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# ABSTRACT

A proof-of-concept experimental study was performed to investigate the use of Orbit Technologies polysiloxane grouting material for encapsulation of U.S. Department of Energy mixed waste salts leading to a final waste form for disposal. Evaporator pond salt residues and other salt-like material contaminated with both radioactive isotopes and hazardous components are ubiquitous in the DOE complex and may exceed 250,000,000 kg of material. Current treatment involves mixing low waste percentages (less than 10% by mass salt) with cement or costly thermal treatment followed by cementation of the ash residue. The proposed technology involves simple mixing of the granular salt material (with relatively high waste loadings—greater than 50%) in a polysiloxane-based system that polymerizes to form a silicon-based polymer material. This study involved a mixing study to determine optimum waste loadings and compressive strengths of the resultant monoliths. Following the mixing study, durability testing was performed on promising waste forms. Leaching studies including the accelerated leach test and the toxicity characteristic leaching procedure were also performed on the promising waste forms. Department of Transportation oxidizer testing was performed on a high nitrate salt waste form. In addition to this testing, the waste form was examined by scanning electron microscope. Preliminary cost estimates for applying this technology to the DOE complex mixed waste salt problem is also given.

# EXECUTIVE SUMMARY

A proof-of-concept experimental study was performed to investigate the use of Orbit Technologies polysiloxane grouting material for encapsulation of U.S. Department of Energy (DOE) mixed waste salts leading to a final waste form for disposal. The DOE salts are basically evaporator pond material or other solid salt residuals that resulted from neutralizing acid-dissolving processes. The use of polysiloxane for encapsulating DOE salts is novel; however, it has been proposed that polysiloxane be used for helping fill void space in the sacoughagous at the Chernobly plant to eliminate subsidence. As part of this Chernobly work, polysiloxane has been shown to be highly resistant to chemical and radiation damage. Throughout the DOE complex there are stored over 254,000,000 kg of mixed waste salt material as part of the Weapons Complex operations. The present best available technology for stabilizing this waste results in a large volume increase (mixing with cements at extremely low waste loadings [10% by weight maximum loadings]). Another option for the salt materials is thermal treatment. These thermal techniques involve expensive and questionable off-gas systems (especially difficult for transuranic materials); therefore, application of a nonthermal, noncementatious technique has the potential to reduce cost and augment implementability.

The experimental study involved mixing the polysiloxane material with three different surrogate salt materials and performing a variety of leaching, compressive strength and durability testing on the final surrogate waste forms. The surrogate salts were modeled after several DOE complex salt materials including Pad-A salts from the Idaho National Engineering and Environmental Laboratory's Subsurface Disposal Area at the Radioactive Waste Management Complex and two generic surrogates from the Mixed Waste Focus Area (MWFA): one high nitrate and one high chloride with several heavy metals as contaminants. The surrogates did not have radioactive components; however, heavy metal components were included. Two different formulations of Orbit Technologies polysiloxane-based material were used called CSF#1 and CSF#2 (*CSF* stands for ceramic silicon foam) in this study. The study involved the following analysis of the waste forms: (1) a mixing study to optimize the waste loading (salt mass to polysiloxane mass) relative to basic compressive strength of the monolith, (2) laboratory hydraulic conductivity of the sample, (3) durability testing including wet/dry cycling, base immersion, water immersion, (4) leachability (toxicity characteristic leaching procedure [TCLP]), (5) Department of Transportation (DOT) oxidizer testing, and (6) evaluation of volatile organic content (zero head space testing) and finally the samples were subjected to a scanning electron microscope and evaluated for encapsulating properties. The following are specific results of the study. Two different formulations of the polysiloxane material are reported and are referred to in the report as CSF#1 and CSF#2.

It was determined that the Orbit Technologies polysiloxane mixture with Pad-A salts results in a cohesive monolith with compressive strength well in excess of the proposed 500 psi Nuclear Regulatory Commission (NRC) requirement for cemented waste. It was further found that the waste form polymerizes at room temperature and is essentially a nonisothermic process. During the mixing process, it was found that the polysiloxane has a high enough viscosity that waste particles remain suspended prior to curing. The final waste form remains somewhat elastic and therefore not prone to brittle fracture if subjected to earthquake during shallow land burial. A basic mixing study was conducted involving creating 2 x 4 in. cylindrical samples of the

surrogates/polysiloxane mixtures. A variety of mixtures were attempted up to 65% waste loading by weight. The American Society for Testing and Materials (ASTM) D-695 technique was applied to measure compressive strength. Five replicate tests were performed for each of the formulations with the result that CSF#1 had a compressive strength of 320 +/-23.7 psi and CSF#2 had a compressive strength greater than 637 psi for all five samples (637 psi was the maximum compressive strength that can be tested). For both the high chloride and high nitrate MWFA suggested material, monoliths with compressive strength higher than 637 psi could be formed at 30% surrogate waste and greater than 420 psi for surrogate waste at 50% waste loadings (the high chloride waste test showed >637 psi compressive strength for 50% waste loading).

A laboratory-scale study of the final waste form showed that one of the formulations was an order of magnitude less than the NRC hydraulic conductivity requirement of $1 \times 10^{-7}$ cm/s. A hydraulic conductivity test of the two formulations using ASTM D-5084 resulted in an average hydraulic conductivity for CSF#1 of $2.2 \times 10^{-6}$ cm/s and for CSF#2 of $6.4 \times 10^{-8}$ cm/s.

Durability testing showed that one of the Orbit Technologies formulations had compressive strengths that remained above the instrument detection limit when subjected to wet/dry cycling and 30-day water and base immersion. A variety of durability testing was performed to investigate the effects on compressive strength (using ASTM D-695) due to water immersion, wet/dry cycling, and base immersion. For the water immersion testing, material CSF#2 remained above the upper limit of the testing apparatus at 637 psi; however, for material CSF#1, there was sufficient deterioration to the point that the waste form was not tested for compressive strength. For the wet/dry cycling testing, material CSF#1 had up to a 10.5% mass loss after 6-48 hour wet/dry cycles; however, material CSF#2 showed only a 1.22% mass loss and again a compressive strength value above the maximum 637 psi for the apparatus. When subjected to base resistance (ph 12.5 for 30 days), material CSF#1 showed significant swelling and deterioration and therefore was not tested for compressive strength; however, material CSF#2 showed again a compressive strength above the maximum value obtainable of 637 psi.

The waste form created by mixing polysiloxane and salt showed excellent resistance during a variety of leach testing. At 30% waste loadings, the waste form involving CSF#2 material actually meets the current and proposed TCLP values of 5 ppm and 0.86 ppm, respectively. It is speculative at this time whether at 50% waste loadings the standard could be met without the addition of heavy metal scavengers. The Pad-A surrogate material had approximately 1045 ppm of the highly soluble $Cr^{+6}$ (1400 ppm potassium dichromate and 800 ppm chromium trioxide) and, when subjected to the accelerated leach test defined in ASTM C-1308, there was a nondetect for chromium in the leachate. The test was repeated at the University of Akron using the TCLP protocol, and the results were 1.36 ppm for the 30% waste loadings and 5.6 ppm for the 50% waste loadings. Additionally, samples of waste forms based on CSF#2 at both 30% and 50% loadings were evaluated by an independent laboratory (Ecology and Environment, Inc.) with 2.4 ppm and 5.9 ppm, respectively (basically the same values as the University of Akron). At 30% waste loadings, the waste form meets current TCLP requirements, and, when considering that the source term for $Cr^{+6}$ was approximately 5.8 times that found in the actual waste, it is almost certain that the waste form will also meet the proposed Universal Treatment Standard (UTS). It is speculative at this time whether the proposed UTS can be met for the 50% waste loadings; however, considering that the source term is higher for the surrogate than the actual Pad-A salts, suggests that the current standards can also be met (5 ppm); however, metal scavengers may be required at the higher waste loadings (greater than 30%) to meet the proposed UTS. The waste form generated by mixing the suggested MWFA surrogate and CSF#2 also passed TCLP for a variety of metals and were all below treatment standards. Examples of TCLP results for material CSF#2 at 50% waste loadings and the high chloride waste material are .17 ppm cadmium down from 1000 ppm cadmium oxide, 0.68 ppm chromium down from 1000 ppm chromium oxide, 0.01 ppm mercury down from 1000 ppm mercuric oxide, nondetect for lead down from 1000 ppm lead oxide.

A mixture of the Pad-A surrogate waste and polysiloxane material CSF#2 passed the DOT oxidizer test in that a mixture of sawdust and the waste form material did not burn after 20 minutes compared with the surrogate salt material alone which burned under similar conditions in 30 seconds. Testing was performed at Hark Laboratories, Inc. (Barberton, Ohio). The result is not surprising in that the polysiloxane material has less than 5% organic content.

A "zero head space" evaluation for trichloroethylene was made of the waste form created with the MWFA surrogate high chloride salt and CSF#2. The result was nondetect for trichloroethylene down from a source term in the surrogate of 1000 ppm.

Scanning electron microscope studies show that the simple mixing of polysiloxane material, catalyst, and salt causes a general encapsulation of the salt particles. There is some change (reduction) in the overall size of the salt particles following the encapsulation process suggesting some unknown chemical recombination as part of the polymerization process. The average size of the untreated salt particles are approximately 150 microns and when mixed with the polysiloxane the size appears to be the range of 50-100 microns with some particles in the original 150-micron range.

A preliminary cost estimate shows that the Orbit Technologies polysiloxane system has a cost savings over concrete of at least 3:1, with the added benefit of a superior resultant waste form. Basically, the cost of disposal per volume of waste form overwhelms the cost of encapsulating materials. Once there is a market for the base polysiloxane material, this cost savings could increase.

Overall, the process of mixing the salts with Orbit Technologies polysiloxane-based systems results in stable nonleachable waste forms suitable for disposal. The process of mixing uses only off-the-shelf equipment and has a curing process that is easily controllable. The final waste form can be poured into suitable shapes for shallow land burial. When considering the relatively high waste loadings (durability and leaching testing was performed up to 50% by mass) the process appears cost effective even though the base material may be as high as $8/lbm.

It is recommended that the retrieved Pad-A salts presently stored at the INEEL Waste Reduction Operations Complex be sent to the University of Akron for encapsulation, testing, and eventual disposal at a permitted facility as a demonstration of the whole process.

It is further recommended that the application of polysiloxane encapsulation be extended to other waste streams such as retrieved buried transuranic waste or alpha mixed low-level waste.

# CONTENTS

## FIGURES

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# ACRONYMS

| | |
|---|---|
| DOT | U.S. Department of Transportation |
| INEEL | Idaho National Engineering and Environmental Laboratory |
| MWFA | Mixed Waste Focus Area |
| ND | not detected |
| NRC | Nuclear Regulatory Commission |
| OST | Office of Science and Technology |
| RCRA | Resource Conservation and Recovery Act |
| RWMC | Radioactive Waste Management Complex |
| SEM | scanning electron microscope |
| TCE | trichloroethylene |
| TCLP | toxicity characteristic leaching procedure |
| TRU | transuranic |
| UTS | Universal Treatment Standard |

# Mixed Waste Salt Encapsulation Using Polysiloxane—Final Report

## 1. INTRODUCTION

This final report gives technical details of a proof-of-concept experiment for the Salt Encapsulation Project tasks associated with the U.S. Department of Energy (DOE) Office of Science and Technology (OST) Mixed Waste Focus Area (MWFA) Technical Task Plan TTP-ID-77MW41. The basic technology is to create a waste form suitable for shallow land disposal by mixing DOE complex salts with polysiloxane materials. Polysiloxane is a silicon dioxide-based grouting material that is nonthermal and nonhazardous and involves only off-the-shelf equipment for mixing with salts. In addition, the material contains less than 5% organic material. The polysiloxane binder material is applicable to a wide range of target problem salts including chloride, sulfate, and nitrate salts and can also be extended to calcined materials. Orbit Technologies is developing the material for use as a void filler in the Chernobly sarcoughagous. In prior studies, the material has been shown to be highly resistant to radiation damage up to 100 mR.[1]

The subject work focuses on encapsulating several surrogate waste materials including a special nitrate salt form found at the Idaho National Engineering and Environmental Laboratory (INEEL) and two generic salt surrogates suggested by the MWFA. The INEEL salt is a nitrate salt recovered from Pad A at the INEEL Subsurface Disposal Area. The salts are primarily potassium nitrate with the contaminant of concern chromium with only tenths of pCi/g of transuranic material. These salts were shipped to the INEEL from the Rocky Flats Plant and approximately 8,000 cubic meters of material is "stored" above ground with earthen cover on an asphalt pad: Pad A. The surrogate materials suggested by the MWFA are high nitrate salts and high chloride salts both containing several Resource Conservation and Recovery Act (RCRA) metals. Appendix A contains the composition of the Pad-A salt and of the MWFA suggested salts. This report describes testing performed on the three surrogate materials and gives an analysis of results. The testing involved mixing studies to obtain an optimum waste form, toxicity characteristic leaching procedure (TCLP) testing and U.S. Department of Transportation (DOT) oxidizer testing on the resultant waste form and a variety of durability tests on the waste form.

Polysiloxane systems have a variety of unique properties that make them attractive as potential waste encapsulation systems. They are extremely chemically resistant to normal solvents, acids, and bases in the appropriate formulations. They are an environmentally friendly system and use either very low or no toxicity materials in their formulations. The systems generally operate at ambient or slightly elevated temperatures and as such have no associated off-gas treatment processes. The system's viscosity can be modified from a very free-flowing liquid like water to an extremely viscous material like peanut butter, depending on the application. Set times can be controlled from a few seconds up to several days, depending on the catalyst used and the temperature of the materials undergoing curing. The actual curing process is very tolerant of a number of different chemicals in the system and does not undergo disruption with a large variety of different contaminants present. To the extent possible, all surrogates used in testing were technical-grade versus reagent-grade materials to more closely approximate the trace contaminants that would be expected in any typical waste form. The viscosity of the chosen systems were such that mixing was not unreasonably difficult, but the viscosity was sufficient to hold the contaminants in uniform distribution while curing took place. The processing technology to support the use of polysiloxanes is quite mature, and there is a large industrial base for conventional applications.

# 2. BACKGROUND

The MWFA Technology Development Requirements Document (INEEL/EXT-97-00315) addresses the need to improve upon salt stabilization by increasing the waste loading from the state-of-the-art amount of about 10% waste loadings using Portland cement as a binder to higher values using more innovative binders. In the requirements document, salt waste streams including chloride, sulfate, nitrate, and metal oxides/hydroxides are listed. In addition, the document addresses that the final waste form must conform to the Resource Conservation and Recovery Act (RCRA), including passing TCLP testing. Additionally, the waste form must not exhibit ignitability, corrosivity, or reactivity.

DOE has accumulated multiple tons of salts as part of the refinement of weapons materials (see Appendix B). These salts are in a dry granular form and are the by-product of solidifying neutralized acidic solutions used to recover and reformulate weapons materials. At the INEEL alone there is approximately 8,000 cubic meters of nitrate salts (potassium and sodium nitrate) stored above ground and buried in shallow land burial. The waste is from the Rocky Flats Plant. The INEEL has recovered a sample of this salt from an above ground storage area at the INEEL Radioactive Waste Management Complex (RWMC) called Pad A. Analysis of the Pad-A salt indicates sodium and potassium nitrate lightly contaminated with plutonium 238 and 239 and americium 241 at the tenths of pCi/g levels with the main contaminant of concern being soluble chromium compounds (elemental chromium at 404 ppm). Natural uranium (U-238) has been analyzed at 58 pCi/g.

Appendix A gives a detailed analysis of the Rocky Flats-generated nitrate salts recovered from Pad A. This Pad-A salt is thought to be representative of approximately 8,000 cubic meters of salt stored above ground at the RWMC at the INEEL. One of the options for final disposal of this mixed waste salt is to encapsulate it in a grout material that will isolate the contaminants from leaching for long-term disposal. The salts are in the dry granular form and are primarily stored in 55 gal containers. The proposed technique is mixing the salts with a proprietary patent-pending polysiloxane encapsulating agent that can pass a battery of leach and durability tests. The mixture of salts and polysiloxane would ultimately be poured into approximately 4 x 4 x 8 ft monoliths with lifting lugs and placed in shallow land burial with a top clay cap for permanent disposal.

# 3. TEST OBJECTIVES

Objectives for the Surrogate Waste Demonstration were (1) determine whether the polysiloxane binder can encapsulate the salt material and form a cohesive material suitable for disposal, (2) perform TCLP testing and DOT oxidizer testing on the final surrogate waste form, and (3) perform a variety of durability and compressive strength testing on the surrogate waste form. These tests are designed to help decision makers assess the long-term stability of the waste form and the suitability of the waste form for disposal or transportation to a disposal facility.

# 4. RESULTS

To meet the objectives, a variety of tests were conducted starting with a mixing study to determine the maximum waste loading to achieve a cohesive monolith (greater than 60 psi). Next, using promising combinations of waste loadings and polysiloxane binder, compressive strength, TCLP, and DOT oxidizer testing were performed. Concurrent with the above testing, durability testing was performed on the resultant waste form including effects on compressive strength caused by water immersion, base immersion, and wet-dry cycling. In addition, the hydraulic conductivity of the resulting waste form was determined and the matrix was examined with an electron microscope to establish the extent of microencapsulation. The complete procedure was performed on the Pad-A salt surrogate, and only the compressive strength, TCLP and DOT oxidizer testing were performed on the MWFA suggested high chloride and high nitrate salt surrogates listed in Appendix A. Appendix C gives an experimental equipment list.

## 4.1 Mixing Study

### 4.1.1 Pad-A Surrogate Waste

A mixing study resulted in two promising formulations of polysiloxane and surrogate salt waste both of which formed cohesive monoliths with up to 50% waste loadings. The two polysiloxane-based materials are labeled CSF#1 and CSF#2 and represent different base polysiloxane materials, the exact ingredients of which are proprietary. The objective of the mixing study was to determine the maximum waste loading that can be achieved and still obtain a cohesive cured product with at least 60 psi of compressive strength. The basic stabilized waste form consists of polysiloxane, polymer catalyst, and surrogate nitrate salt waste. Depending on the waste loading, the appropriate amount of polysiloxane, catalyst, and waste was measured to the nearest 0.1 g. Mixing of all samples took place in 1-gal plastic buckets using a hand-held drill with a stainless steel mixing paddle. The order of addition was polysiloxane, salt waste, and catalyst. The mixture was thoroughly stirred and then transferred to plastic test cylinders (usually 2 in. in diameter by 4 in. tall). The mixture was allowed to cure for at least 24 hours before compressive strength testing. The waste loading was varied from 30-90%.

### 4.1.2 MWFA Wastes

For both the high chloride and high nitrate surrogate material suggested by the MWFA, cohesive monoliths could be formed using at least 50% by mass mixture of polysiloxane and salt. Higher waste loadings were not attempted because of budget and schedule concerns. Based on experience with the two formulations and budget concerns, only the CSF#2 material was used for the MWFA surrogate materials mixing study.

## 4.2 Compressive Strength

### 4.2.1 Pad-A Surrogate

Compressive strength testing showed that much greater than 60 psi compressive strength could be achieved for both the CSF#1 and CSF#2 formulations at waste loadings as high as 50%.

A waste form's mechanical integrity and ability to withstand loading pressures in a disposal environment is directly related to compressive strength. Compressive strength is also used as a gauge of the resistance of samples that have undergone durability tests. Five replicate samples at 30% and 50% waste loading of both CSF#1 and 2 were tested for compressive strength. The Geotest Instrument load cell apparatus used for this study has a maximum load capacity of 2,000 lb, which results in a maximum compressive strength of 637 psi for a 2 in. diameter x 4 in. tall sample. Results for each material are summarized in Table 1. Although not shown on Table 1, CSF#1 was able to maintain a compressive strength greater than 60 psi up to 60% waste loading, and CSF#2 maintained a compressive strength greater than 60 psi up to 65% waste loading. The systems were similar to a hard rubber, with no permanent deformation of the samples.

**Table 1.** Compressive strength for CSF#1 and CSF#2.

| Material | Waste Loading | Compressive Strength (psi) |
|---|---|---|
| CSF#1 | 30% | 389.2 ± 25.2 |
| CSF#1 | 50% | 320.5 ± 43.7 |
| | | |
| CSF#2 | 30% | > 637 |
| CSF#2 | 50% | > 637 |

It is expected that the material could withstand significant shear such as that generated during an earthquake or differential settling during a shallow land burial application.

### 4.2.2 MWFA Surrogate

Compressive strength results using CSF#2 and both the high nitrate and high chloride wastes are shown in Table 2. Only the 50% waste loading high nitrate sample with CSF#2 had a compressive strength less than the target value of 500 psi.

**Table 2.** Compressive strength with high nitrate and high chloride wastes using CSF#2.

| Waste | Waste Loading | Compressive Strength (psi) |
|---|---|---|
| High Nitrate | 30% | > 637 |
| High Nitrate | 50% | 420.3 ± 28.4 |
| | | |
| High Chloride | 30% | > 637 |
| High Chloride | 50% | > 637 |

## 4.3 Hydraulic Conductivity

Hydraulic conductivity measurements of the final waste form for the CSF#1 and CSF#2 formulations mixed with the Pad-A surrogate showed that the CSF#1 formulation had an order of magnitude higher hydraulic conductivity than the NRC hydraulic conductivity requirement of

$1 \times 10^{-7}$ cm/s; however, the CSF#2 formulation mixed with the Pad-A surrogate resulted in a value one order of magnitude lower.

Hydraulic conductivity is a direct measurement of the potential ability of a waste form to leach out contaminants. Test samples (50% waste loading) were prepared using 3 in. diameter by 6 in. tall cylinders. According to ASTM D-5084, test samples were placed in a flexible wall permeameter, sealed with a latex membrane, and filled with de-aired water at a cell pressure of 5 psi. Once the cells were stabilized, the cell pressure was set at 96 psi, the lower cap at 93 psi, and the upper cap at 90 psi. A hydraulic gradient of 50 in./in. was used throughout the tests. Using a falling head constant tailwater method, the flow of water through the samples was recorded versus time using a graduated pipette, and the hydraulic conductivity calculated. The hydraulic conductivity for CSF#1 was $2.2 \pm 0.6 \times 10^{-6}$ cm/s and for CSF#2 was $6.4 \pm 1.6 \times 10^{-8}$ cm/s, respectively.

## 4.4 Water Immersion

For the mixture of Pad-A surrogate salt and the CSF#2 formulation, there was no visual signs of deterioration nor measurable reduction in compressive strength after water immersion testing. However, for the CSF#1 material there was enough deterioration that the compressive strength test was not performed.

Waste forms may be subjected to periodic exposure to aqueous solutions, which may cause significant changes in the wastes' structural integrity. Five samples (50% waste loading) were immersed in deionized water maintained at 20°C for 30 days. Samples were periodically removed and inspected for structural changes. The CSF#1 samples showed significant swelling and deterioration around the edges. CSF#2 showed no signs of deterioration and only minimal swelling. Since the CSF#1 samples had significant deterioration, they were not tested for compressive strength. The compressive strength for the CSF#2 samples, however, was measured to still be greater than 637 psi, exceeding the target value of 500 psi.

## 4.5 Wet/Dry Cycling

Wet/dry cycling testing had the same results as the water immersion testing in that the CSF#1 material failed and the CSF#2 material showed basically no measurable change in compressive strength and only a 1% change in total mass as a result of the cycling.

This set of tests was conducted to determine the effects of repeated wet and dry conditions on the integrity of the waste form. Three specimens for testing (50% waste loading) and three for control were selected for CSF Formulation #1 and #2, and subjected to wet/dry cycling according to ASTM D-4843. Each sample was machined to 1.73 in. in diameter by 2.91 in. tall, placed in a beaker, and covered. Test specimens were placed in an oven and subjected to 24 hours of drying at 60°C. Control samples were placed in a moisture chamber at 20°C for 24 hours. After 24 hours, all samples were removed and immersed in deionized water at 20°C for 24 hours.

Mass loss was determined for each sample after each interval. After the second cycle, the CSF#1 began exhibiting swelling and surface cracking, while CSF#2 remained unchanged. The average relative mass loss, after six cycles, for the CSF#1 was 10.49%, and 1.22% for the CSF#2 system. The CSF#1 broke apart upon simple compression (<60 psi), indicating significant loss in strength. CSF#2 samples had a measured compressive strength greater than 637 psi, exceeding the target value of 500 psi.

## 4.6 Base Resistance

The same basic trend in results occurred for base resistance as water immersion testing in that the CSF#2 material showed essentially no visual signs of deterioration and no measurable change in compressive strength while the CSF#1 material exhibited considerable change.

At waste sites, buried waste forms are expected to see alkali conditions that may cause significant changes in structural integrity. Five replicate samples (50% waste loading) of CSF#1 and 2 were immersed in an aqueous, sodium hydroxide solution at pH 12.5 for 30 days. The pH was checked daily and adjusted if it varied by ± 0.5 units. During the test period, samples were removed and inspected for cracking and swelling. After the 30-day test period, samples were subjected to compressive strength testing. The average change in length for CSF#1 was +5%, while change in diameter averaged +5%. The CSF#2 samples showed no sign of deterioration or swelling (<1%). Since CSF#1 samples exhibited deterioration and cracking, they were not tested for compressive strength. The maximum load recorded for CSF#2 was greater than 637 psi, exceeding the target value of 500 psi.

## 4.7 Leach Testing for Waste Forms

The waste forms (Pad A, MWFA high nitrate, MWFA high chloride) created by mixing surrogate salts with Orbit Technologies polysiloxane material showed excellent leach resistance for a variety of leach procedures including University of Akron "in-house" scoping testing and outside laboratory evaluation of TCLP. The following summarizes the leach study for both Pad-A salts and the MWFA suggested high nitrate salts and high chloride salts. The current standard for TCLP testing for chromium is 5 ppm in the leachate, and the proposed Universal Treatment Standard (UTS) is 0.86 ppm chromium in the leachate.

### 4.7.1 Accelerated Leach Test—"In-House" TCLP for Pad-A Salts Surrogate

For the Pad-A salt surrogate (see Appendix A for composition) material both accelerated leach testing and TCLP were performed at the University of Akron. The accelerated leach procedure was applied to minimize the amount of samples analyzed in the outside laboratory. Using an acidic sulfuric acid solution with a ph of 2 and the ASTM-C-1308 method, there was no $Cr^{+6}$ detected; however, when applying techniques more associated with TCLP, $Cr^{+6}$ values between 8.5-9 ppm (respectively for 30% and 50% waste loadings) was detected in the leachate for the CSF#1 material but only 1.56-5.6 ppm (respectively for 30% and 50% waste loadings) for the CSF#2 material.

Leachability is the ability of a waste form to retard the release of hazardous constituents to the environment. In accordance with ASTM C-1308, three replicate samples (50% waste loading) of CSF#1 and 2 were placed in beakers with the prepared leachant and covered. Samples were 1 in. in diameter by 1 in. tall, in order to maintain a 1:1 diameter-to-height ratio. The leachant volume for each interval was 100 cm times the surface area of the specimen at a temperature of 20°C, which corresponds to 3,000 mL of leachant. Leachant replacement took place at the recommended time intervals (2 hours, 5 hours, 17 hours, and each day up to a cumulative time of 11 days). When the samples were transferred to fresh leachant, the old leachant was stirred, acidified to pH 2 using 1.0 N sulfuric acid, and analyzed for $Cr^{+6}$ via spectrophotometer. The $Cr^{+6}$ levels measured were below detection limit for each interval. Therefore, the samples were analyzed for specific conductance via a conductivity probe to determine the relative amount of nitrate salts leached. Table 3 shows the cumulative fraction leached and leach rate for both CSF#1 and CSF#2.

Samples of both CSF#1 and 2 at variable waste loading were cut, placed in aqueous solution of sulfuric acid at pH 3, mass-to-liquid ratio of 1:20, and mixed in a shaker bath for 18 hours at 30 rpm. The leachant was sampled, acidified, and analyzed for $Cr^{+6}$ via spectrophotometer. The reported values are shown in Table 4.

**Table 3.** Cumulative fraction leached (CFL) and leach rate ($day^{-1}$) for CSF#1 and CSF#2 at 50% waste loading—Pad-A salts.

| Cumulative Time (days) | CFL CSF 97-1 | Leach Rate (day-1) CSF 97-1 | CFL CSF 97-2 | Leach Rate (day-1) CSF 97-2 |
|---|---|---|---|---|
| 0.1 | 0.14 | 1.58 | 0.09 | 1.12 |
| 0.3 | 0.37 | 1.04 | 0.30 | 1.04 |
| 1.0 | 0.92 | 0.72 | 0.86 | 0.77 |
| 2.0 | 0.96 | 0.04 | 0.89 | 0.03 |
| 3.0 | 0.97 | < 0.01 | 0.89 | < 0.01 |
| 4.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |
| 5.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |
| 6.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |
| 7.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |
| 8.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |
| 9.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |
| 10.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |
| 11.0 | 0.97 | < 0.01 | 0.92 | < 0.01 |

**Table 4.** $Cr^{+6}$ leaching with selected materials ("in-house" TCLP).

| Material | Waste Loading | $[Cr^{+6}]$ (ppm) |
|---|---|---|
| CSF#1 | 30% | 8.50 |
| CSF#1 | 50% | 9.70 |
| | | |
| CSF#2 | 30% | 1.36 |
| CSF#2 | 50% | 5.60 |

### 4.7.2 Outside Laboratory TCLP Results—Pad-A salts

Based on favorable results with CSF#2, stabilized Pad-A samples were sent to Ecology and Environment, Inc. (Lancaster, New York) for TCLP testing (USEPA SW-486). Table 5 shows the results for CSF#2 with 30% and 50% waste loading. Laboratory results reports are given in Appendix D.

**Table 5.** TCLP results with CSF#2.

| Material | Waste Loading | Chromium (ppm) |
|---|---|---|
| CSF#2 | 30% | 2.40* |
| CSF#2 | 50% | 5.90 |

* Average of two samples (2.3 and 2.5 ppm).

### 4.7.3 Outside Laboratory TCLP Evaluation of MWFA Surrogates

Samples of CSF#2 mixed with the MWFA high nitrate and high chloride salts were sent to Ecology and Environment, Inc. (Lancaster, New York) for TCLP testing (USEPA SW-486) of cadmium, chromium, mercury, lead, and trichloroethylene (TCE). Table 6 shows the results for CSF#2 with 30% and 50% waste loading. All samples showed levels under current RCRA regulatory limits. Outside laboratory reports are given in Appendix D.

**Table 6.** TCLP results with high nitrate and high chloride wastes using CSF#2.

| | High Nitrate Waste | | High Chloride Waste | | |
|---|---|---|---|---|---|
| Metal | Waste Loading 30% | Waste Loading 50% | Waste Loading 30% | Waste Loading 50% | Regulatory Level |
| Cadmium | 0.32 | 0.04 | 0.70 | 0.17 | 1.00 |
| Chromium | 0.36 | 1.30 | 0.50 | 0.68 | 5.00 |
| Mercury | ND | 0.06 | 0.01 | 0.01 | 0.20 |
| Lead | ND | ND | ND | ND | 5.00 |
| TCE | Not Tested | Not Tested | Not Tested | ND | 1.00 |

ND = not detected.
TCE = trichloroethylene.

## 4.8 DOT Solid Oxidizer Test

A mixture of the Pad-A waste and polysiloxane material CSF#2 passed the DOT oxidizer test in that a mixture of sawdust and the waste form material did not burn after 30 minutes compared with the surrogate salt material, which burned under similar conditions in 30 seconds.

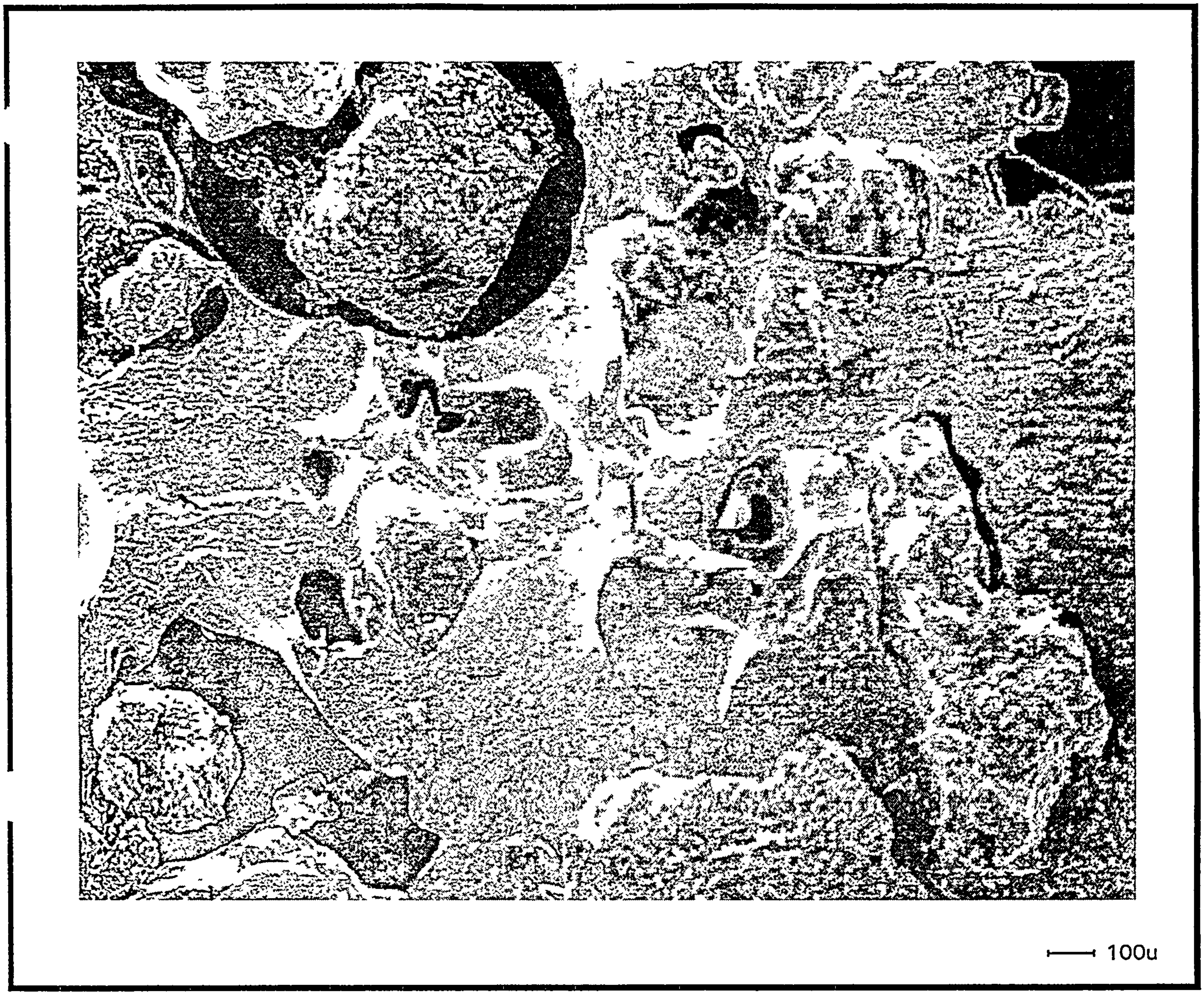
The basis of this test is to compare a test substance and three reference substances with regard to their ability to increase the burning rate or burning intensity of a combustible solid. According to 49 CFR 173.127, samples must be able to pass a sieve size of 0.3 mm and are mixed with sawdust at a 1:1 mass ratio. Testing was carried out by Hark Laboratories, Inc. (Barberton, Ohio). Each mixture was arranged in a conical pile and a wire loop placed inside the pile. The wire was then heated to 1000°C until the first sign of combustion or until it was clear that the mixture would not ignite. The time for combustion was then recorded for each substance. Based on favorable compressive strength and leaching results, only CSF#2 was tested. At 30% waste loading, the CSF#2 material did not burn after approximately 30 minutes, while the surrogate salt waste had a burn time of approximately 30 seconds. Based on these results, the salt waste would be placed in Packing Group I, while the stabilized CSF#2 waste form would not have a specific packing requirement as an oxidizer, i.e., it did not burn.

## 4.9 Zero Head Space Testing for TCE

An outside laboratory (Ecology and Environment) also tested the MWFA waste form made with the high chloride salt for trichloroethylene content and received a "nondetect" as shown in Appendix D.

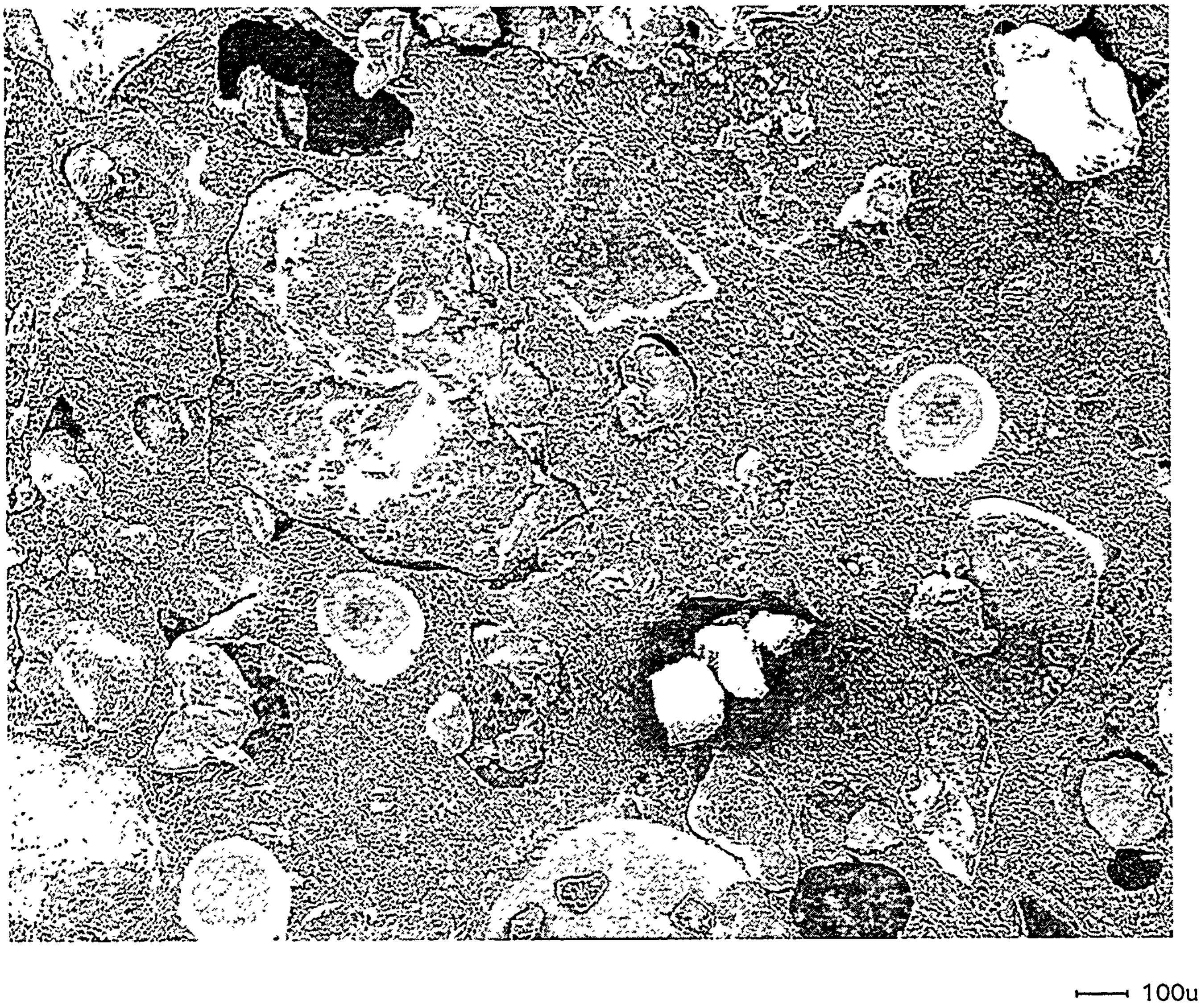
## 4.10 SEM Images of CSF#1 and CSF#2

Images were obtained using a scanning electron microscope (SEM). A SEM gives better depth imaging than a standard microscope. CSF#1 and CSF#2 samples with 50% waste loading were studied. Figures 1 and 2 show the cut surface of CSF#1 and CSF#2 samples. Based on these images, the predominant mechanism of stabilization is macroencapsulation. These images also show that either the mixing procedure or reaction with the catalyst has created variable surrogate crystal size in the polymer-waste matrix. Figures 3 and 4 show the molded surface of CSF#1 and CSF#2 samples. Hairline cracks are evident, attributed to compressive testing of these specific samples. There are also several air pockets (holes), likely due to gas release during curing. Finally, Figure 5 shows the surrogate waste, indicating the relative size of the crystals prior to mixing with polysiloxane. Comparing the size of the crystals in Figure 5 with Figures 1 or 2, it is evident that some physical or chemical mechanism is changing the relative size distribution of the final waste form.



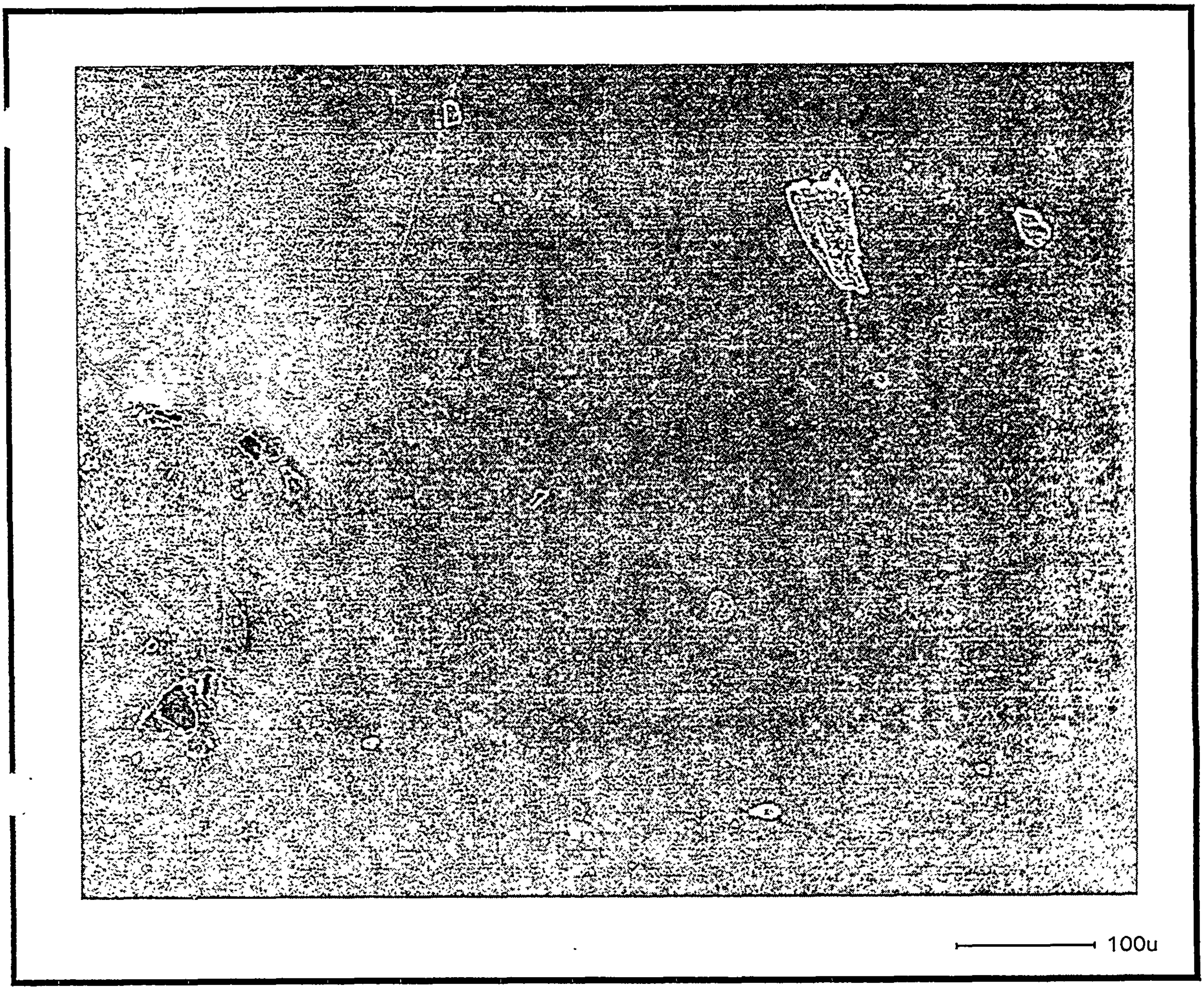
Title
Comment
Mag 50 kV 10 WD 20mm
Date 07-31-97 Spot 10
Time 11:52 Scan

**Figure 1.** SEM image of cut surface for waste form created from CSF#1.



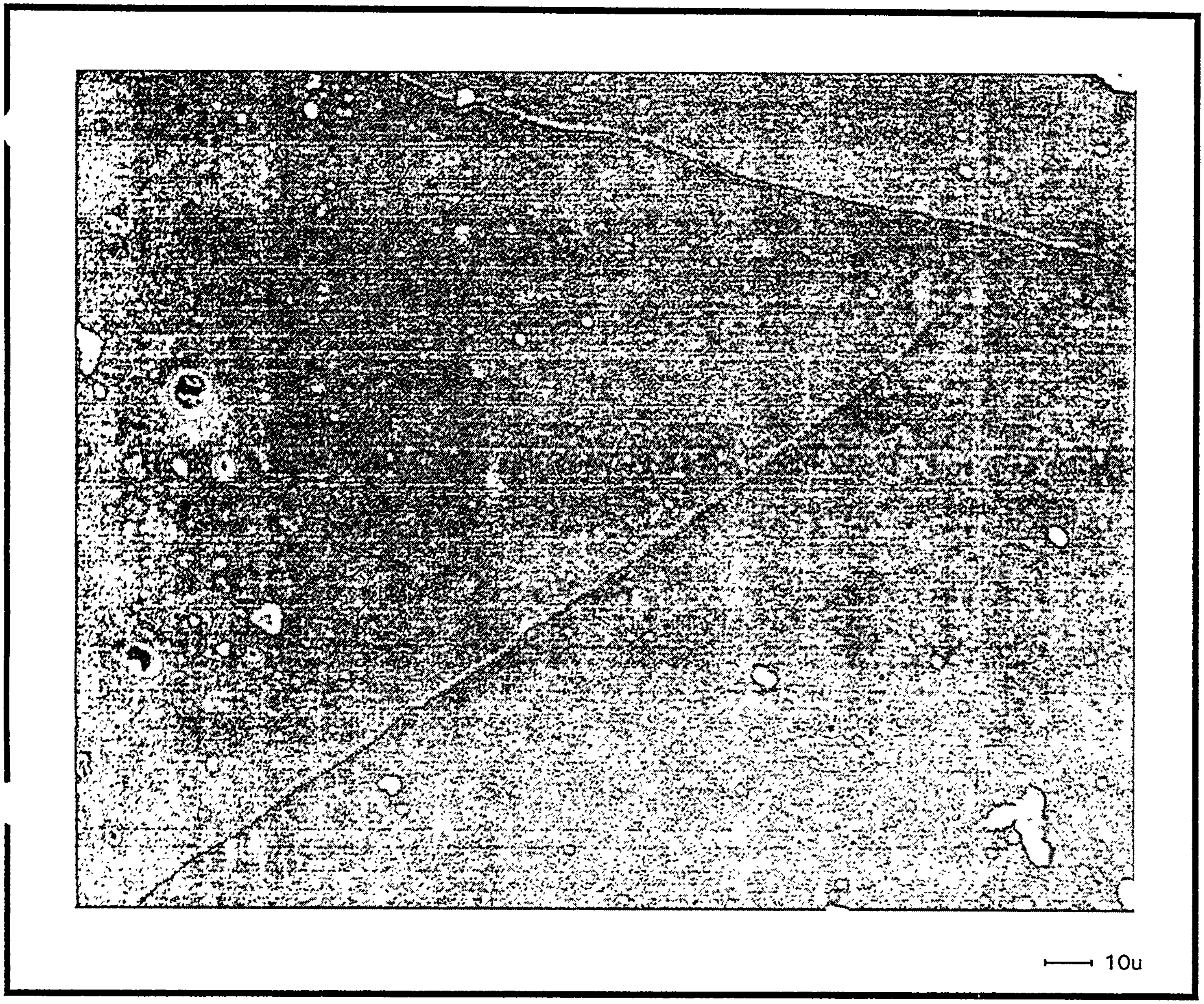
**Title** **Date** 07-31-97 **Time** 12:26
**Comment** 10% Catalyst
**Mag** 50 **kV** 10 **WD** 20mm **Spot** 10 **Scan**

**Figure 2.** SEM image of cut surface for waste form created from CSF#2.



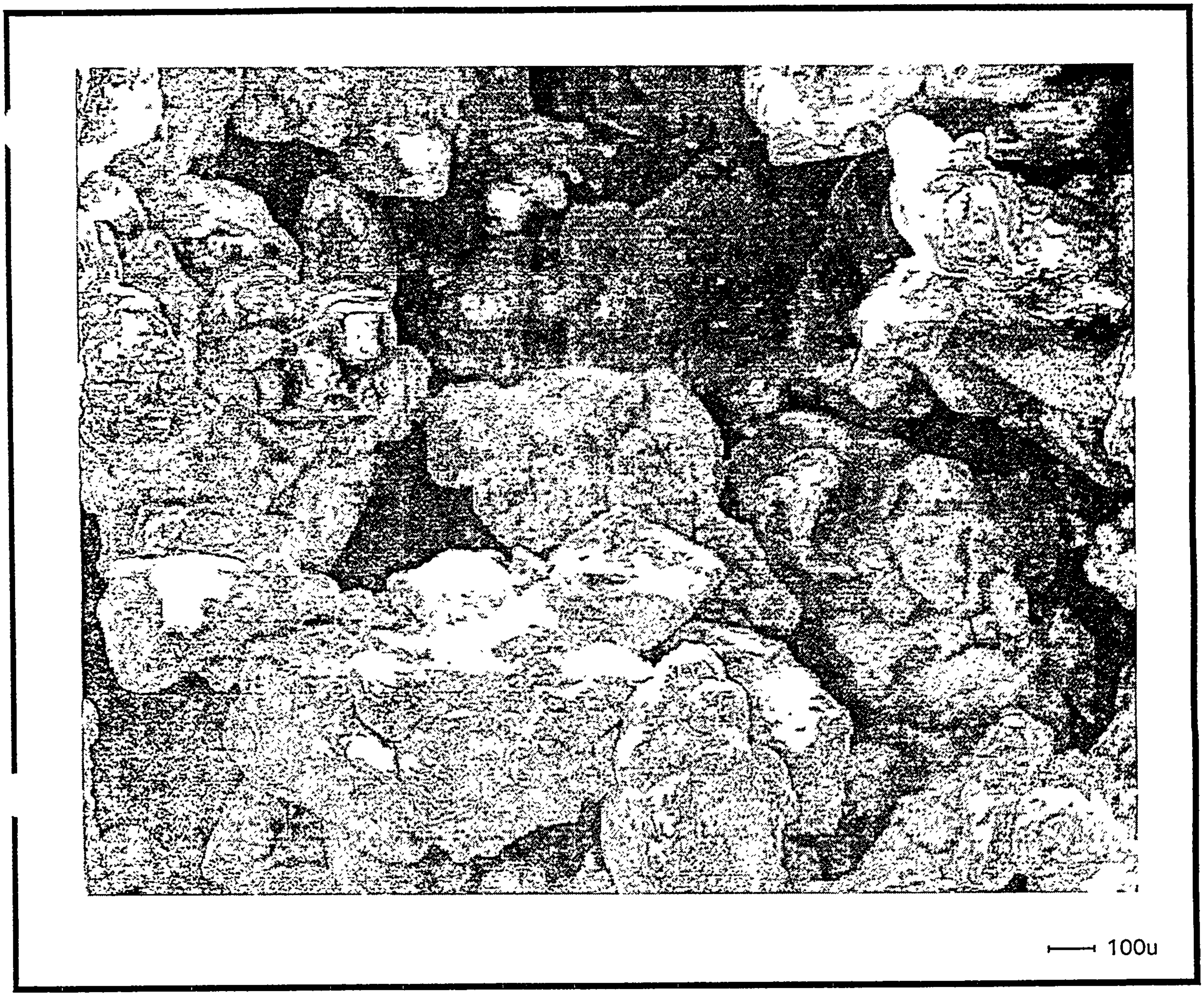
| | | | | |
|---|---|---|---|---|
| **Title** | | | **Date** 07-31-97 | **Time** 12:13 |
| **Comment** | | | | |
| **Mag** 150 | **kV** 10 | **WD** 20mm | **Spot** 10 | **Scan** |

**Figure 3.** SEM image of molded surface for waste form created from CSF#1.



| | | | | |
|---|---|---|---|---|
| **Title** | | | **Date** 07-31-97 | **Time** 11:08 |
| **Comment** | | | | |
| **Mag** 500 | **kV** 10 | **WD** 20mm | **Spot** 10 | **Scan** |

**Figure 4.** SEM image of molded surface for waste form created from CSF#2.



**Title** Nitrate Salt Waste **Date** 07-31-97 **Time** 12:02
**Comment**
**Mag** 50 **kV** 10 **WD** 20mm **Spot** 10 **Scan**

**Figure 5.** SEM image of basic salt waste.

# 5. DISCUSSION OF RESULTS

The results of compressive strength, durability, leach, and oxidizer testing on waste forms created from simple mixing of surrogate salt materials and polysiloxane indicate a positive proof of concept of using this technique for disposing of DOE complex salt in shallow land burial.

## 5.1 Mixing Studies/Compressive Strength

Mixing studies show that both formulations of polysiloxane (CSF#1 and 2) result in cohesive monoliths for waste loadings up to 65% by mass for all surrogate waste forms including Pad-A and the MWFA high nitrate and high chloride salts. At these relatively high salt mass loadings, the compressive strength is still above the suggested American Nuclear Society value of 60 psi; however, for the nominal 50% by mass loadings, the compressive strength for the most promising formulation (CSF#2) is above 637 psi.

The final waste form at 50% by mass in the original mix had a measured density of 90 lbm/ft$^3$. For each cubic foot of waste form there is 45 lbm of salt and 45 lbm of polysiloxane material. In a 55-gal drum there is 7.35 cubic feet or 661 lbm of waste form, of which 330.5 lbm is polysiloxane and 330.5 lbm is salt. The approximate density of polysiloxane is 1.2 g/cc or 74.88 lbm/ft$^3$, meaning that there was 33 gal of polysiloxane in a 55-gal drum of resultant waste form. The approximate density of the salt material is 1.14 g/cc or 71.13 lbm/ft$^3$. This equates to 4.6 cubic feet or 34.7 gal of salt in a 55-gal final waste form. This means that the polysiloxane fills voids in the salt as well as provides macroencapsulation. Each cubic foot of salt then requires 71.7 lbm of polysiloxane at a 50% by mass waste loading.

## 5.2 Hydraulic Conductivity

The CSF#2 material exceeded the NRC goal of $1 \times 10^{-7}$ cm/s hydraulic conductivity; however, the CSF#1 material exhibited a hydraulic conductivity higher than the NRC goal. For long-term disposal, it is important that the media be relatively impervious to water flow. This adds a layer of conservatism to the leaching results in that the leach tests are highly accelerated versions of what actually happens in a buried waste site over thousands of years. For arid sites like in the western United States, low leaching and low hydraulic conductivity may technically not be required because the evaporation rate may exceed the rainfall amount in certain potential site locations.

## 5.3 Durability Testing—Implications for Disposal

Durability testing showed that the CSF#2 material outperformed the CSF#1 material in all cases. Therefore, CSF#1 is not as desirable as CSF#2. For water immersion, base immersion, and wet/dry cycling, there was considerable deterioration of the CSF#1 material but not the CSF#2 material. Durability testing is an accelerated view of climatic and possibly geological changes that might occur when the waste form is disposed in shallow land burial. These tests give only a hint that a waste form may tend to be stable; however, there is no accurate way to model what may happen thousands of years into the future. The philosophy is to leave the waste form in a chemically and physically stable environment. Unless unknown natural forces cause a change to the environment of the shallow land burial, the material should remain stable indefinitely. The only problem at that point is intruder scenarios involving human activities such as well drilling. It is inconceivable that a future generation capable of well drilling would not be able to determine that a mixed waste site exists below ground.

## 5.4 Leach Testing

Examination of outside laboratory TCLP results for the waste form created by CSF#2 and the Pad-A salt surrogate (Table 5) shows that the waste form passes current limits for chromium of 5 ppm unconditionally for 30% waste loadings and most likely for 50% waste loadings when considering that the source term for the surrogate was a factor of 5.8 higher than the actual Pad-A waste (the surrogate had 1405 ppm $Cr^{+6}$ [1400 ppm potassium dichromate and 800 ppm chromium trioxide], and the actual Pad-A salts only has a source term of 180 ppm $Cr^{+6}$). It is most likely that the 30% waste loading case in the actual Pad-A salts will also pass the proposed UTS of 0.86 ppm chromium because the proposed UTS is only a factor of 2.7 lower than the value achieved for the surrogate; yet, the surrogate had a factor of 5.8 too high $Cr^{+6}$ loadings. Nevertheless, for the higher waste loadings (greater than 30%), application of polysiloxane may require a metal scavenger like iron oxide or phosphates in the matrix to meet the UTS. Recent studies have shown that the scavengers can be introduced without affecting the matrix and that the TCLP results are greatly improved.

For the MWFA salt material, all hazardous metals were below current and proposed guidelines except for one test reported in Table 6. The exception is that for 30% waste loading, the chromium TCLP value was 0.36 ppm for the MWFA high nitrate waste loading, which is below the proposed UTS of 0.86 ppm. However, for 50% waste loading the value was 1.3 ppm, which is slightly above the proposed UTS. This is confusing in that the high chloride waste material was below the proposed UTS for chromium for both 30% and 50% waste loadings, even though the physical characteristics of the salt material were essentially the same. It is possible that the TCE loading in the surrogate material affected the high nitrate salt material differently than the high chloride material or alternatively that the chromium behaves differently in the presence of nitrate than chloride salts. It is particularly puzzling in that for the high nitrate salt-based Pad-A surrogate with 1045 ppm $Cr^{+6}$ (highly soluble), the leaching for TCLP was on the same order of magnitude as for the much less soluble chromium oxide used in the MWFA surrogate for the same waste loading.

## 5.5 DOT Solid Oxidizer Test

The high nitrate waste form containing 87% by mass nitrates did not burn in 30 minutes when subjected to the DOT oxidizer test relative to the base salt which burned in 30 seconds. The implications are that the solidified waste form is not flammable and is not subject to flammability issues.

## 5.6 Zero Head Space Testing for TCE

A zero head space detection for TCE resulted in a nondetect for the high chloride MWFA salt material. The source term for this material is 1000 ppm TCE. It is not clear whether the source term evaporated during mixing or that the encapsulation process actually retarded the diffusion of highly volatile TCE through the matrix. Further research would be required on this topic to determine the mechanism for encapsulating the highly volatile TCE.

## 5.7 SEM Studies

The SEM studies suggest that the waste form is created mostly by an encapsulation process; however, there appears to be some size change to the original salt crystals. The explanation for this phenomena and understanding the encapsulation/polymerization process better is the subject of another study.

# 6. COST ESTIMATE—WASTE MANAGEMENT ISSUES

The application of this encapsulation technology is fairly straightforward. It would involve creating a high-efficiency particulate air filter ventilated facility with simple paddle wheel mixing system of screw feeders. The mixture of base material and salt would be extruded through a disposable commercially available nozzle at which point, a platinum catalyst would be added to start the polymerization process. The material upstream of the extruder could be stored indefinitely without polymerization. If the process became stalled, the extruder could be replaced and the "set up" extruder treated as secondary waste. The extruder could pour the polymerizing material into waste containers such as 4 x 4 x 8 ft polyethylene disposable boxes with built-in lifting lugs. The waste form can be designed to set up in 15 minutes to 5 hours to support the required throughput. The waste form could be taken directly to the disposal facility for stacking and possibly shallow land burial with soil. In geological times (millions of years), the high silicon content should tend toward a granite-type reef of material given similar temperature and pressure effects. More practically, once interred in a chemically compatible environment, as long as that environment does not change, the waste form should remain stable.

A preliminary cost estimate has been performed in which the cost of disposing the polysiloxane-based waste form is compared with the cost of mixing salt with cement. It was assumed that the cement could be mixed at 10% waste loading and that the polysiloxane could be mixed at 50% waste loading and that disposal costs are both $500 per cubic foot of salt. It was further estimated that each cubic foot of salt material requires 71.7 lbm of polysiloxane material (see Section 5.1). For cement-based waste forms, each 1 cubic foot of waste becomes 10 cubit feet of disposed material; therefore, if the cost of concrete is free, the disposal cost alone is $5,000 per cubic foot of salt.

Current pricing on the polysiloxane system is approximately $8/lbm. Therefore, for each cubic foot of salt, the mixing cost would be $8 × 71.7 lbm = $573. In addition, the disposal cost of the resultant waste form is the same $500/cubic foot as the concrete. Even though the final waste form is $500 per cubic foot, the cost for disposal of the original salt waste is $793/ft$^3$ because there is only 34.7 gal of salt in each 55-gal waste form (see Section 5.1). The total cost to dispose of a cubic foot of salt then is $573 for polysiloxane and $793 for disposal of the resultant waste form or a total of $1,366 per cubic foot of salt. This compares with approximately $5,000 per cubic foot of salt for the concrete/salt mixture or a savings factor of 3.6. It is not clear whether concrete can meet treatment standards or compressive strength for 10% salt loadings. In addition, once a consumer market develops, the $/lbm of polysiloxane will decrease to perhaps as low as $4/lbm (based on discussions between Orbit Technologies and vendors for base polysiloxane).

# 7. CONCLUSIONS/RECOMMENDATIONS

## 7.1 Conclusions

1. Orbit Technologies polysiloxane-based grouting material appears to be suitable for stabilization of DOE complex salt wastes. A waste form suitable for shallow land burial is created. The waste form passes the TCLP protocol and DOT oxidizer testing.

2. Orbit material CSF#2 outperformed material CSF#1 in most cases for durability, compressive strength, and leaching and is therefore the material of choice for future applications of polysiloxane for mixed waste salts.

3. Polysiloxane is easy to work such that this application would involve a simple off-the-shelf mixing apparatus. In addition, it is a room-temperature process using no hazardous materials. It is concluded that a cost savings in excess of 3:1 over concrete can be achieved by applying polysiloxane encapsulation over simple mixing with concrete, even though the base price for the encapsulation material is higher for polysiloxane.

## 7.2 Recommendations

1. Ship Pad-A salts currently stored at the Waste Reduction Operations Complex of the INEEL to Orbit Technologies (University of Akron laboratories). Mix the salts in a 40% to 50% waste loading by mass with Orbit Technologies polysiloxane, and test the mixture for leaching. Ship the resultant residue from testing and the resultant waste form back to the INEEL for disposal at potentially a subtitle part C permitted facility such as Envirocare of Utah, Inc. While at the University of Akron, TCLP and DOT oxidizer testing could be performed on the resultant waste form (at an approved outside laboratory) prior to shipping back to the INEEL. Although the outcome is fairly predictable based on the results of the Fiscal Year 1997 work described in the report, a treatability study on actual waste will stimulate thinking from other waste management specialists in using the polysiloxane encapsulation technology with other DOE complex salts.

2. Investigate the use of scavengers such as iron oxides or phosphates to improve the leachability during TCLP testing at higher waste loadings (possibly as high as 60% waste loadings by mass).

3. Present these results to a variety of DOE complex mixed waste managers such that the technology can be transferred properly from the technology development side of DOE to the waste management/environmental side of DOE.

4. Investigate the technology for application beyond mixed waste salts. Specifically, this technology could be applied to stored transuranic (TRU) waste, alpha mixed low-level waste, or retrieved buried TRU waste. A waste form suitable for shallow land burial could be created by encapsuling shredded waste material and placing the material in the same 4 x 4 x 8 ft polyethylene boxes for interim storage or disposal.

In past studies of the buried waste, one of the most restrictive waste materials for encapsulation is the mixed waste high nitrate salts that were tested successfully for encapsulation in this study. Another obvious waste stream is the INEEL Idaho Chemical Processing Plant calcined waste material. With the high resultant compressive strength and high gamma ray tolerance, polysiloxane encapsulation offers a cost-effective alternative to expensive thermal treatments.

# REFERENCES

1. I. K. Shvetsov et al., *Determination of the Structural Peculiarities of the Material and the Components Distribution*, Cerametalic Silicone Foam Rubber Research Programme, Kurchatov Institute, April 1994.

# Appendix A

## Surrogate Waste Composition

# Appendix A

## Surrogate Waste Composition

There were three surrogates developed for the encapsulation study including a Pad-A derivative and two Mixed Waste Focus Area suggested salts. For the Pad-A surrogate, the primary material is potassium nitrate, with chromium as the contaminant of concern. The principal components of the Pad-A waste are $KNO_3$ and $NaNO_3$ (-90% by weight). The remaining 10% consists of other salts (e.g., NaCl), anions (e.g., $SO_4^{-2}$), and metals (e.g., iron and chromium). Sodium cyanide is present in the Pad-A waste, but it is not included here because it is less than 20 ppm. The Mixed Waste Focus Area waste consisted of a high nitrate and high chloride salt material, with Resource Conservation and Recovery Act metals as the contaminants.

Table A-1 gives the composition of the Pad-A surrogate, and Table A-2 gives the composition of the Mixed Waste Focus Area material.

**Table A-1.** Mass weight percentages per kilogram of surrogate salt waste.

| | Mass, g | Wt% | Compound | MW |
|---|---|---|---|---|
| | 530.30 | 53.0% | $NaNo_3$ | 85.0 |
| | 348.00 | 34.8% | $KNO_3$ | 101.1 |
| | 1.70 | 0.2% | $Al_2O_3$ | 102.0 |
| | 14.00 | 1.4% | NaF | 42.0 |
| | 54.00 | 5.4% | $Na_2SO_4$ | 142.0 |
| | 1.40 | 0.1% | $K_2Cr_2O_7$ | 294.2 |
| | 31.00 | 3.1% | NaCl | 58.4 |
| | 16.90 | 1.7% | $NaH_2PO_4$ | 120.0 |
| | 0.80 | 0.1% | $CrO_3$ | 100.0 |
| | 0.00 | 0.0% | NaCN | 49.0 |
| | 1.90 | 0.2% | $FeCl_3$ | 162.2 |
| Total = | 1,000.00 | 100% | | |

**Table A–2.** High chloride and high nitrate wastes.

| Constituent | High Chloride (wt%) | High Nitrate (wt%) |
|---|---|---|
| $Fe(OH)_3$ | 12.7% | 6.0% |
| $Al_2(OH)_3$ | 8.4% | 4.0% |
| $Na_2PO_4$ | 4.2% | 2.0% |
| $Mg(OH)_2$ | 8.4% | 4.0% |
| MicroCel E | 16.9% | 8.0% |
| Portland cement | 4.2% | 2.0% |
| $H_2O$ | 29.6% | 13.9% |
| $CaSO_4$ | 5.0% | 0.0% |
| NaCl | 9.9% | 0.0% |
| $NaNO_3$ | 0.0% | 59.6% |
| Metal Oxides: | (mg/kg) | (mg/kg) |
| PbO | 1000 | 1000 |
| $CrO_3$ | 1000 | 1000 |
| HgO | 1000 | 1000 |
| CdO | 1000 | 1000 |
| NiO | 1000 | 1000 |
| Hazardous Organic: | (mg/kg) | (mg/kg) |
| TCE | 1000 | 1000 |

# Appendix B

## Nitrate Salt Waste Storage/Generation in Defense Facilities

# Appendix B

# Nitrate Salt Waste Storage/Generation in Defense Facilities

Table B-1 gives a summary of the total mass of salt already generated and currently being generated in the DOE complex. There are stored within the complex over 250,000,000 kg of mixed waste salts with varying types of radioactive components. The radioactive material includes low-level waste, high-level waste, and transuranic waste listing and the hazardous components include Resource Conservation and Recovery Act metals and volatile organics.

**Table B-1.** Summary of nitrate salt waste storage and generation in defense facilities.

| Site | Type | Amount stored (kg) | Generation rate (kg/yr) | Comments |
|---|---|---|---|---|
| Feed Materials Production Center | Effluent | - | 2,200,000[a] | Biodenitrification planned |
| Hanford-Rockwell | Effluent | - | 1,100,000[a] | Discharged to ponds, cribs, etc. |
| Hanford-Rockwell | LLW, HLW or TRU | 145,000,000[b] | 1,180,000[b] | Stored in tanks |
| Hanford-United Nuclear | LLW | 980,000[a] | 2,020,000[a] | Discharged to solar ponds |
| Idaho Chemical Processing Plant | HLW | 1,460,000[a] | (24,000)[a] reduction | Liquid in tanks, $NO_x$ released |
| Idaho Chemical Processing Plant | HLW | 92,800[a] | 13,000[a] | Calcine in bin sets |
| Idaho-Radioactive Waste Management Complex | LLW or TRU | 11,300,000[b] | - | Buried or stored |
| Los Alamos National Laboratory | Effluent | - | 70,800[a] | Discharged to canyon |
| Lawrence Livermore National Laboratory | LLW | - | <200[a] | Cemented, shipped to NTS |
| Mound Facility | LLW | - | 1,500[c] | Cemented, shipped to NTS |
| Nevada Test Site (NTS) | LLW | 10,400,000[b] | - | Received RFP salts, FY78-FY85 |
| Oak Ridge Y-12 Plant | Effluent | - | 664,000[a] | Acid recycle biodenitrification |
| Rocky Flats Plant (RFP) | LLW | - | 930,000 to 2,050,000[b] | Salts/saltcrete shipped to NTS |
| Rocky Flats Plant | LLW | 1,450,000[b] | - | Solar ponds being decommissioned |
| Savannah River Plant | HLW | 82,900,000[b] | 1,480,000 to 2,960,000[b] | Grouting facility planned |
| TNS-Aerojet | LLW | - | 38,000[a] | Discharged to creek |

**Table B-1.** (continued).

| Site | Type | Amount stored (kg) | Generation rate (kg/yr) | Comments |
|---|---|---|---|---|
| West Valley Pilot Project | HLW | 557,000[a] | - | Basic waste |
| West Valley Pilot Project | HLW | 73,300[b] | - | Acidic waste |
| Total | | 254,000,000 | 7,500,000 to 11,200,000 | |

---

a. Based on nitrate ion.

b. Based on nitrate compounds.

c. As cemented $Fe(OH)_3$ sludge.

# Appendix C

# Equipment List

# Appendix C

# Equipment List

The following equipment will be used in the testing program:

- Water—Nanopure system
- Drill with stainless steel mixing blade
- pH meter—Cole Parmer Model 05669-20
- spectrophotometer—Spectronic Model #501
- INSTRON
- Permeameter
- Microscope with digital analysis
- Water bath—Fisher Model 127
- Drying oven—Grieve Model LW-201C
- Moisture chamber
- Scale—Ohaus Analytical
- Plastic syringe
- Glass and plastic beakers

# Appendix D

# Outside Laboratory Test Results

**ecology and environment, inc.**
International Specialists in the Environment

ANALYTICAL SERVICES CENTER
4493 Walden Avenue
Lancaster, New York 14086
Tel. (716) 685-8080, Fax: (716) 685-0852

September 10, 1997

Dr. Chris Miller
University of Akron
Department of Civil Enginering
Akron, OH 44325-3905

RE: 9702.070

Dear Dr. Miller:

Attached is the laboratory report of the analysis conducted on four samples received at the Analytical Services Center on August 8, 1997. Analysis was performed according to the procedures set forth in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Third Edition, Update 2B, USEPA, January 1995.

The chain of custody form provided herein is integral to this report and must be included with the analytical results forms upon transferral to another data user.

A facsimile of this report was forwarded to you on 9/05/97.

The accuracy of all analyses depends upon the representative nature of the sample and the reliability of collection procedures as well as the accuracy of the laboratory analysis of the sample as submitted. Ecology and Environment, Inc.'s activity and representations with respect to these samples are limited solely to the laboratory analysis of the samples presented to us.

All samples on which this report is based will be retained by E & E for a period of 30 days from the date of this report, unless otherwise instructed by the client. If additional storage of samples is requested by the client, a storage fee of $1.00 per sample container per month will be charged for each sample, with such charges accruing until destruction of the samples is authorized by the client.

Very truly yours,

Gary Hahn

Gary Hahn, Director
Analytical Services Center

GH/par
Enclosure
cc: mr. giansircusa

Results of Analysis of TCLP Extracts Job Number :9702.070
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

CLIENT : MILLER, DR. CHRIS
SAMPLE ID LAB :EE-97-72624
SAMPLE ID CLIENT: 1A DUP

MATRIX: SOLID
UNITS : MG/L

NITRATE SALT
30%
CSF 97-2

| PARAMETER | RESULTS | Q | QUANTITATION LIMIT | REGULATORY LEVEL |
|---|---|---|---|---|
| Chromium | 2.3 | | 0.030 | 5.0 |

QUALIFIERS: C = COMMENT ND = NOT DETECTED
J = ESTIMATED VALUE

Results of Analysis of TCLP Extracts    Job Number :9702.070
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

CLIENT : MILLER, DR. CHRIS
SAMPLE ID LAB :EE-97-72623
SAMPLE ID CLIENT: 1A

MATRIX: SOLID
UNITS : MG/L

NITRATE SALT 30%

CSF97-2

| PARAMETER | RESULTS | Q | QUANTITATION LIMIT | REGULATORY LEVEL |
|---|---|---|---|---|
| Chromium | 2.5 |  | 0.030 | 5.0 |

QUALIFIERS: C = COMMENT    ND = NOT DETECTED
J = ESTIMATED VALUE

Results of Analysis of TCLP Extracts Job Number :9702.070
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

CLIENT : MILLER, DR. CHRIS
SAMPLE ID LAB :EE-97-72625
SAMPLE ID CLIENT: 2A
MATRIX: SOLID
UNITS : MG/L

NITRATE SALT 50%
CSF97-2

| PARAMETER | RESULTS | Q | QUANTITATION LIMIT | REGULATORY LEVEL |
|---|---|---|---|---|
| Chromium | 5.9 | | 0.030 | 5.0 |

QUALIFIERS: C = COMMENT ND = NOT DETECTED
J = ESTIMATED VALUE

**ecology and environment, inc.**
International Specialists in the Environment

ANALYTICAL SERVICES CENTER
4493 Walden Avenue
Lancaster, New York 14086
Tel. (716) 685-8080, Fax: (716) 685-0852

October 9, 1997

Mr. Jimmy Giansircusa
2011 Palomar Airport Road, Suite 100
Carlsbad, CA 92009

RE: 9702.242

Dear Mr. Giansircusa:

Attached is the laboratory report of the analysis conducted on four samples received at the Analytical Services Center on October 9, 1997. Analysis was performed according to the procedures set forth in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Third Edition, Update 2B, USEPA, January 1995.

Under separate cover, you will be receiving an invoice for Job 9702.242.

The chain of custody form provided herein is integral to this report and must be included with the analytical results forms upon transferral to another data user.

A facsimile of this report was forwarded to Dr. Chris Miller 10/03/97.

The accuracy of all analyses depends upon the representative nature of the sample and the reliability of collection procedures as well as the accuracy of the laboratory analysis of the sample as submitted. Ecology and Environment, Inc.'s activity and representations with respect to these samples are limited solely to the laboratory analysis of the samples presented to us.

All samples on which this report is based will be retained by E & E for a period of 30 days from the date of this report, unless otherwise instructed by the client. If additional storage of samples is requested by the client, a storage fee of $1.00 per sample container per month will be charged for each sample, with such charges accruing until destruction of the samples is authorized by the client.

Very truly yours,

Gary Hahn/RC

Gary Hahn, Director
Analytical Services Center

GH/lmc
Enclosure
cc: Dr. Chris Miller

Results of Analysis of TCLP Extracts Job Number :9702.242
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

HIGH
NITRATE
30%
CSF97-2

CLIENT : GIANSIRCUSA, MR. JIMMY
SAMPLE ID LAB :EE-97-73848
SAMPLE ID CLIENT: SAMPLE A

MATRIX: SOLID
UNITS : MG/L

| PARAMETER | RESULTS | Q | QUANTITATION LIMIT | REGULATORY LEVEL |
|---|---|---|---|---|
| Cadmium | 0.32 | | 0.015 | 1.0 |
| Chromium | 0.36 | | 0.030 | 5.0 |
| Mercury | ND | | 0.010 | 0.20 |
| Lead | ND | | 0.15 | 5.0 |

QUALIFIERS: C = COMMENT ND = NOT DETECTED
J = ESTIMATED VALUE

Results of Analysis of TCLP Extracts Job Number :9702.242
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

HIGH
NITRATE
50%
CSF97-2

CLIENT : GIANSIRCUSA, MR. JIMMY
SAMPLE ID LAB :EE-97-73849
SAMPLE ID CLIENT: SAMPLE B

MATRIX: SOLID
UNITS : MG/L

| PARAMETER | RESULTS | Q | QUANTITATION LIMIT | REGULATORY LEVEL |
|---|---|---|---|---|
| Cadmium | 0.041 | | 0.015 | 1.0 |
| Chromium | 1.3 | | 0.030 | 5.0 |
| Mercury | 0.060 | | 0.010 | 0.20 |
| Lead | ND | | 0.15 | 5.0 |

QUALIFIERS: C = COMMENT ND = NOT DETECTED
J = ESTIMATED VALUE

Results of Analysis of TCLP Extracts    Job Number :9702.242
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

HIGH CHLORIDE 30% CSF 97-2

CLIENT : GIANSIRCUSA, MR. JIMMY
SAMPLE ID LAB :EE-97-73850
SAMPLE ID CLIENT: SAMPLE C
MATRIX: SOLID
UNITS : MG/L

| PARAMETER | RESULTS | Q | QUANTITATION LIMIT | REGULATORY LEVEL |
|---|---|---|---|---|
| Cadmium | 0.70 | | 0.015 | 1.0 |
| Chromium | 0.50 | | 0.030 | 5.0 |
| Mercury | 0.01 | | 0.010 | 0.20 |
| Lead | ND | | 0.15 | 5.0 |

QUALIFIERS: C = COMMENT    ND = NOT DETECTED
J = ESTIMATED VALUE

Results of Analysis of TCLP Extracts    Job Number :9702.242
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

HIGH CHLORIDE 50% CSF 97-2

CLIENT : GIANSIRCUSA, MR. JIMMY
SAMPLE ID LAB :EE-97-73851
SAMPLE ID CLIENT: SAMPLE D
MATRIX: SOLID
UNITS : MG/L

| PARAMETER | RESULTS | Q | QUANTITATION LIMIT | REGULATORY LEVEL |
|---|---|---|---|---|
| Cadmium | 0.17 | | 0.015 | 1.0 |
| Chromium | 0.68 | | 0.030 | 5.0 |
| Mercury | 0.01 | | 0.010 | 0.20 |
| Lead | ND | | 0.15 | 5.0 |

QUALIFIERS: C = COMMENT    ND = NOT DETECTED
J = ESTIMATED VALUE

TEST CODE :STCPRH1

JOB NUMBER :9702.242
ELAP ID : 10486

Ecology and Environment, Inc.
Analytical Services Center

CLIENT : GIANSIRCUSA, MR. JIMMY
TEST NAME : TCLP PURGEABLES 80
SAMPLE ID LAB : EE-97-73851
SAMPLE ID CLIENT: SAMPLE D

UNITS : UG/L
MATRIX: SOLID

HIGH
CHLORIDE
50%
CSF97-2

| PARAMETER | RESULTS | Q | QNT. LIMIT |
|---|---|---|---|
| Trichloroethene | ND | | 1.0 |

QUALIFIERS: C = COMMENT
ND = NOT DETECTED
J = ESTIMATED VALUE
B = ALSO PRESENT IN BLANK
N = ANALYTE WAS NOT CONFIRMED BY ALTERNATE PROCEDURE
A = PHENOMENON OF METHODOLOGY WITH ACID PRESERVATION

# METALLURGICAL REPORT

HL-6A

**HARK LABORATORIES, INC.**
P.O. Box 574 871 Wooster Road W.
Barberton, Ohio 44203
330/825-9931

Customer THE UNIVERSITY OF AKRON

Date SEPTEMBER 4, 1997

P.O. No. 152783

Lab No. M57202-PG. 1 OF 1

## CHEMICAL ANALYSIS

| Specimen Identity | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| N/A | | | | | | | | | | | | | | |

## TENSILE TEST

| Specimen Identity | Dimension In. | Area Sp. In. | Yield Lbs. | Ultimate Lbs. | Y. PSI | U.T.S. PSI | % Elong. | % Red. of Area |
|---|---|---|---|---|---|---|---|---|
| N/A | | | | | | | | |

HARDNESS

N/A

1 TEST SAMPLE LOT OF 3 MARKED: 1 BAG "A"; & 2 PCS. "SMALL BLUE" & "LARGE BLUE" FOR OXIDATION TEST:

| SAMPLE I.D. | RESULTS |
|---|---|
| "A": 4 MINUTES TO START - BURN TIME: 30 SECONDS. | |
| "SMALL BLUE" | NO COMBUSTION AT 1000°C |
| "LARGE BLUE" | NO COMBUSTION AT 1000°C |

CSF97-2

TEST PROCEDURE: HEAT TO 1000°C (555°F): OBSERVE FOR SIGN OF COMBUSTION AND NOTE TIME UNTIL COMBUSTION IS COMPLETE. SAMPLE 1 TO 1 MASS TEST; SAMPLE + SAWDUST.

REPORT ISSUED & FAXED TO/PER ATTN: DR. CHRIS MILLER OF THE UNIVERSITY OF AKRON.

Chief Metallurgist