 2					
sample	blank	contam .	clean	clean-blank	D.F
1	106	829	158	51	14
2	121	732	140	19	32
3	112	1033	137	26	36
AVG	113	865	145	32	24
STDev	7	154	11		
Protocol 2					
List 2					
sample	blank	contam .	clean	clean-blank	D.F
1	81	807	113	32	23
2	105	640	121	16	34
3	87	681	104	17	35
AVG	91	709	112	22	29
STDev	13	87	9		
Protocol 2a					
List 5					
sample	blank	contam .	clean	clean-blank	D.F
1	149	755	147	-3	144
2	175	930	160	-16	180
3	177	834	164	-13	156
AVG	167	840	157	-11	160
STDev	16	88	9		
Plastic					
Protocol 1					
List 2					
sample	blank	contam .	clean	clean-blank	D.F
1	88	409	105	17	19
2	97	455	109	12	29
3	97	367	109	12	22

Separation and Extraction of Plutonium in Mixed Waste

4	96	430	106	10	34
5	94	379	106	12	24
6	100	476	106	6	64
AVG	95	419	107	12	28
STDev	5	44	2		
Protocol 2					
List 2					
sample	blank	contam .	clean	clean-blank	D.F
1	94	355	96	1	80
2	93	324	97	4	59
3	96	342	102	6	41
4	96	338	96	0	74
5	97	395	100	4	84
6	95	373	104	9	30
AVG	95	354	99	4	65
STDev	1	16	3		
Protocol 2a					
List 5					
sample	blank	contam .	clean	clean-blank	D.F
1	84	382	91	7	43
2	82	429	96	13	26
3	84	420	96	12	28
4	86	421	90	3	101
5	87	481	96	9	45
6					
AVG	85	427	94	9	39
STDev	1	25	3		

Aluminum					
Protocol 1					
List 3					
sample	blank	contam .	clean	clean-blank	D.F
1	112	767	127	15	44
2	112	905	127	14	56
3	112	1084	113	1	231
AVG	112	919	122	10	80
STDev	0	159	8		
Protocol 2					
List 3					
sample	blank	contam .	clean	clean-blank	D.F
1	118	634	121	4	123
2	118	637	119	2	124
3	118	721	124	6	96
AVG	118	664	122	4	130
STDev	0	50	3		
Protocol 2a					
List 6					
sample	blank	contam .	clean	clean-blank	D.F
1	147	732	141	-6	139
2	165	678	142	-23	122
3	157	781	139	-18	149
AVG	156	730	141	-15	137
STDev	9	51	2		
Plastic					
Protocol 1					
List 3					
sample	blank	contam .	clean	clean-blank	D.F
1	93	426	98	5	63
2	98	430	98	0	102
3	94	479	101	7	57

Separation and Extraction of Plutonium in Mixed Waste

4	93	441	103	10	36
5	96	410	106	11	30
6	98	564	108	10	45
AVG	95	458	102	7	51
STDev	3	30	2		
Protocol 2					
List 3					
sample	blank	contam .	clean	clean-blank	D.F
1	96	354	97	2	79
2	96	367	100	4	76
3	96	344	100	4	57
4	94	366	101	7	38
5	98	380	100	2	87
6	96	302	99	3	63
AVG	96	352	100	4	71
STDev	1	12	1		
Protocol 2a					
List 6					
sample	blank	contam .	clean	clean-blank	D.F
1	85	458	91	7	55
2	85	484	91	6	67
3	88	507	98	9	44
4	91	470	96	5	79
5	95	477	97	2	118
6	91	497	93	2	125
AVG	89	482	94	5	76
STDev	2	24	4		

4) MITNRL NAA Data

ALUMINUM

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	1372	9.03	146840	0.27
Blank 2	1521	6.90	149692	0.26
Blank 3	1375	9.18	156165	0.26
Blank 4	1302	10.00	153185	0.26
Average Blank	1393	8.78	151471	0.26
Standard Deviation of Blank	92	1.32	4065	0.00
Contaminated 1	270127	0.25	157676	0.25
Contaminated 2	273311	0.27	167497	0.26
Contaminated 3	295728	0.23	177407	0.24
Contaminated 4	283098	0.20	165717	0.25
Average (1-4) Contaminated	280566	0.24	167074	0.25
Standard Deviation of Contaminated	11517	0.03	8106	0.01
Contaminated 5	449003	0.18	93200	0.33
Contaminated 6	384943	0.20	88266	0.34
Average (5-6) Contaminated	416973	0.19	90733	0.34
Standard Deviation of Contaminated	45297	0.01	3489	0.01
Average (1-6) Contaminated	326035	0.22	141627	0.28

Clean Samples								
List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
1	1	1	1845	5.59	617	149826	0.27	-9
1	1	2	2680	4.78	217	150452	0.27	-15
1	1	Average	2263	5.19	321	150139	0.27	-12
2	1	1	2126	5.19	381	164665	0.27	1
2	1	2	1679	5.95	974	123677	0.29	-1
2	1	Average	1903	5.57	547	144171	0.28	-2
3	1	1	1750	7.28	781	158783	0.26	2
3	1	2	1730	7.43	827	168769	0.24	1
3	1	Average	1740	7.36	803	163776	0.25	1
All	1	Average	1968	6.04	485	152695	0.27	13
1	2	1	2342	4.92	294	145387	0.27	-3
1	2	2	2873	4.12	189	147159	0.27	-4
1	2	Average	2608	4.52	230	146273	0.27	-3
2	2	1	2734	4.04	208	155230	0.28	4
2	2	2	2420	4.77	272	172037	0.24	1
2	2	Average	2577	4.41	236	163634	0.26	1
3	2	1	2564	5.31	238	165199	0.25	1
3	2	2	1791	6.87	701	160784	0.25	2
3	2	Average	2178	6.09	356	162992	0.25	1
All	2	Average	2454	5.01	263	157633	0.26	0
4	2a	1	2408	5.14	409	30876	0.59	1
4	2a	2	2716	4.59	314	29863	0.59	0
4	2a	Average	2562	4.87	355	30370	0.59	1
5	2a	1	2538	4.33	363	36250	0.55	1
5	2a	2	1896	5.35	825	33258	0.57	1
5	2a	Average	2217	4.84	504	34754	0.56	1
6	2a	1	1530	6.89	3022	27699	0.62	0
6	2a	2	1472	7.11	5227	28288	0.61	0
6	2a	Average	1501	7.00	3830	27994	0.62	0
All	2a	Average	2093	5.57	593	31039	0.59	1
All	All	Average	2172	5.54	417	113789	0.37	0

BLACK POLYETHYLENE

	Cerium	Error %	Zinc	Error %
Blank 1	217	14.61	6850	1.27
Blank 2	215	22.67	6780	1.29
Blank 3	195	18.01	7615	1.16
Blank 4	242	29.93	7256	1.21
Average Blank	217.25	21.31	7125	1.23
	Cerium	Error %	Zinc	Error %
Contaminated 1	211080	0.25	33371	0.58
Contaminated 2	212715	0.28	34746	0.62
Contaminated 3	233552	0.23	37765	0.55
Contaminated 4	212281	0.27	35024	0.35
Average (1-4) Contaminated	217407	0.26	35227	0.53
Contaminated 5	209902	0.38	541469	0.14
Average (1-5) Contaminated	215906	0.28	136475	0.45

Clean Samples

List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
1	1	1	570	22.16	616	7215	1.21	313
2	1	1	712	22.51	439	5906	1.36	-23
2	1	2	1808	4.81	137	6632	1.27	-57
2	1	Average	1260	13.66	208	6269	1.32	-33
3	1	1	2803	7.01	84	13032	0.90	5
3	1	2	872	10.02	332	7874	1.16	38
3	1	Average	1838	8.52	134	10453	1.03	8
All	1	Average	1353	13.30	191	8132	1.18	28
1	2	1	3831	3.81	60	6583	1.34	-52
1	2	2	338	14.36	1799	5675	1.17	-19
1	2	3	1450	4.49	176	6252	1.35	-32
1	2	Average	1873	7.55	131	6170	1.29	-29
2	2	1	400	8.25	1188	6180	1.51	-30
2	2	2	671	6.04	479	6676	1.43	-63
2	2	3	2654	3.11	89	6118	1.51	-28
2	2	Average	1242	5.80	212	6325	1.48	-35
3	2	1	223	20.07	37772	6697	1.30	-66
3	2	2	267	14.85	4366	6416	1.33	-40
3	2	3	891	6.35	322	7611	1.23	58
3	2	4	152	26.35	-3329	6482	1.32	-44
3	2	Average	383	16.91	1308	6802	1.30	-87
All	2	Average	1088	10.77	250	6469	1.35	-43
4	2a	1	1479	4.57	166	12563	1.06	98
5	2a	1	416	16.80	1055	6911	1.24	-2494
5	2a	2	1258	6.56	201	6833	1.25	-1828
5	2a	Average	837	11.68	338	6872	1.25	-2110
6	2a	1	400	20.78	1147	7594	1.48	1140
6	2a	2	220	28.56	76249	5968	1.35	-462
6	2a	Average	310	24.67	2261	6781	1.42	-1552
All	2a	Average	755	15.45	390	7974	1.28	630
All	All	Average	1071	12.57	253	7261	1.29	954

Lead Lined Rubber

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	18365	3.29	489368	0.15
Blank 2	19233	1.26	498095	0.13
Average of Blank	18799	2.28	493732	0.14
Standard Deviation of Blank	614	1.44	6171	0.01
Contaminated 1	154459	0.48	545271	0.14
Contaminated 2	173943	0.45	625673	0.12
Average (1-2) Contaminated	164201	0.47	585472	0.13
Standard Deviation of Contaminated	13777	0.02	56853	0.01
Contaminated 3	209902	0.38	541469	0.14
Average (1-3) Contaminated	179435	0.44	570804	0.13

Clean Samples								
List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
1	1	1	23497	0.14	31	599318	0.12	1
1	1	2	24460	4.37	26	622872	0.13	1
1	1	Average	23979	2.26	28	611095	0.13	1
2	1	1	23122	0.97	34	607518	0.11	1
2	1	2	23026	0.60	34	572181	0.12	1
2	1	Average	23074	0.79	34	589850	0.12	1
All	1	Average	23526	1.52	31	600472	0.12	1
1	2	1	23854	2.77	29	537481	0.14	2
1	2	2	20949	3.13	68	517884	0.15	4
1	2	Average	22402	2.95	40	527683	0.15	3
2	2	1	21669	2.84	51	499400	0.15	16
2	2	2	21466	2.89	55	516444	0.15	4
2	2	Average	21568	2.87	53	507922	0.15	6
All	2	Average	21985	3	46	517802	0	4
4	2a	1	21970	3.06	60	609045	0.13	0
4	2a	2	22263	3.11	55	651191	0.13	0
4	2a	Average	22116.5	3.09	58	630118	0.13	0
6	2a	1	28332	2.46	20	665896	0.13	0
6	2a	2	26823	2.61	24	678267	0.13	0
6	2a	Average	27578	2.54	22	672081.5	0.13	0
All	2a	Average	24847	3	32	651100	0	0
All	All	Average	23453	2	35	589791	0	1

IPA TOWELETTE

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	144	23.82	411	5.62
Contaminated 1	216341	0.25	27964	0.64
Contaminated 2	214111	0.25	28735	0.65
Average (1-2) Contaminated	215226	0.25	28350	0.65
Contaminated 3	480453	0.15	55658	0.45
Average (1-3) Contaminated	303635	0.22	37452	0.58

Clean Samples								
List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
3	1	1	16983	1.55	13	4944	1.50	6
3	1	2	27245	0.86	8	7317	1.34	4
3	1	Average	22114	1.21	10	6131	1.42	5
3	2	1	9317	1.80	23	3512	1.76	9
3	2	2	16211	1.22	13	5348	1.62	6
3	2	Average	12764	1.51	17	4430	1.69	7
6	2a	1	33510	0.56	14	8077	1.1	7
6	2a	2	29533	0.74	16	6937	1.33	8
6	2a	Average	31521.5	0.65	15	7507	1.22	8
All	All	Average	22133	1.12	14	6023	1.44	7

IPA TOWEL PACKAGE

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	1573	12.56	6144	1.35
Contaminated 1	205513	0.27	34318	0.55
Contaminated 2	775555	0.13	101535	0.32
Average (1-2) Contaminated	490534	0.20	67927	0.44

Clean Samples								
List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
3	1	1	1645	12.19	2833	6546	1.31	70
3	1	2	2093	9.29	392	6066	1.35	-361
3	1	Average	1869	10.74	689	6306	1.33	174
3	2	1	1915	3.11	596	7103	1.25	29
3	2	2	2760	7.15	172	7782	1.20	17
3	2	Average	2338	5.13	267	7443	1.23	22
6	2a	1	4173	2.24	298	13042	0.90	14
6	2a	2	3787	5.24	350	17245	0.78	9
6	2a	Average	3980	3.74	322	15144	0.84	11
All	All	Average	2729	6.54	423	9631	1.13	18

HEPA Filter

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	205	69.90	262463	0.20
Contaminated 1	404234	0.20	310092	0.18
Contaminated 2	816057	0.14	602949	0.13
Average (1-2) Contaminated	610146	0.17	456521	0.16

Clean Samples								
List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
3	1	1	109517	0.52	4	255964	0.20	-7
3	1	2	103601	0.55	4	264887	0.20	20
3	1	Average	106559	0.54	4	260426	0.20	-23
3	2	1	81628	0.69	5	265612	0.20	15
3	2	2	96238	0.59	4	263610	0.20	42
3	2	Average	88933	0.64	5	264611	0.20	22
6	2a	1	267996	0.36	3	512637	0.14	1
6	2a	2	256084	0.30	3	508020	0.14	1
6	2a	Average	262040	0.33	3	510329	0.14	1
All	All	Average	152511	0.50	4	345122	0.18	2

Black Cardstock

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	3310	2.09	209	8.8
Contaminated 1	641194	0.15	71577	0.38

Clean Samples								
List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
5	2a	1	78324	0.51	9	14784	0.86	5
5	2a	2	125274	0.37	5	22086	0.69	3
5	2a	Average	101799	0.44	6	18435	0.78	4

Rubber Apron

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	13463	1.69	493354	0.15
Contaminated 1	459072	0.26	539813	0.14

Clean Samples								
List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
5	2a	1	40174	2.4	17	503806	0.14	4
5	2a	2	33686	2.79	22	499291	0.14	8
5	2a	Average	36930	2.60	19	501549	0.14	6

Wood

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	58	40.1	4071	5.14
Blank 2	69	52.3	868	4.26
Average Blank	63.5	46.2	2469.5	4.7
Contaminated 1	501746	0.14	58306	0.42

Clean Samples

List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
5	2a	1	42635	0.75	12	11528	0.96	6
5	2a	2	48180	0.43	10	13213	1.01	5
5	2a	Average	45408	0.59	11	12371	0.99	6

Blue Tarp

	Cerium	Error (%)	Zinc	Error (%)
Blank 1	123	18.7	232	11.78
Contaminated 1	353933	0.18	37807	0.55

Clean Samples

List	Protocol	Sample	Cerium	Cerium Error %	Decon Factor Cerium	Zinc	Zinc Error %	Decon Factor Zinc
2		1	1560	3.16	246	539	6.45	122
2		2	3072	3.63	120	600	5.72	102
2		Average	2316	3.40	161	570	6.09	111

IRRADIATED LEAD LINED RUBBER

Sample	Cerium				Zinc			
	Blank	Contaminated	Clean	Decon Factor	Blank	Contaminated	Clean	Decon Factor
1	24999	266829	73711	5	519070	721565	702765	1
2	22132	219038	73244	4	528094	748008	713538	1
3	30310	212385	69426	5	624751	823955	800547	1
4	26047	216457	49969	8	534084	778563	761046	1
Average	25872	228677	66588	5	551499.8	768022.75	744474	1

5) Calculation of Error

Blank Coupons			Contaminated Coupons		
	Aluminum	Plastic		Aluminum	Plastic
1	190.17	100.89	1	740.06	485.74
2	188.61	108.54	2	787.60	486.30
3	192.08	108.61	3	776.35	495.05
4	188.97	107.75	4	785.71	504.10
5	188.52	107.79	5	744.40	489.39
6	184.77	108.40	6	765.19	513.07
7	191.87	109.08	7	782.26	513.05
8	187.04	109.09	8	793.84	485.29
9	193.84	106.18	9	750.51	478.21
10	194.98	107.20	10	799.58	479.47
AVG	190.08	107.35	AVG	772.55	492.97
STDEV	2.98	2.32	STDEV	20.20	12.26

The Table above presents the statistical data for a blank aluminum and plastic coupon as well as for a contaminated aluminum and plastic coupon. Each coupon had its image acquired, was re-positioned, and had its image acquired again. Ten images were taken for each coupon.

APPENDIX G SYSTEM INSTALLATION

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Appendix G – System Assembly Protocol

Introduction

This document describes the systematic procedure to follow when assembling the Pu-238 Decontamination System in a glove box. Once all equipment for this system is in the glove box, there is very little room to move anything, so it is important that each component be in its correct position from the start.

Sub-Assembly Order of Operation

Sub-Assemblies		
<i>Order</i>	<i>Component</i>	<i>Weight (lbs)</i>
1	<u>ULTRASONIC GENERATOR</u>	20.5/
2	Internal Bulkhead Connections	-
3	Turbidity Meter (TM-1)	10.5
4	Filter F-3 Apparatus (F-3a and F-3b)	12.5
5	Holding Tank (T-1)	26.5
6	Filter F-2/Bracket Assembly (F-2)	16.5
7	Pump J-1	18
8	Heat Exchanger (C-1)	2
9	Activated Carbon Filter (ACF) Housing (D-1)	26.5
10	<u>MOVE ELECTRICAL CABLES</u>	-
11	Cleaning Chamber/Chamber Base Assembly	77.5
12	ELECTRICAL CONNECTIONS 1	-
13	Pump J-2	16
14	Gear Motor	18
15	Electrical Connections 2	-
16	External Bulkhead Connections	-

Appendix G – System Assembly Protocol

1) Blackstone-Ney Multi-Frequency Ultrasonic Generator:

Place the ultrasonic generator adjacent to the glove box. Then connect the signal cable (W-2) that relays information to the programmable logic controller (PLC) and the 220V power cable (W-1).

2) Internal Bulkhead Fittings

There are three electrical bulkhead connections: power, signal, and ultrasonic power (Fig. 2.1) Connect the internal power and signal cable connections to the bulkhead fittings in the rear right of the glove box. Layout these cables in the rear of the gloveboxes shown in Fig. 2.2.

Fig 2.1 – Inside glovebox electrical bulkhead connections

Fig 2.2 – Cable layout to the rear of the glovebox

3) Turbidity Meter/Bracket Assembly (TM-1):

The turbidity meter/ bracket assembly should be positioned on the shelf in the rear left corner of the glove box.

Figure 3.1 - Turbidity meter on the shelf in the corner of the glove box

4) Filter F-3:

The filter F-3 assembly consists of two F-3 filter modules attached in parallel and supported by four legs. Slide the F-3 assembly into the left corner directly below the shelf on which TM-1 sits. The Solenoid valve AV-2 should be pointing to the right.

Figure 4.1 - Filter F-3 assembly positioned under the shelf in the left corner of the glove box.

5) **Tank T-1:**

1. *Pass the T-1 platform with the attached legs into the glove box.*
2. *Pass tank T-1 through the glove box door, bottom first, with all connections pointing towards the window (see Figure 5.1). Lay T-1 on the floor of the glove box and lineup the Velcro tapes so that the T-1 platform can be attached to the bottom of T-1 (see Figure 5.2). Then rotate the T-1/T-1 platform assembly and place it on the glove box shelf.*
3. *Slide the T-1 assembly to the left until it is 2" from the TM-1 bracket.*
4. *Pass the T-1 Swagelok VCO connection in into the glove box*
5. *Using a wrench, connect the VCO fittings. This connects Con 6.*
6. *The quick disconnect fitting that includes the pressure gauge, PG-2, and the pressure transducer, P-2, is passed into the glove box, and then attached to the right side of T-1 through the mating quick disconnect fitting provided (no Con number)..*
7. *Connect Con 5*

Appendix G – System Assembly Protocol

Figure 5.1 - Passing T-1 through glove box window

Figure 5.2 T-1 attached to the platform

Figure 5.3 - T-1 assembly in place

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Figure 5.4 - Connect Con 5

6) **Filter F-2/Bracket Assembly:**

1. Filter F-2 is supported in a bracket that slides into a welded mount on the front of T-1. Once in place, this piece simply hangs from Tank T-1 as seen in 5.1. Take care not to damage pressure transducers P-3 and P-5 when bringing this piece into the glove box.
2. Connect Con 4
3. Connect Con 10

Figure 6.1 - F-2 mounted on the front of T-1

Figure 6.2 - Connect Con 4 and Con 10

7) **Pump J-1:**

1. *Place Pump J-1 in the space directly between the right of the F-3 apparatus and the left of the T-1 platform leg (see Figure 6.1). Position the pump so that the head faces the glove box window and it is even with the T-1 platform leg*
2. *Connect Con 2.*

Figure 7.1 - Position of pump J-1

Figure 7.2 - Connect Con 2

8) Heat Exchanger (C-1)

The heat exchanger C-1 should be placed in front of J-1 and lined-up with the pump head (see Figure 7.1). The port connector from C-1 should line-up with the female swage adapter on the pump head. **Do not make** this connection until the cleaning chamber has been added to the glove box.

Figure 8.1 - C-1 positioned in front of J-1

9) **Activated Carbon Filter (ACF) Housing, D-1:**

Insert the ACF housing through the entry port top first and place it on the right edge of the shelf (in front of the pair of gloves on the right). The ACF housing overhangs the edge of the shelf and has two legs in front that go to the floor. These support legs are attached to the housing inside the glove box one at a time.

The legs consist of a threaded rod covered with shrink tubing with a foot at one end and an extended nut at the other end, as shown in the close-up in Figure 9.1. Screw the nut down onto the threaded rod as far as it will go and then place it just under the front edge of the ACF housing.

Screw the connecting nut from the leg onto the threaded rod protruding from the bottom of the housing. The ACF housing should now be stable and in the correct position (see Figure 9.2).

4. *Connect Con 1 and Con 13 (see figure 9.3)*

Figure 9.1 - Close-up of ACF Housing Support Leg

Figure 9.2 - ACF housing on right end of shelf and supported by two legs

Figure 9.3 - Con 1 and 13

10) Electrical Cables

1. *Prior to the adding the cleaning chamber to the glove box, make sure the following cables are accessible:*
 - *W-3: Power Cable for TM-1*
 - *W-4: Signal Cable for TM-1*
 - *W-5: Power Cable for Pump J-1*
 - *W-8: Signal Cable for Pressure Transducer P-1*
 - *W-9: Signal Cable for Pressure Transducer P-2*
 - *W-10: Signal Cable for Pressure Transducer P-3*
 - *W-12: Signal Cable for Pressure Transducer P-5*
 - *W-13: Power Cable for AV-1*
 - *W-14: Power Cable for AV-2*
 - *W-16: Power Cable for AV-4*
 - *W-17: Thermistor TC-1*
 - *W-18: Power Cable for AV-5*

11) Cleaning Chamber/Cleaning Chamber Base Assembly:

It is significantly easier to install the cleaning chamber and cleaning chamber base as a single unit than to insert them separately and position the chamber on the base inside the glove box. For this reason, the cleaning chamber base is secured to the cleaning chamber by means of Velcro strips. There is a length of Velcro on the front and back of the base and chamber and a wider piece that covers both of these smaller strips. To support the chamber while positioning it, the liquid drain tube assembly (on the left) and the pulley wheel on the right of the chamber are used as "handles." These provide enough support to maneuver the chamber once inside the glove box.

1. *First, insert the end of the chamber with the drain tube assembly.*
2. *Once the person receiving the chamber has enough of a grip to maneuver the chamber and set it down, they can slide the cleaning chamber into place. The drain tube assembly should be perpendicular to C-1 (see Figure 11.1).*
3. *When aligned correctly, C-1 should be able to slide easily into the female adapter on the J-1 pump head and the elbow of the drain tube assembly simultaneously.*
4. *Tighten the ¾" swage nuts to secure the assembly.*
5. *Connect Con 3, Con 9, and Con 11 (see Figure 11.2 and 11.3).*
6. *Connect Inlet and Outlet for water lines.*

Figure 11.1 - Positioning of the drain tube assembly with the C-1

Figure 11.2 - Connect Con 3

Figure 11.3 - Connect Con 9 and Con 11

12) Electrical Connections 1

At this point, it is important that all of the following electrical connections be made.

- *W-3: Power Cable for TM-1*
- *W-4: Signal Cable for TM-1*
- *W-5: Power Cable for Pump J-1*
- *W-8: Signal Cable for Pressure Transducer P-1*
- *W-9: Signal Cable for Pressure Transducer P-2*
- *W-10: Signal Cable for Pressure Transducer P-3*
- *W-12: Signal Cable for Pressure Transducer P-5*
- *W-13: Power Cable for AV-1*
- *W-14: Power Cable for AV-2*
- *W-16: Power Cable for AV-4*
- *W-17: Thermistor TC-1*
- *W-18: Power Cable for AV-5*

Lay the electrical cables out in the right corner so they do not interfere with other components in the glove box. These cables are attached to the bulkhead fittings that are on the right wall of the glove box.

Figure 12.1 - First set of complete electric connections: AV-1, P-3, TM-1

Figure 12.2 - First set of complete electric connections: AV-3, P-1, TC-1, P-3

Figure 12.3 - First set of complete electric connections: AV-2, J-1, TM-1, P-3

13) **Pump J-2:**

1. Pump J-2 is placed behind the cleaning chamber by the 10" x 12" door in the rear of the glove box (see Figure 13.1). The end of the suction line is heavy and, must be guided behind the cleaning chamber to where it will connect to the T-1 drain.
2. Connect Con 6, Con 7, and Con 8 (see Figures 13.2 and 13.3).

The only obstacle faced when installing this piece of equipment is making the connection of Con 6 directly behind the cleaning chamber.

Figure 13.1 - Pump J-2 positioned behind the spray chamber

Figure 13.2 - Connect Con 7 and Con 8

Figure 13.3 - Connect Con 6

14) Gear Motor:

1. Hang the drive belt from the pulley wheel located on the side of the spray chamber.
2. Insert the gear motor into the glove box.

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3. *Slide the metal dowel found on the bottom of the motor into the receiver on motor bracket (see Figure 12.1). Do not slide it in all the way at this time (because the belt guard must be attached first).*
4. *Insure that the belt is around the pulley wheel attached to the gear motor.*

Figure 14.1 - Gear motor positioned on the side of the cleaning chamber

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5. *Remove the wing nut from the side of the spray chamber just above the gear motor (see Figure 12.2).*
6. *Maneuver the guard into place and push the motor toward the spray chamber. When the guard is mounted correctly, it will rest on the top of the motor and the knob found near the pulley wheel will be in the cutout section of the guard (see Figure 12.2)*
7. *Tighten the screw at the base of the bracket so that the gear motor will not move.*

Figure 14.2 - Belt guard and gear motor secured.

15) Electrical Connections 2

Now that all of the equipment is in place in the glove box, mate the rest of the electrical connections. As indicated in Figure 13-1, the following electrical connections can now be made inside the glove box:

- *W-7: Power Cable for Gear Motor M-1*
- *W-6: Power Cable for J-2*
- *W-11: Signal Cable for Pressure Transducer P-4*
- *W-15: Power Cable for AV-3*
- *W-? : Power Cable for Ultrasonic Generator to the inside of the ultrasonic bulkhead fitting inside the glovebox*

Figure 15.1- Electrical connections 2

16) Attach External Cables

1. *Connect the power cable from the ultrasonic generator and that communicates with the ultrasonic transducers to the bulkhead fitting on the exterior of the glove box.*
2. *Connect the cable from the electrical control box that connects power lines to the bulkhead fitting on the exterior of the glove box.*
3. *Connect the cable from the electrical control box that connects signal lines to the bulkhead fitting on the exterior of the glove box. See figure 16.1*

17) Assembled System

At this point, the Pu-238 decontamination system is completely assembled inside the glove box (see Figure 17.1) and the system is connected to the PLC and PanelView that are outside the glove box. The system is now ready to be filled with process liquid.

Figure 17.1 - Pu-238 decontamination system completely assembled in a glove box

APPENDIX H BILL OF MATERIALS

Bill of Materials for DOE Pu-238 System

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<i>Cleaning Chamber</i>	<i>Description of Item</i>	<i>Number</i>	<i>Manufacturer or Supplier</i>	<i>Model Number</i>	<i>System Part (if applicable)</i>
Item 1	Steel cleaning chamber (18" x 12" x 10.25")	1	ADF	Custom	cleaning chamber
Item 2	Cleaning chamber base (18" x12" x 3.5")	1	ADF	Custom	cleaning chamber base
Item 3	Perforated steel cleaning chamber baskets (13.375" x 7" diameter)	8	ADF	Custom	Baskets
Item 4	Steel belt guard (8.5" x 4" x .125")	1	ADF	Custom	belt guard
Item 5	AC gear motor (9" x 7" x 6") with custom ADF steel bracket	1	Dayton	71901596	motor drive

Connections to Cleaning Chamber	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1 a	1/2" Quick Connect stem	1	Swagelok	SS-QC8-D-8PM	Con 3
Item 1 b	1/2" Quick Connect body	1	Swagelok	SS-QC8-B-8PM	Con 3
Item 2	1/2" PFA tubing, length: 20"	1	Swagelok	PFA-T8-062-50	3
Item 3	1/4" Street elbow	1			
Item 4	1/4" NPT x 3/8" swage	1			
Item 5	3/8" PFA tubing, length: 20"	1			
Item 6	1/4" PFA tubing, length: 27"	1	Swagelok	PFA-T4-062-100	11
Item 7	1" x 3/4" Reducing bushing	1	Cajon	SS-16-RB-12	
Item 8					
Item 9 a	3/8" Quick connect body	1			
Item 9 b	3/8" Quick connect stem	1			
Item 10 a	1/4" Quick Connect stem	1	Swagelok	SS-QC6-D-4PM	Con 11
Item 10 b	1/4" Quick Connect body	1	Swagelok	SS-QC6-B-4PM	Con 11
Item 11	1/2" Street tee	1	Swagelok	SS-8-ST	
Item 12	1/2" x 3/8" Reducing bushing	3	Cajon	SS-8-RB-6	
Item 13	3/8" Female elbow	1	Swagelok	SS-600-8-6	
Item 14 a	3/8" Quick Connect stem	1	Swagelok	SS-QC6-D-6PM	Con 9
Item 14 b	3/8" Quick Connect body	1	Swagelok	SS-QC6-B-6PM	Con 9
Item 15	3/8" PFA tubing, length: 29"	1	Swagelok	PFA-T6-062-50	9
Item 16	1/2" VCO male connector	1	Swagelok	SS-8-WVCO-1-8	
Item 17	1/2" VCO male connector	1	Swagelok	SS-8-VCO-1-8	
Item 18	1/2" Solenoid valve	1	Parker	7D100P3 41 1SHF7	AV-4
Item 19	3/8" Nylon male elbow	1	Swagelok	NY-600-2-6	
Item 20	1/4" Male branch tee	1	Swagelok	SS-400-3-4TTM	
Item 21	1/4" PFA male connector	1	Swagelok	PFA-420-1-4	
Item 22	Thermistor	1	ADF	custom	
Item 23	0-5 psi pressure transducer	1	Sensym Ict	SPT4V0005PG5W06	P-1
Item 24	Electrical quick connect 4 wire shielded	1	ADF	custom	Con P-1

Separation and Extraction of Plutonium in Mixed Waste

PUMP J-1	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Iwaki magnetic pump	1	Iwaki Walchem	MD-70RLZT	J-1
Item 2	Bracket (1" x 9.5" x 5.75")	1	ADF	custom	
Item 3	3/4" male connector	1	Swagelok	SS-1210-6-12WBT	
Item 4	3/4 x 1/2" Reducing bushing	1	Cajon	SS-12-RB-8	
Item 5	1/2" PFA male connector	2	Swagelok	PFA-820-1-8	

PUMP J-2	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Iwaki pump	1	Iwaki Walchem	WMD-30RLZT-115	J-2
Item 2	Bracket	1	ADF	Custom	
Item 3	3/4" x 1/2 Reducing adapter	1	Swagelok	SS-12-RD-8	
Item 4	1/2" Plastic nipple, length: 3"	1			
Item 5	Reducing cross 1/2" x 1/2" x 1/2" x 1/4"	1	ADF	custom	
Item 6	1/4" Union elbow	1	Cajon	SS-4-TSW-9	
Item 7	1/4" Snubber fitting	1	McMaster Carr	4034K58	
Item 8	Ashcroft 60 psi pressure transducer	1	McMaster Carr	31685K1	P-4
Item 9	Electrical quick connect 4 wire shielded	1	ADF	custom	Con P-4
Item 10 a	3/8" Quick Connect stem	2	Swagelok	SS-QC6-D-6PM	Con 7 + 8
Item 10 b	3/8" Quick Connect body	2	Swagelok	SS-QC6-B-6PM	Con 7 + 8
Item 11	3/8" PFA tubing, length 25" and 16"	2	Swagelok	PFA-T6-062-50	7 + 8

Separation and Extraction of Plutonium in Mixed Waste

<i>Turbidity Monitor</i>	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Turbidity monitor	1	HF Scientific, Inc.	Microtol turbidity meter: 20023	TM-1
	Bracket	1	Leo engineering	custom	
Item 2	Electrical quick connect 4 wire shielded	1	ADF	custom	Con TM-1
Item 2	1/4" Male connector	3	Swagelok	SS-400-1-4	
Item 3	1/4" SS tubing	2			
Item 4	1/4" Female elbow	1	Swagelok	SS-400-8-4	
Item 5	1/4" Needle valve	1	Whitey	SS-IRS4	

<i>Ultrasonic Generator</i>	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Multi-frequency ultrasonic generator, 720w, 208v	1	Blackstone Ney Ultrasonic	MultiSonik dualsweep, 40-72-104-170-MSG-18	Ultrasonic generator

Separation and Extraction of Plutonium in Mixed Waste

<i>Ceramic Filter</i>	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Laboratory scale module with 0.2 µm alpha-alumina membrane	1	Ceramem	LM-0200-A	F-3
Item 2	Filter housing in laboratory scale module	1	Ceramem	LH	F-3
Item 3	Bracket	1	ADF	custom	
Item 4	1" Sanitary clamp	2	Tri-clover	13MHM	
Item 5	3/4" x 1/2" Reducing bushing	1	Cajon	12-RB-8	
Item 6	1/2" SS short nipple	1			
Item 7	1/2" Female connector	1	Swagelok	SS-810-1-8	
Item 8	1/2" Elbow	1	Cajon	SS-8-E	
Item 9	1/2" Close nipple	1	Cajon	SS-8-CN	
Item 10	1/2" Solenoid valve	1	Parker	7D100P3 41 1SHF7	AV-2
Item 11	1/2" PFA male connector	1	Swagelok	PFA-820-1-8	
Item 12	1/2" PFA tubing, length: 23"	1	Swagelok	PFA-T8-062-50	3

<i>Primary Filter</i>	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Primary filter capsule, SS, 19 µm	1	Pall	Rigimesh: 7EHD-RK capsule	F-2
Item 2	Bracket	1	ADF	custom	
Item 3	3/4" x 1/2" Reducing bushing	2	Cajon	SS-12-RB-8	
Item 4	1/2" x 1/2" x 1/4" Reducing street elbow	1	ADF	custom	
Item 5	1/4" Snubber fitting	2	Swagelok	4034K58	
Item 6	Ashcroft 60 psi pressure transducer	2	McMaster Carr	31685K1	P-5 and P-3
Item 7	Electrical quick connect 4 wire shielded	2	ADF	custom	Con P-5/P-3
Item 8 a	1/2 Quick Connect stem	2	Swagelok	SS-QC8-D-8PM	Con 4 + 2
Item 8 b	1/2 Quick Connect body	2	Swagelok	SS-QC8-B-8PM	Con 4 + 2
Item 9	1/2" PFA union elbow	2	Swagelok	PFA-820-9	
Item 10	1/2" PFA tubing, lengths 1, 4, 7.5, and 23"	4	Swagelok	PFA-T8-062-50	4a, 4b, + 4c
Item 11	1/2" Tee	1	ADF	custom	

Separation and Extraction of Plutonium in Mixed Waste

Primary Filter	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 12	Reducing cross 1/2" x 1/2" x 1/2" x 1/4"	1	Swagelok	custom	
Item 13	1/2" Solenoid valve	1	Parker	7D100P3 41 1SHF7	AV-1
Item 14	1/2" Street elbow	1	Swagelok	SS-8-SE	
Item 15	1/2" PFA male connector	1	Swagelok	PFA-820-1-8	
Item 16	1/4" Street elbow	1	Swagelok	SS-4-SE	
Item 17 a	1/4" Quick connect stem	1	Swagelok	SS-QC4-D-4PM	Con 10
Item 17 b	1/4" Quick connect body	1	Swagelok	SS-QC4-B-4PM	Con 10
Item 18	1/4" PFA tubing, length: 29"	1	Swagelok	PFA-T4-062-100	10

Storage Tank	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Storage tank 15.5" x 13" x 12"	1	ADF	custom	T-1
Item 2 a	3/8" Quick Connect stem	1	Swagelok	SS-QC4-D-4PM	Con 1
Item 2 b	3/8" Quick Connect body	1	Swagelok	SS-QC4-B-4PM	Con 1
Item 3	1/4" Street elbow	2	Swagelok	SS-4-SE	
Item 4	1/2" Street elbow	1	Swagelok	SS-8-SE	
Item 5 a	1/2" Quick Connect stem	1	Swagelok	SS-QC8-D-8PM	Con 5
Item 5 b	1/2" Quick Connect body	1	Swagelok	SS-QC8-B-8PM	Con 5
Item 6	1/2" PFA tubing, length: 23"	1	Swagelok	PFA-T8-062-50	5
Item 7	1/4" Manual valve	1			
Item 8	1/4" Street tee	1	Swagelok	SS-4-ST	
Item 9	0-5 psi pressure transducer	1	Sensym Ict	SPT4V0005PG5W06	P-2
Item 10	Electrical quick connect 4 wire shielded	1	ADF	custom	Con P-2
Item 11	1/2" Nipple, length: 3"	1	McMaster Carr	4830K175	
Item 12	1/2" Elbow	1	Swagelok	custom	
Item 13 a	1/2" Quick Connect stem	1	Swagelok	SS-QC8-D-8PM	Con 6
Item 13 b	1/2" Quick Connect body	1	Swagelok	SS-QC8-B-8PM	Con 6
Item 14	1/2" Manual ball valve	1	McMaster Carr	46495K61	
Item 15	1/2" PFA tubing, length: 20"	1	McMaster Carr	PFA-T8-062-50	6
Item 16	3/4" x 1/2" Reducing bushing	1	Cajon	SS-12-RB-8	

Adsorption Chamber	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Carbon adsorption chamber (12.06" x 10.5" diameter)	1	Leo Engineering	custom	ACF Housing
Item 2	Mandrel wrapped with carbon adsorption felt	1	ESI	custom	
Item 3	3/4" Male connector	2	Swagelok	B-1210-1-12	
Item 4	3/4" Female swage elbow	1	Swagelok	SS-12108-12	
Item 5	3/4" x 3/8" Reducing bushing	1	Cajon	SS-12-RB-6	
Item 6	3/8" Male connector	3	Swagelok	B-600-6-1	
Item 7	3/8" SS tubing, lengths: .5" and 1"	2			
Item 8	3/8" Male swage elbow	1	Swagelok	SS-600-2-6	
Item 9	1/4" SS Tubing, lengths: 1" and 1.25"	2			
	1/4" L series metering valve	1	Cajon	SS-4L	
Item 10	3/8" x 1/4" Reducer	2	Swagelok	SS400-R-6	
Item 11	1/2" Solenoid valve	1	Parker	7D100P3 41 1SHF7	AV-3
Item 12	1/2" x 3/8" Reducing bushing	1	Cajon	SS-8-RB-6	
Item 13	3/8" Quick connect body	1	Swagelok	SS-QC6-B-6PM	Con 12
Item 15	1/4" Close nipple	1	Swagelok	B-4-CN	
Item 16	1/4" Cross	1	Swagelok	SS-4-CS	
Item 17	1/4" 0-15 psig pressure gauge	1			
Item 18	1/4" CPA adjustable check valve	1	Swagelok	SS-4 CPA 2-3	
Item 19	1/4" Purge valve	1	Swagelok	SS-4P-4M	
Item 20	1/4" Female branch tee	1	Swagelok	SS-600-3TTF	
Item 21	Support legs with feet	2	McMaster Carr		
Item 22	Short connecting nuts	10			
Item 23	Long connecting nuts	2			
Item 24	1/2" Shrink fit PE tubing, length: 9.5"	8	McMaster Carr	7864K35	

Separation and Extraction of Plutonium in Mixed Waste

Heat Exchanger	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1	Stainless Steel Brazed Plate Heat Exchanger, 8"x3"x1-11/16"	1	McMaster Carr	35115K21	C-1
Item 2	3/4" Female swage elbow	3	Swagelok		
Item 3	3/4" port connector	2	Swagelok		
Item 4	3/4" Close nipple	1	Cajon	SS-12-HN	
Item 5	3/4" x 1/2" Female adapter	1	Swagelok	SS-12-TA-7-8	
Item 6	1/2" Close nipple	1	Swagelok		
Item 7	1/2" Solenoid valve	1	Parker		
Item 8	1/2" x 1/4" Hex bushing	1	McMaster Carr	48805K527	
Item 9	3/4" x 1/4 Female adapter	1			
	1/4" Hex coupling	1	Swagelok	SS-4-HCG	
Item 10 a	3/8" Quick connect body NPT	2	Swagelok	SS-QC8-B-8PM	
Item 10 b	3/8" Quick connect stem	2	Swagelok		

Draining Tubes et al.	Description of Item	Number	Manufacturer or Supplier	Model Number	System Part (if applicable)
Item 1 a	1/2" Quick connect stem	1	Swagelok	SS-QC8-D-8PM	5a
Item 1 b	1/2" PFA tubing	1	Swagelok	PFA-T8-062-50	5a
Item 2 a	3/8" Quick connect stem	1	Swagelok	SS-QC6-D-6PM	12a
Item 2 b	3/8" PFA tubing	1	Swagelok	PFA-T8-062-50	12a
Item 3 a	1/4" Quick connect stem	1	Swagelok	SS-QC4-D-4PM	10a
Item 3 b	1/4" PFA tubing	1	Swagelok	PFA-T8-062-50	10a
Item 4	Thermometer, 0-150 °C	1			

APPENDIX I ELECTRICAL WIRING

I-1 Electrical Wiring

I-2 Bulkhead Connectors and Cables

I-1 Electrical Schematic – Patrick Cassidy, Auto CAD – hardcopy (not in bytes)**I-2 Bulkhead Connectors and Cables** – Cut Sheets – hardcopy (not in bytes)**APPENDIX J PANELVIEW PROCEDURE****TITLE SCREEN**

- 1) Touch the screen to continue
- 2) Enter Password
 - a) For general entry and running of system: **6007**
 - b) For programming, general entry and running of system: **3791**
- 3) Press enter

Entering 6007 allows access to the Process Interface Screen. Entering 3791 allows access to the following three screens:

- 1) Process Interface Screen
- 2) Programming Screen
- 3) Maintenance Screen

Note: Selections can be made from the active blue window with the use of the up and down arrows. When the white triangle is to the left of the desired selection it can be accessed by pressing the enter button.

Process Interface Screen – This is the central screen of the program and from this location other parts of the program and outputs can be monitored while running the system. The screen displays the time and date as well as the process time and the option to stop the present cycle. There are six exit choices on this screen.

- 1) Title Screen
- 2) Programming Screen
- 3) Maintenance Screen
- 4) Pressure Sensors
- 5) Turbidity Meter
- 6) Output Status

Programming Screen – To access this screen, the password 3791 must be entered once again. Here the setting for the individual system steps and alarm settings are programmed. To exit this screen, select the Process Interface Screen.

Settings

- 1) Degas Settings
- 2) Step 1 time
- 3) Step 2 time
- 4) Step 3- Turned off
- 5) Step 4 time
- 6) Step 5a time
- 7) Step 5b time
- 8) Step 6 time
- 9) Step 7 time
- 10) T-1 Level settings
- 11) F-1 Alarm settings
- 12) F-2 Alarm settings
- 13) F-3 Alarm settings

The system is provided with the following preprogrammed operating protocol:

- 1) **Step 1**
 - a) P-1 0.06 psig
- 2) **Step 2**
 - a) Subset A 30 seconds
 - b) Subset B 30 seconds
 - c) 10 NTUs
- 3) **Step 3**
 - a) Turned off
- 4) **Step 4**
 - a) P-1 0.215 psig
- 5) **Step 5a**
 - a) 40 kHz 300 seconds.....100% Duty Cycle
 - b) 72 kHz..... 30 seconds.....100% Duty Cycle
 - c) 104 kHz..... 450 seconds.....100% Duty Cycle
 - d) 170 kHz..... 30 seconds.....100% Duty Cycle
- 6) **Degas Settings (Visible at top of list after Step 5 a has been selected)**
 - a) Degas ON 0 seconds
 - b) Degas OFF 0 seconds
 - c) Degas total 0 seconds
 - d) Duty Cycle 50%
- 7) **Step 5b**
 - a) Subset A 180 seconds
 - b) Subset B 30 seconds
 - c) 10.00 NTUs
- 8) **Step 6**
 - a) 10.00 NTUs
 - b) Repeat Steps 1-6, 2 Times

9) Step 7

- a) P-1 0.06 psig
- b) 250 sec

10) T-1 Level settings

- a) 0.100 psig

11) F-1 Alarm settings

- a) 50 psi

12) F-2 Alarm settings

- a) First 10 psig Second 15 psig

13) F-3 Alarm settings

- a) 50 psig

Maintenance Screen- This screen allows individual components to be turned on and off for diagnostic purposes.

- 1) Gear motor normal speed.....On/Off
- 2) Gear motor slow speedOn/Off
- 3) J-1 Pump.....On/Off
- 4) J-2 Pump.....On/Off
- 5) AV-1 ValveOn/Off
- 6) AV-2 ValveOn/Off
- 7) AV-3 ValveOn/Off
- 8) AV-4 ValveOn/Off

The Maintenance Screen exit options are:

- 1) Title Screen
- 2) Process Interface Screen
- 3) Programming Screen
- 4) Ultrasonic Maintenance

Ultrasonic Maintenance - Ultrasonic generator settings can be altered from this screen.

- 1) GeneratorOn/Off
- 2) High/Low Screen Box 1
- 3) High/Low Screen Box 2
- 4) DegasOn/Off
- 5) Duty cycle 10-100% by increments of 5%

Note: By selecting different options of High/Low any of the 4 US frequencies can be selected.

- 1) High/High = 170 kHz
- 2) High/Low = 104 kHz
- 3) Low/High = 72 kHz
- 4) Low/Low = 40 kHz

APPENDIX K STARTUP PROCEDURE

Equipment Installation

Installation of the system is described in detail in the installation procedure document that is presented as Appendix G. A video of the installation procedure is available for users.

Filling with Sonatol® Liquid

1. Transfer perfluoroheptane (Sonatol ®70) in one-gallon bottles to glove box. The system holds approximately 11 gallons (42 liters) of liquid.
2. Pour the contents of four one-gallon bottles of Sonatol®70 into the empty Cleaning Chamber.
3. Go to Maintenance Screen on Panel View.
4. Transfer liquid from Cleaning Chamber to Tank T-1 by opening valve AV-2 and turning on pump J-1.
5. Turn off J-1 and close AV-2 when the cleaning chamber is nearly empty (if all the liquid is transferred the pump will cavitate). Draining the Cleaning Chamber should take approximately 2 and a half minutes.
6. Transfer liquid from tank T-1 to adsorber, D-1 by opening AV-3 and turning on pump J-2.
7. While D-1 is filling, MV-3 should be open to vent the accumulated air out of D-1.
8. When there is a release of liquid through MV-2, close MV-2, and subsequently vent filter F-4 located on line 9. This is accomplished by turning the vent on the top of the filter counter-clock wise until all the air is released and then closing it by turning it clock-wise until it is tight. Then shut off pump J-2 and close AV-3. The liquid hold-up in D-1 is approximately 8.0 liters. At the desired flow rate of 1.7 lpm (set by control valve CV-1), it takes slightly less than 5 minutes to fill D-1.
9. Pour the contents of four more one-gallon bottles of 0.4 wt-% Sonatol® surfactant solution into the empty Cleaning Chamber.
10. Transfer liquid from Cleaning Chamber to Tank T-1 by opening valve AV-2 and turning on pump J-1, as in step 4 above.
11. Examine the discharge point of the Turbidity Meter in the cleaning chamber, and assure that there is liquid flowing through the instrument, and that there is a low value for the turbidity reading (1 NTUs or less).
12. Turn off J-1 and close AV-2 when the Cleaning Chamber is empty, as in step 5 above.
13. Go to the Pressure output display screen. If the reading for P-2 is less than 0.50 psi, add another quart (1 liter) of 0.4 wt-% Sonatol® surfactant to the Cleaning Chamber, and transfer this liquid to T-1, as in steps 4 and 5. Continue adding 0.4 wt-%

Sonitol® surfactant to the Cleaning Chamber and transferring it to T-1 until the pressure reading on P-2 reaches 0.50 psi to 0.55 psi.

14. Assure that all components are in their closed or off position, and exit maintenance screen.

6.3.2.3 Blank Run

1. Place empty basket in cleaning chamber.
2. Go to Process Interface Screen
3. Push program start button
4. Observe program operations to ascertain that the system is operating properly.
5. If, at the end of the blank cycle, no anomalies were observed, starting cleaning trials. Correct any detected anomalies, as required.

APPENDIX L OPERATING PROCEDURE

Before operating the system the following series of steps must be completed:

1. Inspect system to insure that all tubing and electrical connections are complete.
2. Turn on power to PanelView.
3. Turn on power to the PLC and electrical box.
4. If this is the first time the system is being turned on, follow the procedure outlined in section 3.6.2, and add the Sonitol® solution.
5. If this is not the first time the system is being turned on, check if more liquid must be added to D-1 and F-4, and whether they need to be cleared of air. Open AV-3 and turn on J-2 from the PanelView. Then open MV-1 and allow the solution from T-1 to fill D-1. This should take less than a minute (the time will depend on how long the system was left inactive). When there is a release of liquid through MV-1, close MV-1, and subsequently vent filter F-4 located on line 9. This is accomplished by turning the vent on the top of the filter counter-clock wise until all the air is released and then closing it by turning it clock-wise until it is tight. Then shut off pump J-2 and close AV-3.
6. From the PanelView Process Interface Screen, access the Pressure Sensor Screen. The pressure sensors should be approximately:

SENSOR NUMBER	PRESSURE
P-1	0
P-2	> 0.5 psig
P-3	0
P-4	~1 psig
P-5	0

7. From the PanelView Process Interface Screen, access the Turbidity Screen. The turbidity should be close to 1 NTU.

8. Insure that MV-1 and CV-1 are fully open.
9. From the PanelView Maintenance Screen, open AV-4 and turn on J-2. After the Cleaning Chamber has been filling for a minute or two, turn off J-2 and close AV-4. Now open AV-2 and turn on J-1. This should return the liquid to T-1. While it is draining, look at the rear left corner of the cleaning chamber. There should be a slow trickle of liquid returning from TM-1 (Not through the low pressure nozzles but in the far rear corner). If this is not the case, adjust CV-2 slowly until there is visible slow movement of liquid going into the cleaning chamber.

6.3.3.2 Operating Steps (Protocol IIa) (see Table 6.3.3.2-1)

- Step 0:** The cleaning chamber basket is loaded with contaminated material and placed on the rollers in the cleaning chamber. Then the pre-measured 55 grams of Krytox 157FS surfactant is slowly poured over the contaminated material so that it is evenly coated.
- Step 1:** The basket rotates as the cleaning chamber is filled to 0.06 psig.
- Step 2:** The objective of this step is to circulate the liquid in the cleaning chamber, thus distributing the Krytox.
- Step 3:** Inoperative - deleted from the system
- Step 4:** The cleaning chamber now continuous to rotate while being filled to 0.215 psig. This is the normal operating liquid level and entirely submerges the cleaning basket.
- Step 5a:** This is the sonication step. The initial 2 minutes of the 40 KHz sonication is used to degas the liquid and prepare it for the ultrasonics. The rest of the ultrasonic cycle removes particles for the contaminated material.
- Step 5b:** The objective of this step is to cool the liquid through the heat exchanger.
- Step 6:** The liquid is drained and all of the particles in solution are removed and captured in the filters.

Return to Step 1:

By returning to step 1, the cleaning process is now repeated with new Sonatol® solution. After steps 1 through 6 are repeated the cycle continues with the final rinse.

Step 7: The final rinse fills the cleaning chamber to 0.06 psig while rotating the basket. The final rinse is surfactant free and used to remove surfactant and any additional particles from the material.

Step 8: This is the final drain step and the basket rotates as all the liquid is removed from the cleaning chamber.

Step 9: This is the final step of the cycle and the cleaning chamber lid can be opened and the cleaning basket removed from the cleaning chamber.

Table 6.3.3.2-1 Chart of Process Protocol IIa

Step No.	Step	Step Time	Total Process
		min	Time, min
0	Load Basket in Chamber	3.00	3.00
1	Fill to 0.06 psig	1.17	4.17
2	Spray Part a	0.50	4.67
	Spray part b	0.50	5.17
3	Deleted	0.00	5.17
4	Fill basket to 0.21 psig	2.00	7.17
5a	Sonicate @ 40 kHz	5.00	12.17
	Sonicate @ 72 kHz	0.50	12.67
	Sonicate @ 104 kHz	7.50	20.17
	Sonicate @ 170 kHz	0.50	20.67
5b	Circulate a	3.00	23.67
	Circulate b	1.00	24.67
6	Drain	2.75	27.42
	<i>Return to Step 1</i>		
1	Fill to 0.06 psig	1.17	28.58
2	Spray Part a	0.50	29.08
	Spray part b	0.50	29.58
4(2)	Fill basket to 0.21 psig	3.17	32.75
5a(2)	Sonicate @ 40 kHz	5.00	37.75
	Sonicate @ 72 kHz	0.50	38.25
	Sonicate @ 104 kHz	7.50	45.75
	Sonicate @ 170 kHz	0.50	46.25
5b(2)	Circulate a	3.00	49.25
	Circulate b	1.00	50.25

Step No.	Step	Step Time	Total Process
6(2)	Drain	2.75	53.00
	<i>Final Rinse</i>		
7	Fill with rinse	4.00	57.00
8	Drain	1.25	58.25
9	Unload	2.00	60.25

APPENDIX M PARAMETRIC TESTING

APPENDIX M - PARAMETRIC CLEANING TESTS

H-1 Introduction

The parametric tests focused on determining the optimal conditions for decontamination. Table H-1 presents the parametric test matrix of the main variables examined.

Table H-5. Parametric Test Matrix

Number of Runs	Title	Basket	Bath Chemistry	Substrates	Means of Applying Shear	Ultrasonic Frequency (kHz)	Temperature
4	Spray Tests	ADF	Neat Sonatol A70	Aluminum and Plastic	Spray	N/A	Room (70°F - 85°F)
4	Spray Tests	ADF	0.4% Krytox	Aluminum and Plastic	Spray	N/A	Room (70°F - 85°F)
16	Initial Ultrasonic Tests	ADF	Neat Sonatol A70	Aluminum and Plastic	Ultrasonic	40 - 170	Varied
16	Initial Ultrasonic Tests	ADF	0.4% Krytox	Aluminum and Plastic	Ultrasonic	40 - 170	Varied
6	Baffle Tests	ADF	0.4% Krytox	Plastic	Ultrasonic	104	Varied

8	Material Interference Tests	Varied	0.4% Krytox	Plastic	Ultrasonic	104	Room (70°F - 85°F)
1	Tests with Improved Basket	Rotating Basket 2	0.4% Krytox	Mixed Waste Lot	Ultrasonic	104	Room (70°F - 85°F)

The main variables examined were the bath chemistry and temperature, means of applying shear, basket design, and coupon agitation. Tests performed in neat Sonatol A 70 showed little or no contaminant removal, while those in 0.4% Krytox solutions showed significant removal under most test conditions. Ultrasonic tests far and away outperformed spray tests with the highest level of decontamination occurring at 104 kHz. A lighter basket design with a greater percent open area caused less ultrasonic interference and resulted in significantly better decontamination than heavier baskets with less open area. Rotation of the basket increased coupon movement and coincided with better decontamination. There was little effect of temperature over the range of 70° F (21°C) to 120°F (50°C).

H-2 SPRAY TESTS:

Pressure spray tests were performed on both aluminum and plastic coupons (6-mil thick black polyethylene) to determine the effectiveness of pressure spraying on particle removal. These tests were performed in the ADF basket at room temperature for a period of 5 minutes. The results of the spray tests are listed in Table H-2

Spray Tests:		Neat Sonatol A70				0.4% Krytox Solution			
Duration is 5 minutes for all listed trials.									
ADF Basket used for all listed trials.		Blank	Cont.	Clean		Blank	Cont.	Clean	
Normal Liquid Level used for all listed trials.		GPL/A	GPL/A	GPL/A	D.F.	GPL/A	GPL/A	GPL/A	D.F.
Room Temperature used for all listed trials.									
Test	Substrate								
Just Aluminum	Aluminum	150	1502	1301	1.17	159	1873	981	2.09
Just Plastic	Plastic	150	235	248	0.87	102	1671	825	2.17
Mixture	Aluminum	150	1799	1462	1.26	154	1950	867	2.52
	Plastic	150	408	385	1.1	102	1661	671	2.74

Table H-2 Spray Tests Results

The spray test results show ineffective contaminant removal on the coupons sprayed in neat Sonatol A 70 for both plastic and aluminum. The spray tests in 0.4% Krytox solution resulted in somewhat better removal, with approximate decon factors of about 2.5.

H-3.3 ULTRASONIC TESTS:

Ultrasonic agitation was used in all the subsequent parametric tests as the means of applying shear for contaminant removal. Ultrasonic tests were performed on both aluminum and plastic coupons to determine the effectiveness of different ultrasonic frequencies on particle removal. Other variables considered were the bath chemistry and temperature. Aluminum and plastic tests were performed separately with 10 coupons processed in each trial.

H-3.3.1 Initial Test Series with ADF Basket:

The ADF basket tests were performed in the ADF basket that came with the system.. The ADF basket is a 0.060" thick perforated steel cylinder, 6" in diameter, 13" long, with 3/16" hole diameter and 51% open area. The ends are constructed from solid 0.060" thick stainless steel, as shown in Figure H-1. It weighs about 6.6 lb. (3.0 kg).

Figure H-.27. ADF Basket

Cleaning tests were performed with both aluminum and plastic coupons, with neat Sonatol A 70 and with the 0.4% Krytox solution, at each of the four ultrasonic frequencies that can be generated in the ultrasonic bath. The duration of each test was 5 min.

The test results for aluminum coupons are presented in Figure H-

.2.

Figure H-.2. Aluminum Ultrasonic Test Results

The results show clearly that all tests performed in 0.4% Krytox solutions resulted in much better decontamination than those in neat Sonatol A 70. The highest decon factor, 81, occurred with 0.4% Krytox solution at 104 kHz and at a high temperature. The corresponding test performed in neat Sonatol A 70 resulted in a decon factor of 11. Figure H-2.3 compares representative images from these tests.

With Krytox

Without Krytox

Figure H-3. Cleaned Aluminum Coupons – 104 kHz – High Temperature

The results also show clearly that 104 kHz is the optimal ultrasonic frequency for removing the contaminant for all bath conditions tested. At the lower frequencies, 40 kHz and 72 kHz, the effect of temperature is minimal on particle removal, but at the higher frequencies, 104 kHz and 170 kHz, sonication at higher temperatures coincided with higher decon factors.

The test results for plastic coupons are presented in Figure H-4.

Figure H-4. Plastic Ultrasonic Test Results

The striking difference between the aluminum and plastic results is the performance level. With aluminum, it was not unusual to obtain decon factors upwards of 40 under a variety of operating conditions. With plastic, the highest decon factor observed was 8.5, for coupons sonicated at 104 kHz in 0.4% Krytox solution at room temperature. Secondly, the plastic test results confirm that Krytox is essential for particle removal. All tests in neat Sonatol A 70 showed negligible contaminant removal with decon factors near 1. Figure H-5 shows representative images of plastic test coupons.

Figure H-5. Processed Plastic Coupons – 104 kHz – Room Temperature

The plastic ultrasonic tests confirm that 104 kHz is the optimal frequency for removing the contaminant for all bath conditions tested. Temperature had little effect on plastic decontamination aside from marginally better cleaning occurring at room temperature for coupons sonicated at 104 kHz in a 0.4% Krytox solution.

Besides attaining lower decon factors, plastic coupons were cleaned in a less uniform fashion than the aluminum coupons. Figure H-6 shows the ten processed coupons from the test performed in 0.4% Krytox solution at room temperature, for which an average decon factor of 8.5 was obtained.

Figure H-6. Variability of Processed Plastic Coupons

The variable cleaning of the plastic coupons is partially due to their “sticking” behavior. The plastic coupons tended to migrate together in the ultrasonic bath and ended up sticking together. Other plastic coupons also frequently stuck to the solid steel side walls of the ADF basket.

In an effort to solve the “sticking problem”, three equally spaced baffles were added to an ADF basket in an attempt to create more agitation and less coupon sticking. One basket was outfitted with 12 mesh screen angles, and another with solid $\frac{3}{4}$ ” brass angles as shown below in Figure H-7.

Figure H-7. ADF Basket with $\frac{3}{4}$ " Brass Angles

All baffle tests were run at 104 kHz in 0.4% Krytox solution and at varying temperatures and bath heights. The results of the baffle tests are presented in Table H-3.

Table H-3 Results of Baffle Tests

Duration is 5 minutes for all listed trials. 104 kHz frequency used for all listed trials. Plastic was used for all listed trials. 0.4% Krytox solutions used in all trials. ADF basket used for all listed trials.				Blank	Cont.	Clean	
Baffle Type	Liquid Level	Temp. °F		GPL/A	GPL/A	GPL/A	D.F.
none	high	97 - 101		118	719	195	7.8
screen	high	103 - 108		118	703	272	3.8
solid	high	112 - 117		118	747	205	7.2
solid	normal	96 - 100		118	645	186	7.8
solid	normal	111 - 113		118	604	267	3.3
solid	low	108 - 111		118	601	295	2.7

Visual observation of the rotating basket and its contents during the baffle tests confirmed that baffles indeed increased the agitation in the cleaning chamber, but this increased agitation did not coincide with increased cleaning performance. The baffles did increase coupon movement, eliminating “sticking” and decreasing clean coupon variability, as shown in Figure H-8. They may have, however, interfered with the ultrasonic wave pattern, leading to somewhat poorer overall cleaning, 7.2 decon factor versus 7.8 without baffles.

Figure H-8. Coupons with Tumbling Agitation

The test results also indicate that a higher liquid level, complete submersion of the basket, and higher temperatures promote better particle removal.

H-3.2 Effect of Materials on Ultrasonics:

The relatively poor results obtained in the ADF basket series, with and without baffles, raised questions as to whether the basket interfered with the ultrasonic waves, resulting in their damping. To address this issue, different sheets of material were submerged in the ultrasonic bath in order to examine the effects on the ultrasonic wave pattern. These observations are presented in Table H-4.

The materials test clearly shows that the ADF basket material has a large effect in damping the ultrasonic wave intensity. The test also revealed that lighter materials with greater open area have less of a damping effect on the ultrasonics than heavier materials with a lower open area. The test also showed that the opening in the basket material should be greater than 6 mm (1/4").

Table H-4. Effect of Materials on Ultrasonics

Material	Thickness in.	% Open Area	Visible Loss in Intensity		
			No Effect	Small Effect	Large Effect
Aluminum Sheet	0.016	0	X		
Aluminum Sheet	0.032	0	X		
Steel Foil	0.002	0	X		
Copper Sheet	0.060	0		X	
Perforated Steel	0.036	63		X	
Perforated Steel	0.060	51			X
2-Mesh Screen	0.047*	82.1	X		
3-Mesh Screen	0.042*	73.6	X		
4-Mesh Screen Welded	0.032*	76	X		
4-Mesh Screen	0.047*	65.9	X		
6-Mesh Screen	0.035*	62.7			X
10-Mesh Screen	0.025*	56.3			X
20-Mesh Screen	0.016*	46.2			X
60-Mesh Screen	0.0075*	30.5			X

* **FOR SCREEN MATERIAL, VALUE PRESENTED IS THE WIRE DIAMETER**

To confirm the suspected correlation between ultrasonic wave interference and poor cleaning, a series of material interference tests were performed with baskets of different material. The first test was to simply place the plastic coupons inside the ultrasonic bath, without any basket. Five coupons were placed inside the cleaning chamber and sonicated at 104 kHz. After five minutes three of the coupons were removed, and after ten minutes, the remaining two coupons were removed. The exact same test was performed in a 5 gallon Crest ultrasonic bath with 1 gallon of liquid at 132 kHz and 500 W for a comparison. Removal of the basket resulted in significantly higher decontamination factors, as indicated in Figure H-9.

Figure H-9 104 kHz Ultrasonics With No Basket

Figure H-9 presents the Net GPL/A, defined as the pixel intensity of coupon with contaminant less the pixel intensity of the blank coupon divided by the total area of interest, versus time. The results show better performance in the Crest bath, but more importantly decon factors greater than 20 and low coupon variability in the system bath. Figure H-10 shows the five coupons removed from the system bath.

Figure H-10 System Bath with No Basket

To confirm that the ADF basket was indeed interfering with ultrasonics another five-minute basket test was performed at 104 kHz in the system cleaning chamber, this time with no rotation of the basket. Figure H-11. shows representative coupons from this test that resulted in a decon factor of 1.8 confirming ultrasonic wave interference by the ADF basket.

Figure H-11. System Bath with ADF Basket – No Rotation

Two more material interference tests were performed in the system cleaning chamber with two different baskets made by Crest. These baskets were designed to be used in other cleaning baths in ESI's possession. The first Crest basket (basket 1) is made with a perforated steel bottom, 0.036" thick, with 5/32" hole diameter, and with 63% open area. The second Crest basket (basket 2) is made of 4-mesh screen. These baskets, shown in Figure 4.3.12, are much lighter and have a greater open area than the ADF basket. Five minute 104 kHz ultrasonic tests were performed on plastic coupons in both Crest baskets.

Crest Basket 1

Crest Basket 2*Figure H-.12 Crest Baskets 1 & 2*

Decontamination factors of 13.5 and 19.6 were obtained for Crest baskets 1 and 2 respectively, compared to a decon factor of 1.8 for the ADF basket under similar conditions. A final material interference test was performed with basket 2 to determine the apparent cleaning after ten minutes; clean coupons are in shown in Figure H-.13

Figure H-.13 Crest Basket 2 After Ten Minutes

A decon factor of 23.3 was achieved in ten minutes, but more importantly the variability among the clean coupons was very low. Table H-.5 illustrates the importance of basket design on the level and uniformity of cleaning, as endorsed by the decon factor, and most importantly in terms of variability, the standard deviation of the final net GPL/A values.

Table H-5. Static Material Interference Tests Statistics – System Bath

Basket	Time (min.)	Initial Net GPL/A	Final Net GPL/A	Decon Factor	Standard Deviation of Net Final GPL/A
None	5	466.7	22.8	20.5	4.7
None	10	466.7	23.0	20.3	7.1
ADF	5	499.5	277.5	1.8	143.7
Crest Basket 1	5	417.5	31.0	13.5	10.6
Crest Basket 2	5	501.9	25.6	19.6	6.7
Crest Basket 2	10	593.7	25.5	23.3	3.3

The results of the material interference tests, presented above, show clearly that the lighter and more open Crest baskets resulted in reduced ultrasonic wave interference, and thus higher decon factors, and less coupon variability.

The next series of material interference tests explored utilizing better agitation through basket rotation with the more open and lighter baskets. The first, rotating basket 1, is 4" diameter, 0.030" thick perforated steel cylinder, with 0.1875" hole diameter and 63% open area, 10 ¼" in length. The second, rotating basket 2, is a homemade modification of Crest basket 2. A lid constructed of 4-mesh screen was fastened to the top of Crest basket 2, and 6" diameter wheels were outfitted from paint can covers to allow rotation. Figure H-14 shows pictures of the rotating baskets.

Rotating Basket 1

Rotating Basket 2

Figure H-14 Rotating Baskets 1 & 2

Ten-minute 104 kHz ultrasonic tests were performed on plastic coupons in both rotating baskets. Decon factors of 33 and 60 were accomplished for rotating baskets 1 and 2 respectively. The basket rotation promoted increased coupons movement and thus subsequent increased decontamination level. The clean coupons for rotating basket 2 are shown in Figure H-2.15 below.

Figure H-15. Clean Coupons From Rotating Basket 2 (4-mesh)

These material interference tests confirmed the suspected correlation between ultrasonic wave interference and poor decontamination, and established the 4-mesh screen rotating basket as the optimal basket design.

H-3.3 Tests with Improved Basket:

Preliminary mixed waste trials were performed in rotating basket 2 (4-mesh). The mixed waste drawn from the list of material in Table 4.1.4 is outlined in Table H-6 below.

Table H-6. Preliminary Waste Composition

Item	Quantity	Item	Quantity
Tin Paint Can Cover	1	Stainless Steel Bushing	1
Aluminum Coupon	2	Stainless Steel Elbow	1
Black Polyethylene Coupon	2	Lined Neoprene Glove Tips	2
Rubber Apron Coupon	1	Lined Neoprene Glove Fingers	2
Clear Polyethylene Glove Tip	2	Yellow Rubber Glove Tips	2
Black Napkin Strip	2	Yellow Rubber Glove Fingers	2
Stainless Steel Union	1		

The mixed waste was sonicated at 104 kHz in rotating basket 2 for ten minutes at room temperature. Blank, contaminated, and clean images of selected items are presented in Figure H-16.

Figure H-16. Preliminary Mixed Waste Images

In all cases, the processed items were visibly cleaner than the entering blanks. Table H-7 confirms this quantitatively by presenting the GPL/A values for the blank, contaminated, and clean pieces that could be photodigitally analyzed.

Table H-7. Preliminary Waste Trial Statistics

	Sample	Blank GPL/A	Contaminat ed GPL/A	Clean GPL/A
Aluminum Coupon	1	127	1658	106
	2	147	1801	122
Black Polyethylene Coupon	1	90	668	87
	2	91	708	93
Stainless Steel Union	1	155	890	102
Stainless Steel Bushing	1	213	899	140
Stainless Steel Elbow	1	302	1957	198
Tin Paint Can Cover	1	130	1700	103
Rubber Apron Coupon	1	591	1471	498

In the above table, the GPL/A of the clean sample was lower than the GPL/A of the blank before contamination, except for the second set of black polyethylene coupons. For this set, a decontamination factor of 309 was calculated. For the other items, especially the metal fittings, the cleaning process not only removed the fluorescent contaminants that were applied, but also any prior surface contaminant that may have existed on blank items.

Initial blank pieces, such as the tin paint can cover and the stainless steel fittings, were pulled directly from the package such that minor surface residue may have existed (i.e. oil or particulates).