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Preliminary Laboratory Study of Plutonium-238 Dissolution from Mound Soil by Means of the ACT*DE*CONSM Process

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MASTER

**Brown is affiliated with Information and Publishing Division and Heinrich with Chemical Technology Division.*

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Foreword

In this report, the tests performed, results achieved, conclusions drawn, and recommendations made with respect to the effectiveness of the ACT*DE*CONSM process are presented. This material was reviewed by an EG&G Mound Applied Technologies (Mound) staff member, who concurred with the conclusions and recommendations presented in this report regarding the potential for using the ACT*DE*CONSM process to treat Mound soils.

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Abstract

The treatment of contaminated soil presents a significant technical problem. Soil-washing and chemical-extraction methods have proven to be effective for specific applications, but a process with more comprehensive treatment properties that is both cost-effective and environmentally propitious is needed. Bradtec, Inc., has developed a process, the ACT*DE*CONSM process, that has been tested on soil contaminated with plutonium. The process effectively extracted Pu-238 after three washes, reducing the contamination levels from approximately 20 Bq/g to 1.6-1.9 Bq/g and yielding a decontamination factor ranging from 11 to 13. By using four or more ACT*DE*CONSM washes or a continuous-flow process with ACT*DE*CONSM solvents on a pilot-scale test, a target decontamination level of 0.93 Bq/g might be achievable.

1 Introduction

1.1 Background

The removal and/or treatment of contaminated soils is a major problem facing the U.S. Department of Energy (DOE) and the U.S. Environmental Protection Agency (EPA) Superfund program. The Department of Energy has identified several sites that have contaminated soil as a result of past disposal and waste burial practices. In terms of the volume of contaminated material, two sites illustrate the magnitude of this problem: the EG&G Mound Applied Technologies site (referred to as the Mound site) in Miamisburg, Ohio, at which the estimated total volume of soil to be recovered or treated is approximately 4×10^6 ft³, and the Idaho National Engineering Laboratory (INEL), at which the total volume of contaminated soil in the Radioactive Waste Management Complex is approximately 10×10^6 ft³. Other sites with significant soil-contamination problems include Hanford and Savannah River.

Approaches to the treatment of soils contaminated with radionuclides and heavy metals have included the removal of the soil and its subsequent disposal in a controlled burial site, soil washing, and chemical extraction. Each of these methods has environmental, procedural, and

regulatory shortcomings. Therefore, a more effective treatment process is needed. Such a process would have the following characteristics:

- Be selective (i.e., recover only contaminants, leaving nonhazardous minerals with the treated soil),
- Produce low volumes of waste (ideally, close to the volume of contaminant recovered) and no secondary wastes,
- Use environmentally benign solvents, and
- Allow for the recovery of the contaminants for recycling or disposal with relative ease.

Ideally, the goal of treating contaminated soil is to decrease the levels of contaminants to meet EPA standards while the treated, natural soil is allowed to remain in place.

Bradtec, Inc., has developed and tested a dilute, aqueous-based chemical-extraction process that, according to Bradtec representatives, has the desired characteristics. This process is called the ACT*DE*CONSM process. The ACT*DE*CONSM process can be applied either ex situ or in situ. Bradtec reports that in tests of the process, the levels of contamination in soil have been decreased to less than 1.3 Bq/g for uranium and less than 0.93 Bq/g for plutonium. The process can also be used to treat materials other than soil, including large process vessels (e.g., enrichment plant components), hot cells, or military vehicles contaminated with depleted uranium.

The Department of Energy, through Argonne National Laboratory (ANL), issued a contract to Bradtec for testing the ACT*DE*CONSM process in the treatment of plutonium-contaminated soils at the Mound site. This contract was for the first phase of the research, described herein as the Phase I laboratory study. The testing performed during and the results obtained from the Phase I work are provided in this report. The tasks to be undertaken in subsequent phases are described in Section 4.

1.2 Principle of ACT*DE*CONSM Testing

Clean soil from a chosen site is contaminated with the radionuclide(s) of interest by using a technique known as spiking. Spiking involves bringing the soil into contact with a known amount of the contaminant — in this instance, Pu-238. The soil and a solution containing the contaminant are stirred together for several hours, and then the solution is filtered. The contact solution is analyzed for any residual contamination. The soil is then washed at least three times with water to remove interstitial contamination (i.e., contamination that is not truly sorbed onto the soil but lies between the particles in the soil). The amount of contaminant sorbed onto the soil is determined by

subtracting the concentration of contaminant found in the contact solution and washings from the original concentration of contaminant used in the spike. The amount of contaminant sorbed onto the soil also can be determined by leaching, a procedure that uses strongly acidic solutions under forcing conditions to recover the contaminant from the soil.

The spiked soil was used in tests of the ACT*DE*CONSM process. The soil was washed with ACT*DE*CONSM wash solutions, and the extent of decontamination was determined by measuring the concentration of contaminant in each of the ACT*DE*CONSM wash solutions. After the ACT*DE*CONSM treatment, the soil was leached to determine the concentration of contaminant that remained on the soil. The ratio of the contaminant concentration on the spiked soil to the contaminant concentration on the treated soil is the decontamination factor (DF).[†]

The test described in this report details the dissolution and extraction of Pu-238 from Mound soil that had been contaminated by spiking it with Pu-238.

[†] The DF gives the level of decontamination of the soil obtained after three ACT*DE*CONSM washes. The DF is calculated by dividing the Pu-238 activity on the spiked soil by the Pu-238 activity left on the soil after the ACT*DE*CONSM washes.

2 Study Approach

The objective of this study is to validate the potential of the Bradtec proprietary ACT*DE*CONSM process for extracting plutonium contaminants from soil. This study represents Phase I of the ACT*DE*CONSM tests on Mound soil. The tests were carried out in Bradtec's laboratory and were witnessed by Robert R. Heinrich of ANL. The tests were conducted on uncontaminated soil, which was spiked by Bradtec with Pu-238. The study encompassed soil preparation and analysis, as well as the ACT*DE*CONSM dissolution studies. These tasks are described in the following sections.

2.1 Soil Preparation and Analysis

The raw soil sample (sediment) received from the Mound site was dry-sieved to collect and homogenize the silt and clay fractions of the soil and to produce a batch of soil typical of the sediment in the Miami Erie Canal. This standard batch of soil was thoroughly characterized. The uncontaminated, sieved, and blended Mound soil was analyzed by Bradtec and outside laboratories for the following parameters (see Appendix A for the results of the characterization):

- Particle size;
- Mineralogy;
- Organic carbon content;
- Iron, calcium, magnesium, and manganese content;
- Ion exchange capacity;
- Carbonates;
- pH; and
- Soil Eh.

Because the Mound soil has a high calcite content, there was concern that the spiking procedure would dissolve the calcite, thereby changing the characteristics of the soil to be tested. To minimize these effects, the soil sample used for dissolution testing consisted of approximately four parts sieved, unspiked soil and one part sieved, spiked soil blended together. Mixing ensured

that the natural calcite content of the spiked soil would be replenished by the calcite in the unspiked soil should the spiking procedure dissolve the calcite.

After the characterization, portions of the soil were spiked with Pu-238 for use in the ACT*DE*CONSM dissolution tests (see Appendix B, Section B.1, for the soil spiking procedure). The contaminant was applied as a spike of Pu-238 in the form of plutonium nitrate under mildly acidic conditions. To compensate for any losses in soil volume attributable to the dissolution of the calcite, the spiked sample was combined with four times its weight of unspiked soil. The soil was spiked initially with 99.9 ± 1.8 Bq/g (2697 ± 48.6 pCi/g) of Pu-238 so that a sample of 19.8 ± 0.4 Bq/g (533.3 ± 9.7 pCi/g) Pu-238 could be obtained after the soil was mixed with a larger volume of unspiked soil. The clean soil was stirred with the spiked soil for a limited period (the contact time of the spiked soil with the clean soil was shortened so that the test could be completed while the ANL representative was present).

2.2 ACT*DE*CONSM Dissolution Studies

The dissolution studies were conducted in two parts. First, the ACT*DE*CONSM formulation was optimized, and second, the Mound soil was tested by using the optimized formulation. Because the Mound soil sample arrived late at Bradtec's laboratory, the optimization runs were performed on a non-Mound soil sample blended by Bradtec. This soil was high in clay and silt and was made up of a blend of various soils. After the optimization runs, the ACT*DE*CONSM process was tested by using two samples of spiked soil from the Mound site. An outline of the test procedure is provided in Appendix B, Section B.2.

After the soil was spiked and washed, it was divided into three 5-g portions. The dissolution tests were performed on two of these portions, labeled A and B, by using three ACT*DE*CONSM washes. The third portion, labeled C, was used to determine the concentration of Pu-238 sorbed onto the soil. Some of the soil from sample C was sent to Harwell Laboratories in the United Kingdom and to ANL for independent analyses.

After completion of the dissolution studies at Bradtec, Robert Heinrich applied custody seals to all sample vials. Soil samples were then sent to ANL, Harwell Laboratories, and British Rail (BritRail) Laboratories (in the United Kingdom) for analysis of Pu-238. Bradtec also analyzed the soil samples and the wash solutions and completed a mass balance on the soil and dissolution liquors.

3 Results and Discussion

The data gathered as a result of ACT*DE*CONSM dissolution studies quantitatively demonstrated that the ACT*DE*CONSM process could be used to extract Pu-238 from Mound soil. In this section, the data are presented and discussed.

Table 1 shows the activity balance for the spiking of clean soil from the Mound facility with Pu-238. Because 1.5 mL of Pu-238 standard (Pu-238 activity equal to 199.8 ± 3.6 Bq/g [5394.6 ± 97.2 pCi/g]) was used to spike 3 g of clean soil, the amount of Pu-238 used was 299.75 ± 5.4 Bq (8093.3 ± 146 pCi). A total of 3.35 Bq (90.47 pCi) of Pu-238 was found in the contact liquor and washings, which was subtracted from the original spike to give the theoretical amount of Pu-238 on the spiked soil: 296.4 ± 5.4 Bq (8002.8 ± 146 pCi). The 3 g of spiked soil was then blended with 12 g of clean soil. Thus, the theoretical amount of Pu-238 on the blended soil was 19.75 ± 0.4 Bq/g (533.3 ± 9.7 pCi/g). This value was confirmed by analyzing two leachates from the spiked soil. The results from these analyses were 20.51 ± 0.63 Bq/g (553.9 ± 16.4 pCi/g) Pu-238 and 20.34 ± 0.38 Bq/g (549.1 ± 10.1 pCi/g) Pu-238 in the soil, respectively.

Tables 2 and 3 show the activity balance for the ACT*DE*CONSM test on sample A and the duplicate, sample B, respectively. The total Pu-238 activity used in the test was obtained by multiplying the Pu-238 activity per gram of soil by the weight of the soil used in each test. The activity removed is recorded for each ACT*DE*CONSM wash and is expressed as a percentage of the original activity. The percentage removal of Pu-238 after three ACT*DE*CONSM washes was found to be 83.8% for sample A and 89.5% for sample B. Tables 2 and 3 also show that by using ACT*DE*CONSM washes, a DF of 11 was achieved for sample A, and a DF of 13 was achieved for sample B. The percentage recoveries for each test are shown in Figures 1 and 2 for samples A and B, respectively.

The soil remaining after treatment with the ACT*DE*CONSM process was analyzed by using the same leaching procedure as that for the spiked soil. The amount of Pu-238 found in treated soil sample A was 1.85 ± 0.2 Bq/g (50 ± 5.4 pCi/g), and the amount of Pu-238 found in treated sample B was 1.59 ± 0.1 Bq/g (42.8 ± 3.2 pCi/g). The total quantity of Pu-238 recovered in the ACT*DE*CONSM process for each sample was obtained by adding the total amount of Pu-238 in the wash solution after the ACT*DE*CONSM treatment to the amount of Pu-238 remaining on the treated soil. Each total was expressed as a percentage of the original activity — 92.8% for sample A and 97.2% for sample B.

To confirm the accuracy of the analyses, Harwell Laboratories and ANL analyzed replicate samples used in the ACT*DE*CONSM process. Table 4 lists the results obtained from each research laboratory. The results were found to be in excellent agreement.

TABLE 1 Material Balance for Spiking of Mound Soil

Sample	Total Activity of Pu-238 (Bq)	Proportion of Pu-238 (Bq/g)
Original spike on 3 g of soil	299.8 ± 5.4 (8093.3 ± 146) ^a	99.9 ± 1.8 (2697.3 ± 48.6) ^b
Contact liquor	1.5 ± 0.6 (40.2 ± 17) ^a	0.49 ± 0.25 (13.3 ± 5.7) ^b
First water wash of soil	0.48 ± 0.04 (13 ± 1.0) ^a	0.16 ± 0.01 (4.3 ± 0.4) ^b
Second water wash of soil	0.44 ± 0.025 (12 ± 0.75) ^a	0.15 ± 0.01 (4.0 ± 0.25) ^b
Third water wash of soil	0.34 ± 0.025 (9.2 ± 0.75) ^a	0.11 ± 0.01 (3.0 ± 0.25) ^b
Water wash from blending spiked soil with unspiked soil	0.60 ± 0.025 (16.4 ± 0.8) ^a	0.20 ± 0.01 (5.4 ± 0.25) ^b
Total activity in water washes	3.35 (90.5) ^a	1.12 (30.1) ^b

^a Values in parentheses are in pCi.

^b Values in parentheses are in pCi/g.

TABLE 2 Material Balance for Mound Sample A

Sample ^a	Total Activity of Pu-238 (Bq)	Proportion of Pu-238 (Bq/g)	Activity in Different Streams (%)
Spiked soil	102.9 ± 3.5 (2779.4 ± 84.4) ^b	20.51 ± 0.63 (553.9 ± 16.4) ^c	—
First ACT*DE*CON SM wash	75.7 ± 8.2 (2044.6 ± 220.5) ^b	15.1 ± 1.6 (408.2 ± 44.1) ^c	73.7
Second ACT*DE*CON SM wash	4.7 ± 1.0 (125.9 ± 26.5) ^b	0.93 ± 0.25 (25.2 ± 0.5) ^c	4.5
Third ACT*DE*CON SM wash	5.8 ± 1.3 (156.5 ± 31.5) ^b	1.15 ± 0.25 (31.0 ± 6.3) ^c	5.6
Total Pu-238 after ACT*DE*CON SM washes	86.2 (2327.0) ^b	17.2 (464.4) ^c	83.8
Treated, spiked soil	9.3 ± 1.3 (251.1 ± 35) ^b	1.85 ± 0.2 (50.0 ± 5.4) ^c	9.0
Total Pu-238 recovered	95.5 (2578) ^b	19.0 (514.3) ^c	92.8

^a Weight of soil used in the ACT*DE*CONSM test = 5.0177 g; DF (102.9 Bq/9.3 Bq) = 11.1.

^b Values in parentheses are in pCi.

^c Values in parentheses are in pCi/g.

TABLE 3 Material Balance for Mound Sample B

Sample ^a	Total Activity of Pu-238 (Bq)	Proportion of Pu-238 (Bq/g)	Activity in Different Streams (%)
Spiked soil	102.9 ± 3.2 (2779.4 ± 84.4) ^b	20.51 ± 0.63 (553.9 ± 16.4) ^c	—
First ACT*DE*CON SM wash	72.8 ± 5.5 (1966.4 ± 149.9) ^b	14.6 ± 1.1 (393.0 ± 30.2) ^c	70.8
Second ACT*DE*CON SM wash	14.5 ± 0.8 (391.2 ± 18.9) ^b	2.9 ± 0.1 (78.2 ± 3.8) ^c	14.1
Third ACT*DE*CON SM wash	4.8 ± 0.9 (129.3 ± 22.7) ^b	1.0 ± 0.1 (25.8 ± 5) ^c	4.6
Total Pu-238 after ACT*DE*CON SM washes	92.1 (2486.9) ^b	18.4 (496.9) ^c	89.5
Treated, spiked soil	7.9 ± 0.6 (213.3 ± 16.2) ^b	1.59 ± 0.1 (42.8 ± 3.8) ^c	7.7
Total Pu-238 recovered	100.0 (2701.2) ^b	20.0 (539.8) ^c	97.2

^a Weight of soil used in the ACT*DE*CONSM test = 5.004 g; DF (102.9 Bq/7.9 Bq) = 13.

^b Values in parentheses are in pCi.

^c Values in parentheses are in pCi/g.

FIGURE 1 Percentage Recovery of Pu-238 from Sample A

FIGURE 2 Percentage Recovery of Pu-238 from Sample B

TABLE 4 Comparison of the Results of Pu-238 Analyses

Sample	Results of Pu-238 Analyses, by Laboratory (Bq/g)		
	Argonne	Bradtec	Harwell
Spiked Mound soil	22.38 $\pm$ 1.28 (604.35 $\pm$ 34.62) ^a	20.51 $\pm$ 0.63 (553.9 $\pm$ 16.4)	20.3 $\pm$ 0.7 (548 $\pm$ 19)
Spiked Mound soil, duplicate	19.77 $\pm$ 1.79 (533.86 $\pm$ 48.26)	20.34 $\pm$ 0.38 (549.1 $\pm$ 10.1)	—
Treated Mound soil, Sample A	2.16 $\pm$ 0.12 (58.43 $\pm$ 3.30)	1.85 $\pm$ 0.2 (50 $\pm$ 5.4)	1.88 $\pm$ 0.08 (50.8 $\pm$ 2.2)
Treated Mound soil, Sample B	1.57 $\pm$ 0.1 (42.51 $\pm$ 2.58)	1.59 $\pm$ 0.1 (42.9 $\pm$ 3.2)	—

^a Values in parentheses are in pCi/g.

The target level of decontamination was 0.93 Bq/g. Three separate ACT*DE*CONSM washes of the soil reduced the level of contamination from approximately 20 Bq/g to 1.85 $\pm$ 0.2 Bq/g for sample A and 1.58 $\pm$ 0.1 Bq/g for sample B. Recent tests in which the ACT*DE*CONSM process was used to remove uranium and thorium from soil have shown that considerably larger DFs are achievable with four or more washes. Thus, by using a system (such as a continuous-flow system) in which the soil is successively washed with fresh ACT*DE*CONSM solvent as the contaminant is removed, the target values may be attainable. Optimization of the experimental parameters used in the ACT*DE*CONSM process for Mound soil, such as modification of the concentration of chelating and oxidizing agents used, may also prove beneficial.

4 Conclusions and Recommendations for Further Work

The results of the Phase I laboratory study indicate that the ACT*DE*CONSM process could be used to decrease the amounts of contaminants in the Mound soils to acceptable levels. Additional study is required to determine whether that potential can be realized.

The recommended approach for additional study is to perform a series of investigations designed to resolve issues significant to the deployment of the ACT*DE*CONSM process at the Mound site. These investigations would begin in the laboratory. On the basis of the advanced laboratory studies, work would progress to pilot- and full-scale demonstrations. These studies would be Phases II, III, and IV, the tasks and performance goals of which are described in the following sections. (In Phase II, tasks 1 through 8 would be carried out on spiked Mound soil, while tasks 9 and 10 would be carried out on active Mound soil.)

4.1 Phase II: Laboratory-Scale Optimization

- Task 1: Test a matrix of three different ethylenediaminetetraacetic acid (EDTA) concentrations and two different H₂O₂ concentrations on spiked Mound soil to determine the minimum effective concentrations that provide the maximum or near-maximum decontamination factor.

Performance Goal: The goal of task 1 is to reduce the soil plutonium concentration to less than 0.93 Bq/g.

- Task 2: Analyze the soil and solution samples from the final test series of task 1 to determine the dissolution of nonhazardous minerals. If appropriate, an additional test series will be run with 8-hydroxyquinoline or other material that may reduce the dissolution of nonhazardous minerals.

Performance Goal: The goal of task 2 is to limit the dissolution of nonhazardous minerals to less than 10% of the total mineral content present in the pretreated soil.

- Task 3: Evaluate spiked Mound soil to study (1) the residual concentrations of ACT*DE*CONSM chemicals in the treated soil, (2) the mobility in the residual plutonium after treatment, and (3) the ability of the soil to support vegetative growth after decontamination.

Performance Goals: The goals of task 3 are to (1) demonstrate that the residual EDTA concentration in the treated soil is less than 15 ppm; (2) demonstrate that

the mobility of the plutonium in the soil after treatment, as measured by K_d , is not significantly different from the mobility of the plutonium in the soil that has not been treated, and that the mobility after treatment is not in a range that poses a risk that greater levels of plutonium will leach into the aquifer than would be expected for the untreated soil residual; and (3) confirm that three healthy growth cycles can be supported in the sample of treated soil.

- Task 4: Evaluate the ion-exchange effectiveness of several candidate surface adsorbers for the magnetic separation of particles.

Performance Goal: The goal of task 4 is to demonstrate greater than 90% recovery of plutonium for the selected media in treating the solutions used in the ACT*DE*CONSM process.

- Task 5: Determine the optimal magnetic particle concentration and contact time for the magnetic particles used for recovery of the dissolved plutonium from the solutions used in the ACT*DE*CONSM process.

Performance Goal: The goal of task 5 is to adsorb 90% of the dissolved plutonium contained in the ACT*DE*CONSM/soil slurry by bringing the magnetic separation particles into contact with the solution in a single contact/recovery period.

- Task 6: Evaluate the appropriate regeneration chemistry for recovery and reuse of magnetic particles for plutonium adsorption.

Performance Goals: The goals of task 6 are to (1) regenerate the magnetic particles to 90% of their initial adsorption capacity and (2) confirm that no adverse effects that are attributable to the natural magnetite content of the soil are encountered in processing.

- Task 7: Evaluate waste-stabilization methods for processing the residues from the treatment process.

Performance Goals: The goals of task 7 are to (1) demonstrate in the lab that the residues can be treated to meet EPA requirements, (2) confirm that the residues can be treated at a cost of less than \$30/ft³ of soil treated, and (3) confirm that the waste product generated by treatment is less than 10% of the treated soil volume.

- Task 8: Demonstrate in a large-scale laboratory test a combined, integrated treatment process that will dissolve plutonium from soil, recover the dissolved

plutonium from the soil, regenerate and recover the plutonium from the magnetic separation particles, reuse the magnetic particles for additional plutonium recovery, and stabilize the resulting waste residue (i.e., the combination of tasks 1 through 7).

Performance Goal: The goal of task 8 is to meet each of the individual performance objectives specified during tasks 1 through 7 (not including task 3).

- Task 9: Perform a simple in-beaker dissolution test by using the optimized dissolution chemistry on an active sample from the Mound site.

Performance Goal: The goal of task 9 is to confirm the ability of the process to decontaminate actual Mound soils to less than 0.93 Bq/g.

- Task 10: After successful demonstration of the ACT*DE*CONSM process in the integrated laboratory-scale test, the test will be rerun with actual contaminated soil from the Mound site.

Performance Goal: The goal of task 10 is to meet each of the individual performance objectives specified during tasks 1 through 7 (not including task 3) with actual soil from a contaminated site.

- Task 11: Perform site hydrogeological studies and testing, if required.

Performance Goal: The goal of task 11 is to obtain sufficient information to model the site hydrogeology at the 85% confidence level in the Phase III pilot-scale (lysimeter) testing.

- Task 12: Perform magnetic-particle recovery tests by using a small production magnetic filter. Testing will be performed at a variety of unit flow rates (g/m² of filter cross-sectional area).

Performance Goal: The goal of task 12 is to demonstrate greater than 99% particle recovery with either one or two magnetic filters in series.

4.2 Phase III: Pilot-Scale Testing

- Task 1: Demonstrate that the ACT*DE*CONSM process can be used in situ to decontaminate plutonium-contaminated soil to less than 0.93 Bq/g. Active soil

from the Mound site will be placed in a lysimeter cell to allow a complete mass balance of the test "cell" to assure that no plutonium has been lost from the test system. The pilot-scale demonstration will test all aspects of the integrated treatment system, including the following: solvent distribution and contact with all soil particles, solvent recovery, plutonium dissolution, plutonium recovery by means of the magnetic separation method, magnetic particle recovery and regeneration, recovery of chemicals during cleanup/flushing, and waste processing.

Performance Goal: The goal of task 1 is to demonstrate the ability to meet the performance objectives established for tasks 1 through 7 (not including task 3) of the Phase II study while a pilot-scale in-situ test is performed.

- Task 2: Test the confinement characteristics of the Cryocell™ cryogenic barrier.

Performance Goal: The goal of task 2 is to demonstrate the ability of the cryogenic barrier to provide zero leakage across the barrier boundary.

4.3 Phase IV: Full-Scale Demonstration

- Demonstrate that the ACT*DE*CONSM process can be used to treat plutonium-contaminated soil in situ. The soil will be contained in a "cell" by means of Cryocell™ Freeze Barrier technology. The purpose of the cell is to contain the soil and ACT*DE*CONSM solvents and to prevent the migration of plutonium to uncontaminated areas. The full-scale demonstration will include all aspects of the integrated treatment system, including the following processes: freeze barrier isolation of the treatment area, solvent distribution and contact with all soil particles, solvent recovery, plutonium dissolution, plutonium recovery by means of the magnetic separation method, magnetic particle recovery and regeneration, recovery of chemicals during cleanup/flushing, and waste processing.

Performance Goal: The goal of Phase IV is to decontaminate the soil, in situ, to less than 0.93 Bq/g while meeting each of the performance objectives established for Phase II, tasks 1 through 7 (not including task 3), and meeting the performance goal for the cryogenic barrier task of Phase III.

Phases III and IV will need to be integrated with one of the DOE Integrated Demonstration projects so that (1) technologies necessary for this work that have already been demonstrated can be used and (2) technologies that are demonstrated for the first time as part of this project can be made available to other DOE efforts.

Appendix A:
External Analysis Reports

PILAMEC

PILAMEC LIMITED
UNIT 18
LYDNEY INDUSTRIAL ESTATE
LYDNEY GLOS. GL15 4EJ
ENGLAND

Tel: Dean (0594) 843660 Telegrams: Pilamec

Bradtec Ltd,
Bristol Polytechnic,
Coldharbour Lane,
BRISTOL,
BS16 1QY

For Attention of Mr G Elder

Date 16 December 1991

EWP/LVM

Your ref

REPORT ON PREPARATION OF SOIL SAMPLE

Initial visual inspection of the soil showed a very high moisture content. Subsequent sieving of the material would have led to blinding of the mesh.

Therefore, in consultation with customer, it was agreed that we should thoroughly dry material before preparing sample. A laboratory oven, pre-heated to 65°C, was used to dry the sample.

Large clods of soil were broken down manually and further size-reduction was effected by gently rolling out the coarser material.

Initial sieving through a 6mm mesh removed coarse aggregates, whilst the final sample was prepared by sieving through a 400#(38 micron) screen and 'cone and quartering' the fine dust.

END RESULT: 1kg representative sample of fine soil particles.

Compiler: E W Pemberton

Director: E W Pemberton

Registered in England No. 1258472 Regd. Office: 4 Cinema Buildings, Eagle Road, Burnham-on-Sea, Dorset BH4 9DW

C. J. Associates Geotechnical Ltd.

Particle Size Distribution

Site - 119.18.05

Job Number - B1237

Sample :-

Date: - 13/ 9/91

[illegible]

BS1377 : Part 3 : 1990 Method 3
Determination of the organic matter content

Site 119.19.05
Job Number B1237

Sample Reference BH	Sample Depth [m]	Passing 2mm [%]	Organic Matter [%]
TPC	MOUND	100	6.4

British Rail research

Scientific Services
British Rail Research
P.O. Box 2
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England
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Bradtech Ltd.
Science Loading Bay
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F.A.O. S.M. Scrivens

Ref : Order No 413

Analytical Services Unit
Hartley House
London Road
Derby
DE2 8YB

Tel 0332-386624

Ref 241-11-13

Date 11/1/92

ANALYSIS OF SOIL SAMPLES AND SOLUTIONS

An analysis has been completed (by Inductively Coupled Plasma) on the three soil samples and four solutions submitted in December. The soil samples were digested as on the previous occasion. All of the results are set out below. As before the actual results are quoted for your convenience where a "less than" figure is given but please bear in mind the comments made by my colleague Neil Fearn in his report to you on 22nd November.

1. Soil Samples

		Sample 1	Sample 2	Sample 3
Uranium	(mg/Kg)	50	<50 (25)	<50 (42)
Thorium	(mg/Kg)	<25 (<5)	<25 (<5)	<25 (<5)
Iron	(mg/Kg)	21400	30500	13000
Cobalt	(mg/Kg)	11	16	7
Copper	(mg/Kg)	9	50	10
Zinc	(mg/Kg)	33	163	54
Manganese	(mg/Kg)	580	800	330
Sodium	(mg/Kg)	<1	<1	
Calcium	(mg/Kg)	20300	24600	50100
Magnesium	(mg/Kg)	6800	9100	16800
Lead	(mg/Kg)	<1	41	
Cadmium	(mg/Kg)	4	5	

NATIONAL PHYSICAL LABORATORY

Teddington Middlesex TW11 0LW England

Certificate of Measurement
OF A
PLUTONIUM-242
LOW-LEVEL STANDARD OF RADIOACTIVITY

DESCRIPTION: ^{242}Pu in the form of a solution in 8 M nitric acid.
Supplied as 10 g nominal of solution in a flame-sealed 10 ml British Standard ampoule.

IDENTIFICATION: Samples E2061 to E2110
Code R15-00

MEASUREMENTS

The solution was prepared by gravimetric dilution of a sample which had been assayed by 4π -alpha liquid scintillation counting. The accuracies of dilution factors were checked by the use of a liquid scintillation counter.

RESULTS

^{242}Pu activity concentration 0.999 Bq/g at 0000 GMT 1 January 1992.

UNCERTAINTIES

Overall uncertainty $\pm 4.56\%$.

Random uncertainty $\pm 0.72\%$ for 3 degrees of freedom.

Non-random uncertainty $\pm 4.51\%$ for components added in quadrature.

The uncertainties are at the 95% confidence level and, except where stated otherwise, the number of degrees of freedom is taken to be infinite.

REFERENCE R986

PAGE 1 OF 3

DATE 1 February 1992

SIGNED

for Director

CHECKED

This Certificate may not be published except in full, unless permission for the publication of an approved extract has been obtained in writing from the Director. It does not of itself impute to the subject of measurement any attributes beyond those shown by the data contained herein.

Amersham International plc
Amersham Laboratories

CALIBRATION
No 0146

Certificate of calibration of absolutely standardized radioactive solution

Description	Principal radionuclide: Pluton m-238	Product code: PPZ.24
		Solution number: S1/25/34
Measurement	<p>Reference time: 1200 GMT on 1 July 1991</p> <p>Radioactive concentration of plutonium-238: 3.974 kilobecquerels per gram of solution which is equivalent to: 107.4 nanocuries per gram of solution</p> <p>Mass of solution: 5.1214 grams</p> <p>Total activity of plutonium-238: 20.35 kilobecquerels which is equivalent to: 550.0 nanocuries</p> <p>Method of measurement used (see reverse of the certificate): B</p>	
Accuracy	<p>Overall uncertainty in the radioactive concentration quoted above: $\pm 1.34 \%$</p> <p>Random uncertainty: $\pm 0.71 \%$ Systematic uncertainty: $\pm 0.63 \%$</p> <p>Overall uncertainty is defined on the reverse of the certificate.</p>	
Radionuclidic Purity	<p>The estimated activities of any radioactive impurities are listed below expressed as percentages of the activity of the principal radionuclide at the reference time.</p> <p>Other alpha emitters less than 0.004 %</p>	
Chemical Composition	Carrier free in 0.5M nitric acid.	
Physical Data	<p>Recommended half life: 87.7 ± 0.7 years</p> <p>100% alpha particle emission.</p>	
Remarks	<p>The isotopic purity of the plutonium-238 is greater than 99.9% as determined by mass analysis.</p>	

This product meets the quality assurance requirements of NRC Regulatory Guide 4.15 for achieving implicit NIST traceability as defined in NCRP 58 (1985).

Approved
Signatory

G.D.M. Parker

Page 1 of 1

Amersham

This certificate is issued in accordance with the conditions of accreditation granted by the National Measurement Accreditation Service, which has assessed the measurement capability of the laboratory and its traceability to recognised national standards and to the units of

Methods of measurement

The measurement techniques listed below are currently in use at Amersham International for the absolute standardization of radioactive solutions. The methods used for this standardization are indicated on the front of the certificate.

Using a gas flow proportional counter

- A 4 π beta counting
- B 4 π alpha counting
- C 4 π internal conversion electron counting
- D 4 π coincidence counting
- E 4 π anticoincidence counting
- F 4 π coincidence and anticoincidence counting

Using a liquid scintillation counter

- G 4 π coincidence counting
- H 4 π anticoincidence counting
- J 4 π coincidence and anticoincidence counting
- K 4 π efficiency tracing

S.I. unit of radioactivity

The S.I. unit of radioactivity is the becquerel

- 1 becquerel (Bq) = 1 nuclear transformation per second, therefore
- 1 curie (Ci) = 3.7×10^{10} becquerels exactly.

Useful conversion factors are:

- 1 microcurie (μ Ci) = 3.7×10^4 Bq = 37 kilobecquerels (kBq)
- 1 millicurie (mCi) = 3.7×10^7 Bq = 37 megabecquerels (MBq)
- 1 kilobecquerel (kBq) = 27.027 nanocuries (nCi)
- 1 megabecquerel (MBq) = 27.027 microcuries (μ Ci)

Overall Uncertainty

The overall uncertainty was calculated in accordance with the recommendations of the International Commission on Radiation Units and Measurements (ICRU Report 12). The limits of uncertainty were taken as the arithmetic sum of the uncertainty due to random variations, calculated at the 99.7% confidence level, and the estimated systematic uncertainties in the measurement.

**AEA ENVIRONMENT
& ENERGY****AEA TECHNOLOGY**

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Dr K J Atkins
Bradtech Ltd
Bristol Polytechnic
Coldharbour Lane
Bristol
BS16 1OY

21 February 1992

Dear Karen

On 17/2/92 I received from you an intact package containing two plastic vials. The vials were custody sealed on 14/2/92 and their seals were intact. The samples were logged into the sample records system and each was given a unique number. The custody seals were broken to remove sub-samples for analysis.

The results of the ^{238}Pu determinations are shown on the attached Table.

I hope these results are satisfactory. Please sign and return the enclosed B/8 form to Joe.

With Best Wishes.

Dr R Carpenter

Enc.

Plutonium-238 in Soil

Our Code	Your Code	Description	^{238}Pu Bq g ⁻¹
D1272	Sample A	Mound Soil. 3x treated	1.88 ± 0.08
D1273	Sample C	Mound Soil. Spiked and blended	20.3 ± 0.7

Notes

1. The uncertainties represent 2 counting statistics

21/2/92

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material: Mound Soil

Date Received: 2/3/92

Submitted by: Bradtec, Inc.

Date Reported: 3/9/92

Submitter's Sample No.	ACL Sample No.	Sample Leached (g)	^{238}Pu (pCi/g $\pm 2\sigma$)
Untreated Mound Soil	92-8030-01	0.5790	<0.09
	-01R	0.5221	<0.08
Spike Mound Soil	-02	0.2030	604.35 $\pm$ 34.62
	-02R	0.2115	533.86 $\pm$ 48.26
Treated Mound Soil - Sample A	-03A	0.4929	58.43 $\pm$ 3.30
	-03B	0.4969	42.51 $\pm$ 2.58
Treated Mound Soil - Sample B	-03B	0.4969	42.51 $\pm$ 2.58
	-03B	0.4969	42.51 $\pm$ 2.58
<u>QA/QC Samples</u>			
^{238}Pu (pCi/L $\pm 2\sigma$)			
	92-8030-BLK		<0.15
	-BLK 2		<0.30

NOTE: Samples will be discarded one (1) month after date of report unless otherwise arranged. When making future inquiries regarding this work, you must reference ACL sample number(s) above. For further information about the results reported here, please call L. Smith, R. Heinrich at 2-7521, 4346.

Reference(s): Notebook No. 36863-01, pp. 127-130.

Copies To: Dr. K. Atkins, Bradtec R. Heinrich Analyst(s): L. Smith

Dr. D. Bradbury, Bradtec L. Smith

/vts Dr. G. Elder, Bradtec File

CMT-84 (4-91) D. Green

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material: Mound Soil

Date Received: 2/3/92

Submitted by: Bradtec, Inc.

Date Reported: 3/9/92

Submitter's Sample No.	ACL Sample No.	^{239}Pu (pCi $\pm 2\sigma$)	
		Laboratory Determined Value	Accepted Value
	92-8030-RFS	0.196 $\pm$ 0.016	0.217 $\pm$ 0.010
	-SPK	0.389 $\pm$ 0.042	0.382 $\pm$ 0.024
NOTE 1: Designation XX-XXXX-XXR refers to replicate sample.			
NOTE 2: Uncertainties (σ) based upon propagation of counting statistical error. Less than (<) values are 3.29σ .			
NOTE 3: Sample 92-8030-BLK processed with customer samples 92-8030-01, 92-8030-03. Sample 92-8030-BLK2 processed with sample 92-8030-02. Reagent blanks consisting of mixed nitric and hydrochloric acids.			
NOTE 4: Sample 92-8030-RFS prepared from NIST SRM 4353: "R F Soil No. 1."			
NOTE 5: Sample 92-8030-SPK composed of liquid control prepared by ACL Analyst.			

NOTE: Samples will be discarded one (1) month after date of report unless otherwise arranged. When making future inquiries regarding this work, you must reference ACL sample number(s) above. For further information about the results reported here, please call L. Smith, R. Heinrich at 2- 7521, 4346.
Reference(s): Notebook No. 36963-01, pp. 127-130.

Copies To:

Analyst(s):

See Page 1.

A D A S

Bradtec
Bristol Polytechnic
Coldharbour Lane
BRISTOL
AVON
BS16 1QY

Your reference

Our reference

Date 09/04/92

ANALYTICAL CHEMISTRY DEPARTMENT

ANALYSIS REPORT

Lab sample no: 384019 Date recd: 30/03/92
Batch number: 00078193 Your ref: Mound Soil
Sample type: Soil (agricultural)

Determination	Result	Units
pH - H2O extractable	7.1	
Cation exch capacity	16.0	me/100g
Calcium carbonate	9.7	%
Sodium - exchange'le	< 0.1	me/100g
Calcium - exchange'le	12.5	me/100g
Magnesium-exchang'le	2.6	me/100g
Potassium-exchang'le	< 1.0	me/100g
Manganese - exch'le	< 0.5	me/100g

COMMENTS:

The Calcium Carbonate result refers to the total carbonates present in the sample expressed as calcium carbonate.

Signed: K I F Way
Duty Analyst

Ministry of Agriculture, Fisheries and Food
Agricultural Development and Advisory Service
Staple Mount, Starcross
Exeter EX6 4PE

Tel: 0626 890481
GTN: 1377 ext.
Fax: 0626 891254

Addendum to report 04/92 - Plutonium-238 recovery from Mound soil using ACT*DE*CON

In accordance with Argonne National Laboratories - work order number 1, data for pH, Eh, carbonate content and cation exchange capacity is as follows:

1. pH = 7.1
2. Eh = 265 ± 2 mV
3. Calcium carbonate content = 9.7%
4. Cation Exchange Capacity = 16.0 meq/100g

Note: Analyses 1, 3 and 4 were performed by the Ministry of Agriculture, Fisheries and Food (MAFF), Agriculture Development and Advisory Service, Staplake Mount, Starcross, Exeter, EX6 8PE, UK. Eh analysis

of the Mound soil was performed by Bradtec Ltd in accordance with standard analytical methods. A 1:1 weight ratio of soil in water was analysed using a combination platinum electrode referenced with a silver/silver chloride electrode.

Appendix B:

**Recovery of Pu-238 from Mound Soil by Means of
the ACT*DE*CONSM Process: Soil Spiking
and Pu-238 Dissolution**

Appendix B:**Recovery of Pu-238 from Mound Soil by Means of
the ACT*DE*CONSM Process: Soil Spiking
and Pu-238 Dissolution****B.1 Soil Preparation[†]**

1. Take 3 g of the Mound soil that has been dried at <70°C and sieved. The soil fraction that was less than 38 µm should be used.

Weight of soil and beaker: 68.51742 g

Weight of beaker: 65.50832 g

Weight of soil: 3.0091 g

2. Suspend the soil in 2.5 mL of 0.1 N HNO₃ and 6 mL of water. Stir the solution.
3. Add 1500 µL of Pu-238 standard (corresponding to 300 Bq) one drop at a time to a 0.5 N solution of HNO₃ while the solution is constantly stirred. The molarity of the final HNO₃ solution will be 0.1.
4. Leave the solution stirring and in contact with the Pu-238 until the solution is required.

Start: 1045 h

Finish: 1415 h

Contact time: 3.5 h

5. Filter the solution through a 2-µm glass-fiber filter paper.
6. Retain the original soak liquor.

Volume of liquor: 16.7 mL

[†] Completion of soil preparation witnessed by Robert Heinrich, Argonne National Laboratory.

7. Add 25.3 mL of concentrated HNO_3 to the original soil soak liquor plus 1 mL of Pu-242 tracer in 8 M HNO_3 . Place the sample to one side for analysis. Make up to 50 mL of solution with water prior to analysis to yield a final acid concentration of 8 M HNO_3 .
8. Wash the soil three times with 10 mL of 18 MΩ water.
9. Filter the suspension and retain each wash solution. Record the volume of each.

Volume of first wash: 15.0 mL

Volume of second wash: 9.8 mL

Volume of third wash: 10.3 mL

10. Add 12.7 mL of concentrated HNO_3 to each of the samples. Add 1 mL of Pu-242 tracer in 8 M HNO_3 to the third wash solution. The samples should then be made up to 25 mL with water prior to analysis. Samples over 12 mL in volume should have 25.3 mL of concentrated HNO_3 added and should be made up to 50 mL of solution with water prior to analysis. The final acidity of these samples will then be 8 M HNO_3 .
11. Transfer the spiked soil to a 200-mL beaker with 50 mL of water.
12. Add 12 g of unspiked Mound soil to the beaker.

Weight of soil and beaker: 15.59429 g

Weight of beaker: 3.59888 g

Weight of soil: 11.99541 g

13. Thoroughly mix and stir the soils together.
14. Divide the solution and place it into 50-mL centrifuge tubes and spin the tubes at 3000 revolutions per minute (rpm) for 10 min.
15. Filter the supernatant. Retain this first wash solution.

Volume of wash: 66 mL

16. Add 50 mL concentrated HNO_3 to this wash solution in a plastic bottle. Add 1 mL Pu-242 tracer and make up to 100 mL of solution with water prior to analysis.

17. Dry the soil at $<70^\circ\text{C}$ and weigh separately three lots of soil.

18. Place one aliquot of soil in a reaction flask: sample A

Weight of flask and soil: 6.76059 g

Weight of flask: 1.74290 g

Weight of soil: 5.01769 g

19. Place a second aliquot of soil in another reaction flask: sample B

Weight of flask and soil: 7.43674 g

Weight of flask: 2.43277 g

Weight of soil: 5.00397 g

20. Place the third aliquot of soil in a sample vial: sample C

Weight of flask and soil: 9.35878 g

Weight of flask: 4.97619 g

Weight of soil: 4.38259 g

21. Remove a 1-g aliquot of soil from the third aliquot for the Argonne Analytical Chemistry Laboratory.

Weight of bottle and soil: 6.11462 g

Weight of bottle: 5.10785 g

Weight of soil: 1.00677 g

22. Weigh a second 1-g aliquot of the soil sample C for an acid digest.

Weight of bottle and soil: 3.35396 g

Weight of bottle: 2.35452 g

Weight of soil: 0.99944 g

23. Add 1 mL of Pu-242 tracer to the second 1-g soil aliquot and leach the soil by using 20 mL of ANALAR-grade nitric acid and 5 mL of ANALAR-grade hydrochloric acid. After a 1-h digest period, add 5 mL of nitric acid and 2 mL of hydrochloric acid.

24. Digest the soil for several hours.

Date: December 18, 1991

Start Time: 1730 h

Finish: 2000 h

Digest Time: 2.5 h

25. Filter the soil sample through a 2- μ m filter.
26. Wash the soil three times with 8 M HNO₃.
27. Transfer the leachate plus washings to an evaporating dish and evaporate the material to dryness.
28. Resuspend the solid in 8 M HNO₃. Retain the solution for analysis.
29. Retain the soil and dry it at <70°C.

B.2 Dissolution Procedure — Outline Form[†]

1. Suspend samples A and B separately in the chemicals used for the ACT*DE*CONSM process. Stir the solutions thoroughly.
2. Record the time of the first dissolution reaction as time zero.
3. Allow the soil suspensions to stir for 1 h.
4. At 1 h, remove a 10-mL sample from each soil sample.
5. Immediately filter both solutions. Record the volume of each sample.
6. Transfer each sample to a scintillation vial. Add 12.7 mL of concentrated HNO₃ and 1 mL of Pu-242 tracer in 8 M HNO₃ to each solution and keep the solutions for analysis.
7. Divide the remainder of the soil solutions from samples A and B into centrifuge tubes and spin the tubes at 3000 rpm for 10 min. Filter the supernatant from the first dissolution liquor for each sample.
8. Record the volume of each solution.
9. Add 127 mL of concentrated HNO₃ to each solution. Add 1 mL of Pu-242 tracer and make up to 250 mL of solution prior to analysis.
10. Resuspend the soil samples in 100 mL of ACT*DE*CONSM solution. Stir the suspension thoroughly.
11. Record the time of the second dissolution reaction as time zero.
12. Allow the soil suspension to stir for 1 h.
13. At 1 h, remove a 10-mL sample from each soil sample.
14. Immediately filter both solutions. Record the volume of each sample.

[†] ACT*DE*CONSM dissolution witnessed by Robert Heinrich, Argonne National Laboratory.

15. Transfer each sample to a scintillation vial. Add 12.7 mL of concentrated HNO₃ and 1 mL of Pu-242 tracer in 8 M HNO₃ to each sample and keep the solutions for analysis.
16. Divide the remainder of the soil solutions from samples A and B into centrifuge tubes and spin the tubes at 3000 rpm for 10 min. Filter the supernatant from the second dissolution liquor for each sample.
17. Record the volume of each solution.
18. Add 127 mL of concentrated HNO₃ to each solution. Add 1 mL of Pu-242 tracer and make up to 250 mL of solution with water prior to analysis.
19. Resuspend the soil samples in 100 mL of ACT*DE*CONSM solution. Stir the suspension thoroughly.
20. Record the time of the third dissolution reaction as time zero.
21. Allow the soil suspension to stir for 1 h.
22. At 1 h, remove a 10-mL sample from each soil sample.
23. Immediately filter both solutions. Record the volume of each sample.
24. Transfer each sample to a scintillation vial. Add 12.7 mL of concentrated HNO₃ and 1 mL of Pu-242 tracer in 8 M HNO₃ to each sample and keep the solutions for analysis.
25. Divide the remainder of the soil solutions from samples A and B into centrifuge tubes and spin at 3000 rpm for 10 min. Filter the supernatant from the third dissolution liquor for each sample.
26. Record the volume of each solution.
27. Add 127 mL of concentrated HNO₃ to each solution. Add 1 mL of Pu-242 tracer prior to analysis and make up to 250 mL of solution with water prior to analysis.
28. Dry each of the soil samples at <70°C.
29. Record the weight of each leached soil sample.

30. Remove a 1-g aliquot of soil from each sample for Argonne National Laboratory.
31. Transfer the remainder of each soil sample to a 250-mL beaker.
32. Leach 1 g of each of the soil samples by using 20 mL of ANALAR-grade concentrated HCl. Spike each of the soils with 1 mL of Pu-242 in 8 M HNO₃.
33. After 1 h, add 5 mL of concentrated HNO₃ and 2 mL of concentrated HCl.
34. Digest the soils for several hours.
35. Filter the soil samples through a 2- μ m filter.
36. Wash the soil three times with 8 M HNO₃.
37. Transfer the leachate plus washings to an evaporating dish and evaporate the material to dryness.
38. Resuspend each solid in 8 M HNO₃ and add 1 mL of Pu-242 tracer. Retain each solution for analysis.
39. Retain the soil and dry it at <70°C.
40. Archive the remainder of the soil.

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