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URANIUM SOILS INTEGRATED DEMONSTRATION: PHASE II PLAN FERNALD CRU5/USID SOIL DECONTAMINATION PILOT PLANT (CARBONATE/CITRATE LEACHING)

03/29/94

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ORNL/TM-12673

URANIUM SOILS INTEGRATED DEMONSTRATION: PHASE II PLAN

Fernald CRU5/USID Soil Decontamination Pilot Plant (Carbonate/Citrate Leaching)

October 1, 1993

U.S. Department of Energy
Office of Technology Development
Fernald Site Office

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EXECUTIVE SUMMARY

To help meet the Department of Energy (DOE) cleanup goals of the nuclear waste sites such as the Fernald Environmental Management Project (FEMP), the Director of Environmental Restoration and Waste Management initiated the Office of Technology Development (OTD). One program initiated by OTD was the Integrated Demonstration (ID) of technologies program. The ID program focuses on improving/validating technologies by demonstrating effectiveness, cost savings, risk reduction potential, site applicability, and regulatory and public acceptance. Once such technologies have been demonstrated to embody the above criteria, efforts are then made for the technologies to be transferred for implementation throughout the DOE and the private sector.

One of the major problems facing the DOE Environmental Restoration Program is the remediation of uranium-contaminated soils. In response to this problem, OTD initiated the Uranium Soils ID program to evaluate and compare the versatility, efficiency, and economics of various technologies for the characterization and remediation of uranium-contaminated soils. The Fernald Environmental Management Project (FEMP) was selected as the host site for the Uranium Soils Integrated Demonstration (ID hereafter) program based on its past operating history and known environmental problems. In support of the ID program, pilot scale soil leaching will be evaluated as a viable technology for remediation of contaminated soils at FEMP.

As part of the remedial investigation/feasibility study (RI/FS) for the Fernald facility, treatability studies directed at leaching uranium from several soil samples from the Fernald site were conducted by the International Technology Corporation (IT). Also, as part of the remedy selection process of the RI/FS, the FERMCO CERCLA[a]/RCRA[b] Unit 5 (CRU5 - the technical strategy adopted by the CERCLA program divides the site into 5 distinct CRUs) constructed and installed a soil decontamination pilot plant (SDPP) at Fernald. FERMCO CRU5 is currently performing batch leaching studies using the SDPP with the support of IT as the final phase of the RI/FS.

The Uranium Soils ID through the Oak Ridge National Laboratory (ORNL) has also carried out bench-scale studies on the leaching of uranium from soils. These studies have included tests with various leachants and pretreatment/leachant

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[a] Comprehensive Environmental Response, Compensation, and Liability Act

[b] Resource Conservation and Recovery Act

combinations. Also, the effect of attrition scrubbing has been examined. In these tests, attrition scrubbing and leaching were carried out simultaneously as well as in sequence. The results showed that attrition scrubbing had a beneficial effect and that high leaching efficiencies could be obtained at residence times on the order of minutes rather than hours. The results of the ORNL studies are summarized in Section 2.3 of this document. After completion of the CRU5 work, modifications will be made to the SDPP to conform to a process configuration that is based on the bench-scale results. Uranium leaching tests with Fernald soils will then be conducted by FERMCO and ORNL personnel.

The pilot scale work of CRU5 and the ID was integrated in 1991 to avoid the expense for the construction of two separate soil washing facilities. The integration of the two program's operations has been divided into three phases. These are:

PHASE I — This phase is the FERMCO CRU5 portion of the pilot plant decontamination studies, cited above. This phase included the SDPP initial constructions and start up.

PHASE II — This phase will be conducted by the ID group with the support of ORNL, CRU5, and the FERMCO treatability group. The tests will be batch leaching studies in a modified batch processing mode. The current SDPP equipment will remain the same; routing and the sequence of use of the equipment will be the process modifications. This demonstration plan addresses how the PHASE II CRU5/ID batch soil leaching treatability studies will be conducted. Data from this phase will be made available to CRU5 and may be used in the RI/FS.

PHASE III — This phase will also be conducted by the ID group, with the same supporting organizations as Phase II. The tests will be leaching studies with the SDPP configuration modified to run in a continuous soil processing mode. This plan is not intended to be used for the conduct of the Phase III studies. Its reference is made only to help the audience for this document obtain an understanding of the overall integration of the CRU5/ID soil leaching treatability studies.

The purpose of the tests on the SDPP is to assess the performance of selected leaching media and engineering process design for removal of uranium from CRU5 soils in a pilot-scale unit. The ultimate goal of this project is to develop data on the efficiency and operability of the soil decontamination process that can be used for the design of a full-scale system. For this goal to be accomplished, there are several specific project objectives to be satisfied.

- The soil leaching process should produce a soil that has an uranium concentration level equal to or less than 52 ppm, an initial technology screening level adopted by the Uranium Soils ID.

- The soil leaching process should also represent an "earth friendly approach" to the treatment of soil in that the process should decontaminate the soil without seriously degrading the soil's physicochemical characteristics (that might require its management or disposal as a waste) or generating a secondary uranium waste form that is difficult to manage and/or dispose.

- The results of bench-scale soil leaching tests should be further demonstrated at the pilot scale. These results will be useful for full-scale application of the technology. This objective will be met by operation of the SDPP, first in a batch mode (in Phase II of the demonstration) and then, after modifications, in a continuous mode (in Phase III of the demonstration).

- The results of the CRU5/ID Phases II and III will be shared with CRU5 and may be incorporated in the CRU5 RI/FS if data is available in time.

The SDPP will be operated in batch and continuous modes to demonstrate the removal of uranium from two contaminated soils from the Fernald site. These soils were taken from near the Fernald waste incinerator and near the Fernald plant 1 storage pad.

In the Phase II batch operating mode, leaching tests will be run using up to four different leaching treatments on each of the two Fernald soils. A leaching treatment will involve either a one-step or a two-step process, as follows:

- One-step - After preliminary processing steps of screening and attrition scrubbing, the soil will be subjected to chemical leaching. The leaching agents to be used include (1) sodium carbonate/sodium bicarbonate, (2) citric acid and (3) sulfuric acid.

- Two-step - Again after preliminary processing steps, the soil is first pretreated to remove iron and aluminum sesquioxides from soil surfaces and then is subjected to further chemical leaching with a sodium carbonate solution. In the pretreatment step, sodium citrate-sodium bicarbonate-sodium dithionite (CBD) is used for oxide removal. The sodium carbonate/sodium bicarbonate combination will be used for the subsequent leaching step.

Because of time limitations on the Phase I RI/FS work, ORNL and the ID team have agreed to complete some limited tests with sulfuric acid to fulfil any potential data gaps.

In Phase III of the demonstration, the SDPP will be run in the continuous mode, using the most effective leachant as determined from these batch tests, to demonstrate the operability of the soil leaching process and to obtain additional data on process efficiency.

This plan also addresses the quality assurance, data management, and health and safety issues related to the performance of this demonstration program. In general, the FEMP project plans for quality assurance, data management, and health and safety were adopted by this program.

The recovery and treatment of process streams from soil washing is part of the ID charter. These streams include the spent leaching and rinse solutions. These solutions will be treated to recover the uranium that was leached from the soil. The recovered uranium will then be handled as a low level waste and the liquid streams will be given additional treatment, if necessary, before recycle to the process or discharge. The treatment of these effluents (spent leaching and rinse solutions) will be done at a later date as part of the overall ID program. This plan specifically does not address treatment and disposal requirements for secondary waste and uranium. Process water generated as part of this study will be treated in the FEMP water treatment system and discharged. The recovered uranium will be drummed and stored as a low level waste. Decontaminated soils will be stored for further testing by the ID. Other waste will be stored for use in the ID waste treatment studies.

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GLOSSARY

ASL	Analytical Support Level
CAA	Clean Air Act
CBD	Sodium citrate-sodium bicarbonate-sodium dithionite
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CRU	CERCLA/RCRA Unit
CWA	Clean Water Act
DOE	Department of Energy
DQO	Data Quality Objective
FEMP	Fernald Environmental Management Project
FERMCO	Fernald Environmental Restoration and Management Corporation
FFCA	Federal Facilities Compliance Agreement
FMPC	Feed Materials Production Center
ID	Integrated Demonstration
IDC	Integrated Demonstration Coordinator
IT	International Technology Corp.
mtu	metric tons of uranium
NPDES	National Pollution Discharge Elimination System
NRC	Nuclear Regulatory Commission
ORNL	Oak Ridge National Laboratory
OTD	Office of Technology Development
ppm	parts per million by weight (mg/kg)
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RI/FS	Remedial Investigation/Feasibility Study
SARA	Superfund Amendments and Reauthorization Act
SCQ	Sitewide CERCLA Quality Assurance Project Plan
SDPP	Soil Decontamination Pilot Plant
SLS	Solids/Liquid Separation
USEPA	Environmental Protection Agency
USID	Uranium Soils Integrated Demonstration

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1. INTRODUCTION

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1.1 Document Purpose

The purpose of this document is to describe the plan for the soil decontamination demonstration to be conducted at the Fernald Site by the Fernald Environmental Restoration and Management Corporation (FERMCO) and the Oak Ridge National Laboratory (ORNL). This demonstration, which will begin in the third quarter of FY93, involves the removal of uranium from contaminated soil at CERCLA/RCRA Unit 5 (CRU5). This demonstration plan is provided to ensure that the requirements of the Fernald Site Integrated Demonstration (ID) program are met, as well as all environmental, safety, and health requirements of the site.

1.2 Integrated Demonstration Project Description

In order to help meet the Department of Energy (DOE) cleanup goals of the nuclear waste sites such as the Fernald Environmental Management Project (FEMP), the Director of Environmental Restoration and Waste Management initiated the Office of Technology Development (OTD). One program formed by OTD was the ID program. The ID program focuses on improving/validating technologies by demonstrating effectiveness, cost savings, risk reduction potential, site applicability, and regulatory and public acceptance.

The results of the ID will be transferred for implementation throughout the DOE and the private sector. Technology transfer involves the transfer of technological information outside the DOE system (i.e., the private sector, United States Environmental Protection Agency [USEPA], Department of Defense, etc.). The DOE strategy for technology transfer, as noted in SEN-30-91, is to increase DOE and industrial participation in an accelerated process. OTD headquarters (EM-55) will administer directives to ensure that technological data, reported in weekly and monthly internal reports, are distributed and disseminated outside the DOE system. Vehicles for the transfer of information include, but are not limited to, publications, presentations, and information exchange conferences.

The FEMP was selected to host an ID program. The FEMP, previously known as the Feed Materials Production Center (FMPC), is a contractor-operated federal facility for the remediation of a site which produced high purity uranium metal for the DOE. As such, this ID program, known as the Uranium Soils Integrated Demonstration (USID) program, will address the issues from "cradle to grave" surrounding the characterization and remediation of uranium contaminated soils, specifically soils of a high clay/silt content. Reduction of contaminated soil quantity,

through the coupling of real time analysis and precise excavation, along with decontamination techniques applicable in high clay/silt soils will be the major technology areas. Secondary waste reduction, cost optimization, risk reduction, and user applicability are other important elements in this ID.

Innovative technologies applicable to the characterization and remediation of uranium contaminated soils will be investigated throughout the DOE National Laboratory network, private industry, and universities. The ID program has a life expectancy of approximately five years with results feeding directly into the FEMP remediation program. Community relations activities shall be conducted as part of the ID in conjunction with the community relation activities currently ongoing under the FEMP Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) program. Information on treatability studies conducted within the ID will be conveyed to the public through community meetings and publications.

Soil leaching is one of the technologies identified as a viable option for remediation of contaminated soils at the Fernald, Ohio, facility of DOE.[1] As part of a remedial investigation/feasibility study (RI/FS) for the Fernald facility,[2] lab-scale treatability studies directed at leaching uranium from several soil samples from the Fernald site were conducted by the International Technology Corporation (IT). Also, as part of the remedy selection process of the RI/FS, FERMCO, the FEMP facility contractor, constructed a soil decontamination pilot plant (SDPP). This pilot plant facility includes several engineering unit operations such as size separation, particle attrition, reaction, filtration, and centrifugation, along with various pumps, and storage and holding tanks. The FERMCO CRU5 recently installed the SDPP at Fernald and FERMCO with the support of IT carried out pilot-scale leaching studies on Fernald soils using leaching agents selected from the treatability studies.

In support of the ID, ORNL has also been involved in studies on the leaching of uranium from soils.[3,4] The results of these studies are summarized in Section 2.3. Based on these results, leaching agents have been identified for testing on the SDPP after completion of the RI/FS studies by FERMCO and IT. These leaching tests are to be conducted by Fernald and ORNL personnel using Fernald soils.

The pilot-scale work of CRU5 and the ID was integrated in 1991 to avoid the expense for the construction of two separate soil decontamination facilities. The integration of the operations of the two programs has been divided into three phases. These are:

PHASE I — This phase is the FERMCO CRU5 portion of the pilot plant decontamination studies, detailed above. This phase included the SDPP initial constructions and start-up.

PHASE II This phase will be conducted by the ID group with the support of ORNL, CRU5, and the FERMCO treatability group. As in Phase I, batch leaching tests will be performed on the SDPP. However, process modifications will be made for Phase II operation. The current SDPP equipment will remain the same; routing and the sequence of use of the equipment will be the process modifications. This demonstration plan addresses how the PHASE II CRU5/ID batch soil leaching studies on the SDPP will be conducted. Data from this phase will be made available to CRU5 and may be used in the RI/FS.

PHASE III This phase will also be conducted by the ID group, with the same supporting organizations as in Phase II. The tests will be leaching studies with the SDPP modified to run in a continuous soil processing mode. This demonstration plan is not intended to be used for the conduct of the Phase III studies. Its reference is made only to help the audience for this document obtain an understanding of the overall integration of the CRU5/ID soil leaching studies.

1.3 Host Site Description

The Atomic Energy Commission, predecessor to the U.S. DOE, established a production complex in the early 1950's for processing uranium and its compounds from natural uranium ore concentrates. This complex, then known as the FMPC and now as the FEMP, is located on 1050 acres in a rural area approximately 18 miles northwest of downtown Cincinnati, Ohio (Figure 1). The villages of Fernald, New Baltimore, Ross, Shandon, and New Haven are all located within a few miles of the plant.

The FEMP consists of a large central production area as well as other surrounding storage and buffer zones (Figure 2). The production area covers approximately 136 acres near the center of the FEMP. The Pilot Plant was completed in 1951 and was the first operational facility at the FMPC. The Pilot Plant was utilized to house many different processes including thorium metal production, uranium metal production, and uranium hexafluoride reduction. Following the completion of the Pilot Plant the metals fabrication plant, Plant 6, began operations in 1952. The metal production plant, Plant 5; the greensalt plant, Plant 4; the recovery plant, Plant 8; the sampling plant, Plant 1; and the refinery (Plant 2/3) began operation in 1953. A

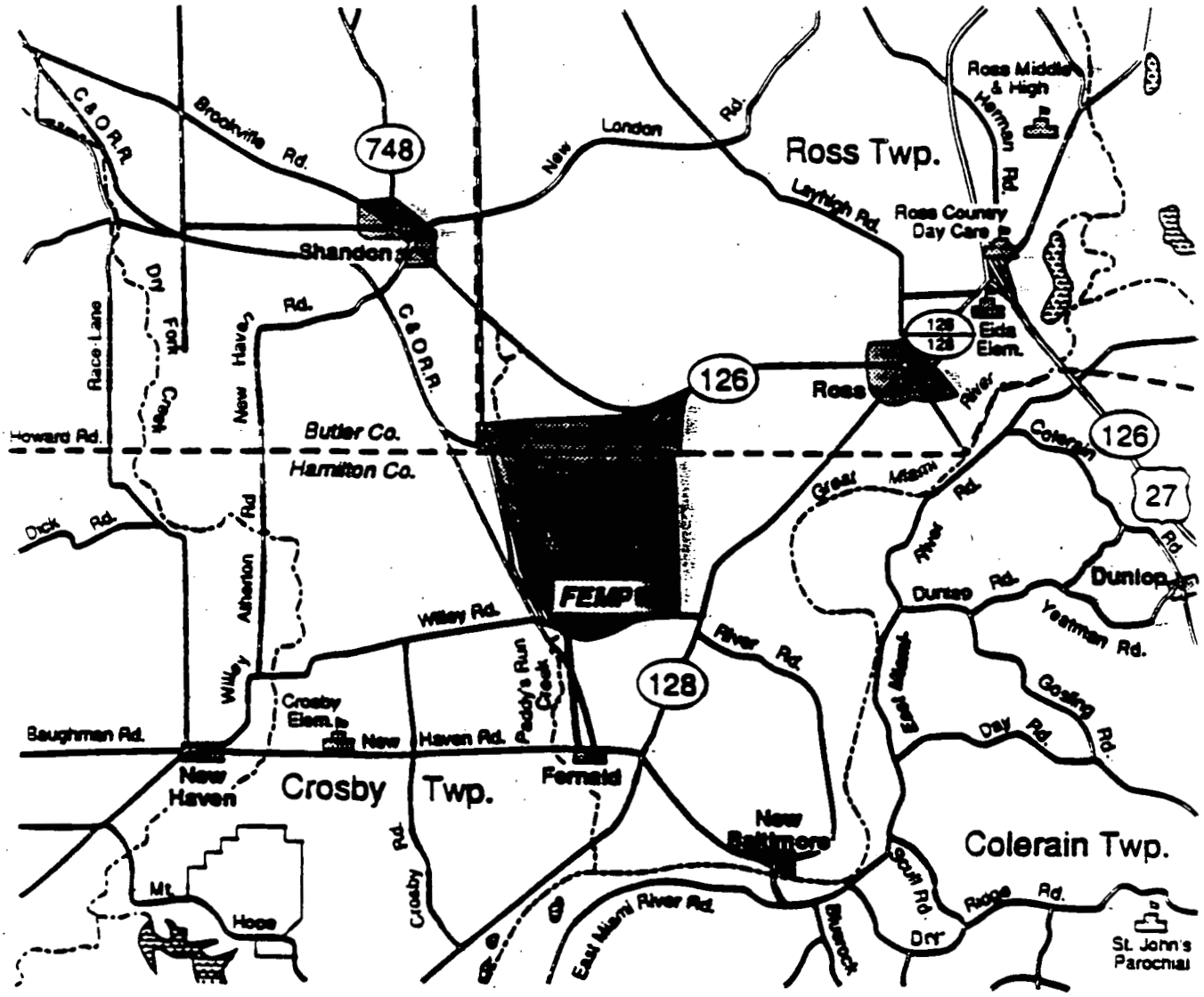
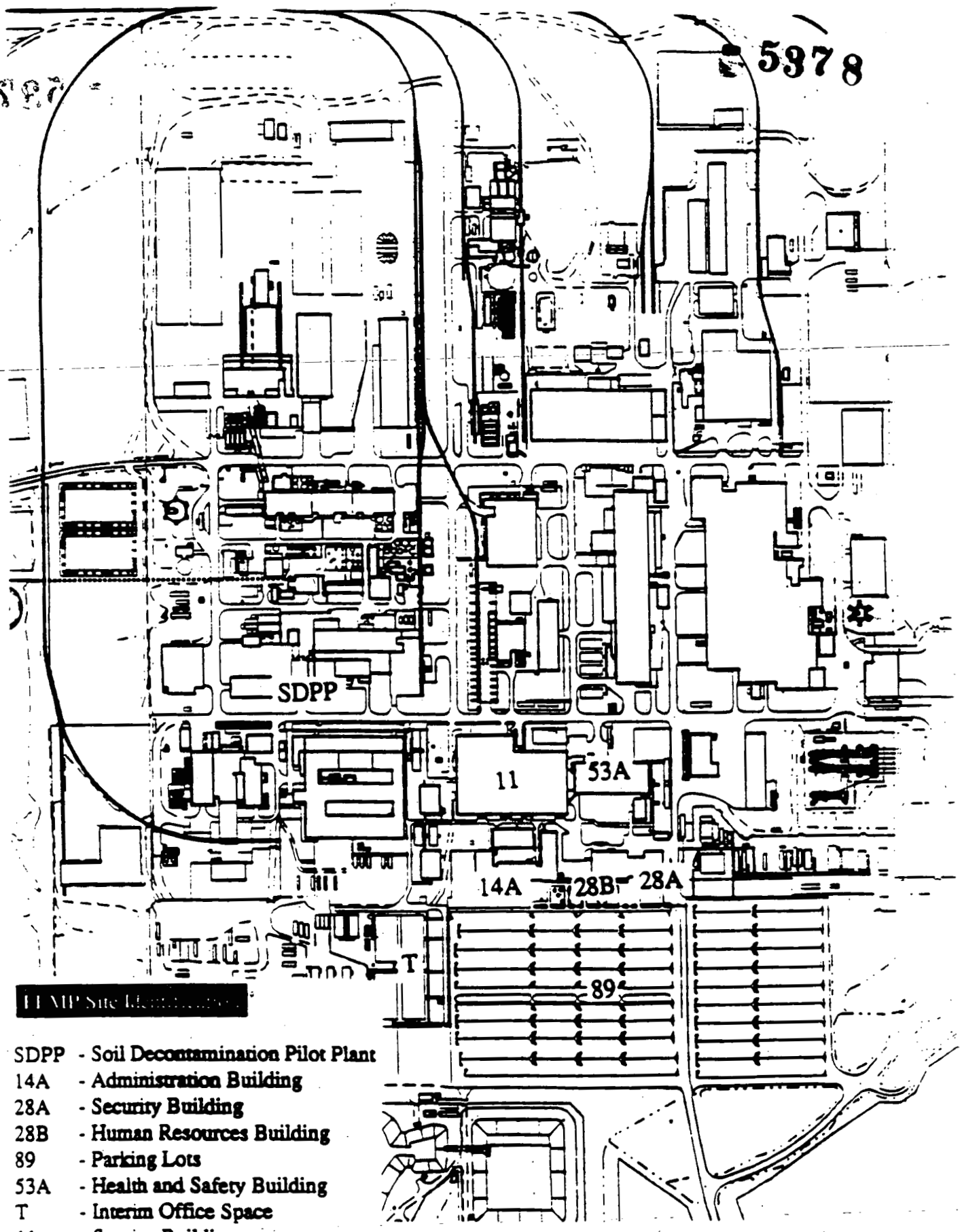


Fig. 1 FEMP site location



FEMP Site Identification

SDPP - Soil Decontamination Pilot Plant
14A - Administration Building
28A - Security Building
28B - Human Resources Building
89 - Parking Lots
53A - Health and Safety Building
T - Interim Office Space
11 - Service Building

Fig. 2 FEMP site plan 0 14

uranium hexafluoride reduction plant, Plant 7, and the special products plant, Plant 9, were operational in 1954.

Production peaked in 1960 at approximately 10,000 metric tons of uranium (mtu) per year. A product decline began in 1964, to a low in 1975 of about 1230 mtu. During the 1970's, consideration was given to closing the FEMP and capital improvements and staffing were minimized. In 1981, the FEMP began to accommodate increased production requirements. Production levels significantly increased and there was a rapid staff buildup for several years. Implementation of a major facilities restoration program followed. Production ceased in the summer of 1989 to focus plant resources on the environmental restoration program.

On July 18, 1986, a Federal Facilities Compliance Agreement (FFCA) pertaining to environmental impacts associated with operation of the FEMP was jointly signed by DOE and the USEPA. The FFCA was entered into pursuant to Executive Order 12088 (43 CFR 47707) to ensure compliance with existing environmental statutes and implementing regulations such as the Clean Air Act (CAA); RCRA; and CERCLA. In particular, the FFCA was intended to ensure that environmental impacts associated with past and present activities at the FEMP are thoroughly and adequately investigated so that appropriate remedial response actions can be formulated, assessed, and implemented. The 1986 FFCA was amended by a Consent Agreement under section 120 and 106(a) of CERCLA. The Consent Agreement became effective on June 29, 1990. In 1991, a renegotiation of the Consent Agreement was initiated to establish a revised schedule for cleanup of the FEMP. This amended Consent Agreement was signed in September, and became effective on December 19, 1991.

In response to the FFCA, a RI/FS is in progress pursuant to CERCLA, as amended by the Superfund Amendments and Reauthorization Act (SARA). The technical strategy adopted by the CERCLA program divides the site into 5 distinct CRUs. The components of the CRUs are:

CRU1 - Waste Pits 1-6, Clearwell, and Burn Pit
CRU2 - Other Waste Units (fly ash pile and Solid waste landfill)
CRU3 - Production Area
CRU4 - Silos 1, 2, 3, and 4
CRU5 - Environmental Media

Soils in the region of the FEMP were formed from source materials that were deposited by the action of Wisconsin and Illinoisan glaciers. These materials consist mainly of glacial till but include sand, gravel, glacial lake clays, and silty clays.

Uranium, present primarily in the hexavalent form, is the principle contaminant

of concern at the FEMP. The acceptable concentration level for uranium in soils has not been established. Consequently, soil decontamination technologies will be evaluated with respect to a level of 52 ppm, the initial technology screening level adopted by the Uranium Soils ID. Using the data generated in this demonstration, the technologies may be evaluated with respect to other levels, as well as compared against other. The majority of soils containing uranium exceeding 52 ppm are located in the top 0.45 m of surficial material.[5]

Uranium has deposited over the years on the soil from various sources. The stacks in the production area, dust blown from the disposal pits in the Waste Storage Area, and the incinerator in the sewage plant were sources of airborne uranium. Also, soil contamination has resulted from leaks and spills during processing activities in the production areas and from the spreading of contamination by vehicles.

1.4 Regulatory Requirements

The FEMP has been classified as a National Priorities List Superfund Site and is governed by the following regulatory agreements: 1986 FFCA, Consent Agreement under CERCLA Section 120 and 106(a), and Consent Decree with the State of Ohio to ensure compliance with regulations such as CERCLA, RCRA, and CAA. It is imperative that environmental regulation/DOE Orders are thoroughly reviewed to ensure that the demonstration operates within all applicable regulations. Compliance with environmental issues and DOE Orders at the FEMP and all subsequent technology user sites, has the potential to impact remediation cost and schedules. Complete evaluation of the demonstration requires total understanding of regulatory/DOE requirements and their effects on cost.

All field activities will meet the conditions of the agreements and adhere to all FEMP environmental policies. The Integrated Demonstration Coordinator (IDC) is responsible for assuring environmental compliance in accordance with Section 9 of the ID Management Plan (DOE 1991). Regulations 29 CFR 1910 and 29 CFR 1926, as well as other DOE standards/codes, shall be complied with during construction, installation, and operation. Full compliance with 29 CFR 1910 (OSHA) shall be maintained for all operational modes, including "testing" phases.

1.5 Demonstration Schedule and Duration

The on-site Phase II demonstration, to be conducted at the FEMP, is tentatively scheduled to begin in November, 1993. Table 1 shows the duration of significant events to be conducted as part of the field demonstration.

Table 1. Phase II Field Demonstration Schedule

Leaching test	Duration (weeks)
Carbonate	10
Citric acid	2
Citrate-bicarbonate-dithionite	2
Sulfuric acid	2

1.6 Overview of Demonstration Plan

This demonstration plan delineates the project objective and outlines the overall strategy for meeting this objective. The plan then describes in detail the experimental equipment to be used, the soils to be tested, the experimental test plan, the data to be collected, and the results to be determined from analysis of the data. Finally the demonstration plan addresses quality assurance and health and safety issues as they apply to the FEMP.

2. PROJECT OBJECTIVE AND STRATEGY

2.1 Uranium Removal from Soils by Chemical Leaching

Soil washing using only water is applicable in the remediation of soils containing highly water soluble constituents or soils containing very low concentrations of silt and/or clay particles (usually < 15% smaller than 50 μm). Such soil washing systems characteristically are not effective in the removal of contaminants from heavy textured soils [those soils containing high concentrations of clays (< 2μm) and silts (50 μm to 2 μm)] which often contain an appreciable proportion of the contaminant within these fractions. This is generally the case for the Fernald uranium-contaminated soils. For example, initial soil characterization studies of samples collected at Fernald[5] indicated that the particle size distribution in three of the six samples contained > 66% clay and silt-size fractions and these fractions contained approximately 50% of the uranium. Most importantly, all of the size fractions contained uranium concentrations > 52 mg/kg , the proposed screening level for applicable cleanup technologies. In the other three samples, the most highly contaminated fraction was the sand fraction (2000 μm to 53 μm particle size range) indicating that a simple physical separation processes would not be an effective cleanup technology. Thus, any successful soil washing approach will likely depend on a chemical leaching process.

The USEPA SITE program[6] recognizes soil washing as a combination of physical and chemical treatments performed on soil in an aqueous solution. The program now includes soil washing processes using leaching media ranging from alkaline mixtures of ionic and non-ionic surfactants and bioremediation agents (which act as biosurfactants[7]) to acid leachings for the removal of heavy metals from contaminated soils and solids[8].

Uranium is characteristically leached from uranium ores using acid and carbonate based leaching agents. Compared with acid leaching systems, however, carbonate leaching has been used less often due primarily to slower kinetics associated with the alkaline route. Use of carbonate became attractive when the uranium grade was high or in cases where the carbonate or lime content of the ore was high. The alkaline leaching also produced a clean separation of uranium from its ores without solubilizing other metals, as many metals are not soluble in such solutions. This advantage is also realized when leaching soils which may contain hazardous metals.

In acid leaching of uranium ores, sulfuric acid has often been used as a leaching agent. Because of strong acid's destructive nature on layer silicates, acid leaching of this type may not be appropriate for leaching uranium from the Fernald soils; however, acid leaching may be useful for removal of uranium from Y-12 soils as the final product is another waste form.

Leaching of ores containing uranium in the hexavalent state with sulfuric acid forms stable highly soluble complexes of uranyl sulfates. Leaching ores containing uranium in the tetravalent state is more difficult, and oxidizing agents (such as sodium perchlorate, pyrolusite, etc.) are often added to convert the uranium to the hexavalent state. Carbonate-bicarbonate solutions may be more applicable for leaching uranium from Fernald soils because these solutions do not destroy aluminosilicate clay minerals to the extent as do strong acid leaching agents. The leaching efficiency is based on the formation of sodium or ammonium uranyl tricarbonates (highly stable water-soluble complexes). Ammonium carbonate leachings generally remove less silica than sodium carbonate leachings and thus may be preferred for leaching of uranium from the Fernald soils. Oxidants, such as potassium permanganate, and catalysts, such as ferrocyanide or copper salts, are often used to increase the carbonate-bicarbonate leaching efficiency of uranium contained in primary minerals in the tetravalent form. Uranium dioxide, uraninite, is not soluble in dilute sulfuric acid; however, it is readily soluble in alkaline solutions of hydrogen peroxide. Also, tetravalent uranium is oxidized to the hexavalent form in carbonate solutions considerably faster than in sulfate solutions, again making carbonate the preferred leaching agent. Work performed at Los Alamos[9] has shown that most of the uranium in the Fernald soils is present as hexavalent uranium, meaning that once dissolution from the solid phase occurs, it is readily complexed by the carbonate or sulfate anion.

When leaching uranium from its numerous mineral forms, a few prerequisites are important to achieve adequate leaching of uranium from either naturally occurring minerals or from contaminated soils. The various prerequisites can be summarized briefly as follows in the necessary order:

(1) The uranium must be exposed to the solution in order to be solubilized. The use of chemical chelating agents such as citrates can be used to remove oxide coatings, or, simply, physical attrition may be adequate to expose uranium by removing weathering products on soil surfaces.

(2) The uranium must be oxidized to the hexavalent state, if not already oxidized, to be effectively removed by either the carbonate or the sulfuric acid leaching process.

(3) The solution chemistry must be controlled to ensure that an adequate supply of complexing anions such as carbonate or sulfate are available to complex and stabilize uranium in solution. Additionally, chemistries which promote premature, unwanted precipitation of uranium must be avoided.

The general approach in the Phase II pilot-scale demonstration will be to emphasize the leaching of uranium from Fernald soils by carbonate-based leachants

(because of their less destructive characteristics on the layer silicates of soils than acid leachants). Also, based on ORNL bench-scale studies that showed high rates of uranium removal from some of the soils, leaching with sodium citrate-sodium bicarbonate-sodium dithionite* (CBD) and with citric acid will be investigated. Finally, if warranted by the Phase I results, additional tests will be made with sulfuric acid.

2.2 Advantages and Limitations of Soil Leaching

Advantages of soil leaching for uranium removal may be listed as follows:

- A significant reduction in waste volume is achieved by transferring the uranium from the soil to the leaching solution, and then recovering the uranium as a concentrated waste stream for subsequent disposal.
- A useful soil is produced with a reduced uranium concentration that is below risk-based contamination levels when uranium is the contaminant of concern.

Limitations of the soil leaching process include:

- Leaching the large volumes of contaminated soil on site will produce large volumes of process water that will require treatment before recycle into the soil leaching process stream.
- If other contaminants are present, soil leaching may not adequately reduce risk when based on uranium decontamination alone. This concern will be addressed for the Fernald site in cooperation with the characterization team.
- Leaching may not reduce levels of uranium in soil to acceptable risk-based levels without producing large volumes of waste.

2.3 Results of Bench-Scale Testing and Rationale for Selection of Pilot-Scale Leaching Media

2.3.1 Overview of Bench-Scale Testing

Bench-scale testing at ORNL has centered around the ability to remove uranium from two soils at the Fernald facility. One soil was sampled adjacent to the waste incinerator and the other soil was sampled from near the No. 1 storage pad (a

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* Sodium dithionite - $Na_2S_2O_4$.

description of these soils and uranium concentrations has been reported[5]). Uranium concentrations in the Fernald soils used in bench-scale testing were ~550 mg U/kg for the incinerator site soil and ~450 mg U/kg for the storage pad soil. Bench-scale testing by the ORNL group consisted of testing several leaching media (Na_2CO_3, citric acid, a combination of citric acid and bicarbonate under aerobic and anaerobic conditions, and two mineral acids, i.e., sulfuric and nitric acid) under different concentrations and pH ranges using several mixing processes (paddle stirrers, rotary extractors, and attrition scrubbers at various liquid-to-solid ratios) over a range of temperature and time. Results from the initial phase of these experiments have been reported.[3].

Later results from bench-scale testing of uranium removal are being reported in the Uranium Soils Decon Task Group's report.[4] This is a report that contains summary data from four projects funded by the ID in FY1992. Four DOE facilities, Argonne National Laboratory (ANL), Los Alamos National Laboratory (LANL), the Savannah River Plant (SRP) and ORNL, directed the projects. The ORNL work was centered around the use of carbonate and citrate to remove uranium from soils. LANL evaluated the potential of using complex organic chelation agents for uranium leaching. ANL developed aqueous biphasic extraction processes to partition uranium particulate into different phases, and SRP (via a subcontract to Westinghouse Science and Technology Center in Pittsburgh, PA) tested the effectiveness of uranium leaching using ammonium carbonate and hypochlorite in combination with a bench-scale mineral jig. Other bench-scale testing included that conducted by the IT Corporation under the RI/FS contract with FEMP. The IT Corporation screened a large number of leaching media (ranging from mild dispersants to strong mineral acids such as sulfuric acid) for their effectiveness to remove uranium from the two Fernald soils. These data will be reported in their upcoming report. Baseline results from these studies have been presented at informal briefings between IT, ORNL, and FERMCO personnel. Their best leaching results were obtained with sulfuric acid (concentrations ranging from 1 to 2 N at liquid-to-solid ratios ranging from 4-to-1 to 7-to-1) and sodium carbonate leachings. Thus, Phase I pilot-scale studies by CRU5 centered around the use of these two media.

A proposed screening level for an applicable cleanup technology was established at 52 mg U/kg (equivalent to 35 pCi/g, assuming the distribution of uranium isotopes in naturally occurring uranium). However, this is not an established cleanup level. Studies are being conducted, both within the structure of the ID and at the Fernald site by FERMCO personnel and contractors, to determine a uranium cleanup level based on a risk analysis protocol. The protocol would utilize such factors as total uranium concentrations in the soil, concentrations of uranium in the respirable fractions of soil, and leachability of uranium from soils under ambient environmental conditions. Studies are also planned to use this same protocol to evaluate the fate of residual uranium in the soils from the various cleanup

methodologies. Data from these studies will be used to compare differences in overall operational costs (costs that also include waste management of secondary waste streams and disposal of the final uranium waste form). The intent is to develop detailed flow sheets for each of the processes selected so that environmental and health risks associated with residual uranium in soil can be related to the costs of each leaching methodology.

2.3.2 ORNL Bench-Scale Tests

In the ORNL bench-scale studies, carbonate leachings generally removed 70 to 90% of the uranium from the storage pad soil, but only 40 to 60% from the incinerator site soil. Increasing leaching temperature from 22 to 40°C increased the fraction of uranium leached from the incinerator site soil to ~80%. The ORNL group concluded that the Fernald soils containing >450 mg U/kg could probably not be lowered to uranium concentrations <52 mg U/kg using carbonate based leaching media in an acceptable engineering design (a design that has a mean residence time on the order of an hour). For example, only after 6 and 23 h leaching at 40 or 60°C was the concentration of uranium in the Fernald storage pad soil taken to values <52 mg/kg. The same leaching conditions with the Fernald incinerator soil resulted in residuals containing 60 to 70 mg/kg. Attrition scrubbing (at room temperature) with sodium carbonate yielded leaching efficiencies of approx. 60 and 80% for the incinerator and storage pad soil, respectively (calculated residuals concentrations of ~210 and ~90 mg/kg, respectively). Attrition scrubbing with ammonium carbonate yielded approximately 20% lower leaching efficiencies than with sodium carbonate. However, one should not automatically rule out the use of carbonate based leaching media to decontaminate the Fernald soils, because of all the leaching media tested, sodium and ammonium carbonate based leachings were the most selective for uranium and generated waste streams with lower concentrations of secondary soil constituents, i.e., iron, aluminum, calcium, and silica, than other leaching agents, namely citric and sulfuric acids. Ammonium carbonate, though not as effective in the leaching of uranium from the two Fernald soils as sodium carbonate, is potentially a credible choice of a leaching medium in that both the ammonium and carbonate can likely be recycled using steam distillation processes, while at the same time leaving a residual containing high concentrations of uranium for disposal. Utilization of carbonate based leaching media may also be merited in a systems engineering approach where particle size segregation techniques are used to fractionate between soil separates containing acceptable and unacceptable levels of uranium. Soil separates containing unacceptable levels of uranium could be disposed of directly (depending on quantity of materials and disposal options) or subjected to a more aggressive leaching procedure. Thus, based on these factors, carbonate-based leachings are strong candidates for testing as leaching media in the pilot-scale demonstrations.

The most effective leaching procedure (in terms of quantities of uranium leached from soils) used in the bench-scale tests was the CBD procedure. This is a reductive dissolution procedure developed to remove iron and aluminum sesquioxides from the surfaces of layer silicates.[4] Using this procedure, Fernald soils containing from 450 to 550 mg U/kg could be lowered to concentrations <52 mg U/kg. Questions remain as to optimum reaction time, quantity of reagents (primarily citrate and dithionite), and engineering design. These are being investigated. Limited data have been compiled, but it appears that considerably less waste will be generated using CBD than sulfuric acid. For example, CBD does bring about some dissolution of calcite; however, not nearly as much as sulfuric acid, since the reaction pH for CBD is 7.3 compared to <2 for sulfuric acid. Thus, from its aggressiveness viewpoint, the CBD procedure is a strong candidate for testing in the pilot-scale demonstration.

Bench-scale studies by IT Corporation found sulfuric acid to be a preferred leaching media for lowering uranium concentrations in the two Fernald soils to <52 mg U/kg. ORNL studies also observed that sulfuric acid could be used to lower uranium concentrations to <52 mg U/kg if the pH of the acid and soil suspension was maintained at values <2. Citric acid could also be used to treat the Fernald storage pad soils to levels <52 mg/kg; however, it appears that long reaction times in batch-stirred reactors may be necessary. Preliminary data using citric acid in combination with attrition scrubbing indicated levels <52 mg/kg with the storage pad soil may be possible. The leaching data using citric acid on the incinerator site soil indicated only 60 to 70% removal rates producing levels in the residual considerably >52 mg/kg. The major problems in the utilization of citric and sulfuric acid are the quantities of acid necessary to treat the soil and the resulting characteristics of secondary waste stream due to the dissolution of the large quantities of calcite and dolomite carbonate minerals. In the laboratory, the addition of either of the acids resulted in considerable foaming, making it difficult to contain the suspension in laboratory-scale reaction vessels. In the case of the Fernald storage pad soil, ~55 gal of concentrated sulfuric acid per ton of soil would be required to bring the pH to <2. Both of these leachants will also remove significant quantities of iron and aluminum from the soil layer silicates. Leaching with citric acid has some advantages over leaching with sulfuric acid; (1) because citric acid is not a highly dissociated acid (pK_1 of 3.1) the pH of the suspension will not go <2 when the quantity of acid necessary to dissolve the carbonate and produce a suspension with a final pH of 2 is added in a single unit (an equivalent addition of sulfuric acid will result in initial pH values significantly <2 and likely in significant dissolution of layer silicates and generation of unnecessary quantities of aluminum, iron, and silica) and (2) citric acid readily biodegrades to carbon dioxide and water in the environment. FERMCO and IT plan to use sulfuric acid in their Phase I pilot-scale studies. Because of time limitations on the Phase I RI/FS work, ORNL and the ID team have agreed to complete some limited tests with sulfuric acid to fulfil any potential data gaps.

In the ORNL bench-scale studies using an attrition scrubber, the entire soil was subjected to scrubbing. In typical applications of attrition scrubbing, the coarse fraction of the soil would be scrubbed. However, lab preparation of sufficient quantities of a coarse soil fraction for bench-scale studies was impractical. Such studies will be done, though, using a coarse soil fraction that will be produced on the SDPP. In the bench-scale tests, at reaction times of up to 15 minutes, higher uranium leaching efficiencies were obtained when leaching and scrubbing were carried out simultaneously than when leaching was performed without scrubbing. Also, the uranium removal efficiencies under the scrubbing/leaching conditions were comparable to those observed in leaching tests without scrubbing at reaction times of several hours. In recent tests, the highest uranium removal efficiencies were obtained in a two-step procedure involving scrubbing of a water/soil slurry at a 0.8/1 weight ratio for 15 minutes, followed by addition of a leaching agent and water to give a 2/1 water-to-soil weight ratio and leaching for 15 minutes. Further studies of the effect of attrition scrubbing are being carried out.

2.3.3 Phase II Pilot-Scale Tests

In Phase II, the effect of attrition scrubbing on uranium leaching will be demonstrated. Both the entire soil and a coarse soil fraction will be treated in the scrubber. Typically, attrition scrubbers are used to process relatively coarse materials, such as sand, in slurries at high solids loadings. Scrubbing, polishing, and disintegration of such material result from grain-to-grain attrition. In leaching of uranium from soil, attrition scrubbing is believed to remove coatings of amorphous iron and aluminum sesquioxides from uranium particulates, exposing the uranium to the leaching agent and thereby enhancing the reaction rate. The presence of finer particles is thought to be detrimental to these actions. While Fernald soils are made up of significant fractions of fine material such as silts and clays, attrition scrubbing of the entire soil may actually be beneficial. By the shearing action of the scrubber, silt and clay aggregates may be dispersed more easily into discrete particles. This would result in an increase in surface area, which produces an increase in the rate of leaching of the uranium associated with these fine soil fractions. Studies of the effect of attrition scrubbing on the leachability of different soil fractions will help to optimize the soil decontamination process.

Based on the ORNL bench-scale results, testing of leaching media in the pilot-scale test in Phase II will take the following priorities. Sodium carbonate leachings will be tested first. The citric acid leaching procedure will then be tested, followed by the CBD tests. Tests with sulfuric acid may or may not be conducted, depending on results obtained in the Phase I sulfuric acid tests. Sodium carbonate and sulfuric acid represent the least and most aggressive media, respectively, that were tested for uranium removal in bench-scale studies.

2.4 Soil Leaching Goals

The purpose of the tests on the SDPP is to assess the performance of selected soil leaching technologies for removal of uranium using CRU5 soils. The ultimate goal of this project is to develop data on the efficiency and operability of the soil decontamination process that can be used for the design of a full-scale system. For this goal to be accomplished, there are several specific project objectives to be satisfied.

- The soil leaching process should produce a soil that has an uranium activity level equal to or less than an acceptable contamination level. Since an acceptable concentration level for the cleanup of uranium in soils has not been established in accordance with the regulatory agencies of USEPA and the state of Ohio, soil decontamination will be evaluated with respect to (1) a preliminary technology screening level of 52 mg U/kg of soil or (2) revised target levels based on risk assessment data.

- The soil leaching process will also represent an "earth friendly approach" to the treatment of soil. That is, the process should decontaminate the soil without seriously degrading the soil's physical/chemical characteristics or generating secondary uranium waste forms that are difficult to manage and/or dispose. The major concern is not to generate a decontaminated soil whose waste form characteristics would require its management or disposal as a waste. Preferably, because of the large volume of soil to be treated, decontaminated soil should not be toxic to plants. The effect on the toxicity characteristics of the soil to grow plants will be determined using short-term root elongation tests and greenhouse trials.

- The results of bench-scale soil leaching tests should be further demonstrated at the pilot scale. These results will be useful for full-scale application of the technology. This objective will be met by operation of the SDPP, first in a batch mode (in Phase II of the demonstration) and then, after modifications, in a continuous mode (in Phase III of the demonstration). The ability to run the SDPP in the continuous mode for an extended period while maintaining leaching efficiency within control limits will be a measure of the operability of the process.

- The results of the CRU5/ID Phases II and III will be shared with CRU5 and may be incorporated in the CRU5 RI/FS as time permits.

2.5 Demonstration Test and Evaluation Approach

As discussed in Section 1.2, CRU/ID Phase II soil leaching studies will be conducted at Fernald on the SDPP. The SDPP operations will be performed by FERMCO personnel according to the experimental plan provided by ORNL. The plan was based on the results of soil characterization studies[4,5] and experimental leaching studies.[3,4] In the latter, leaching agents such as sodium carbonate and citric acid were used to leach uranium from various soil and sediment samples. The objective of these leaching studies was to determine the effectiveness of these treatments in reducing uranium concentrations to acceptable regulatory levels without seriously degrading the physical/chemical characteristics of the soil. As discussed previously, process modifications, which involve routing and the sequence of use of the equipment, will be made to the SDPP for the Phase II batch leaching tests. The process configuration to be used for the Phase II tests is based on the following considerations:

- Uranium is distributed throughout the various particle size fractions of the Fernald soils at concentrations above the regulatory limit.

- There is no experimental evidence that physical treatment alone, such as by treatment with an attrition scrubber and fractionation by particle size, will remove uranium from a particular fraction of Fernald soils (e.g., uranium concentrations in sand, silt, and clay fractions of the storage pad soil and the incinerator site soil at Fernald were all >52 mg/kg, the proposed screening levels for applicable decontamination technologies[3]) Consequently, any successful soil decontamination process will likely depend on a chemical leaching process.

- By using an attrition scrubber to treat the entire soil, high leaching efficiencies were obtained at relatively short residence times in bench-scale studies. These studies involved (1) a one-step test in which attritioning and leaching were done simultaneously and (2) a two-step test in which attritioning and leaching were done in sequence.

- The highest uranium removal efficiencies were obtained in a two-step procedure involving scrubbing of a water/soil slurry at a 0.8/1 weight ratio for 15 minutes, followed by addition of a leaching agent and water to give a 2/1 water-to-soil weight ratio and leaching for 15 minutes. These results indicate that attrition scrubbing is more efficient at high solids loadings and that leaching is more efficient at lower solids loadings.

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- Multiple rinsings of the leached soil are needed to minimize the amount of spent (i.e., containing dissolved uranium) leaching solution retained by the soil (this procedure is common practice in the uranium milling industry[10]). Replacement of sodium from sodium carbonate leaching will likely have to be conducted using calcium-based solutions to avoid sodium toxicity to plants and a dispersed soil system.

In the process configuration developed for Phase II operation, tests will be run in which either the entire soil (actually the portion <4.75mm, as described later) or a coarse soil fraction will be pretreated in an attrition scrubber and then subjected to leaching in a stirred tank. This will be followed by liquid/solid separation, either by filtering or centrifuging, and then rinsing to remove the spent leaching solution from the treated soil.

As discussed previously, the SDPP will be operated in a batch mode in Phase II and then in a continuous mode in Phase III. Batch mode operation requires minor piping modifications to the SDPP Phase I configuration. For continuous mode operation, which will be carried out to simulate a full-scale system, additional equipment will be installed and piping modifications will again be made.

3.0 DESCRIPTION OF SOIL DECONTAMINATION PILOT PLANT

3.1 Batch Operation

In the batch operation of the SDPP, one drum of feed soil will be processed in each experimental run. As shown in Figure 3, the first step of an experimental run is to treat the feed soil in the trommel screen to remove the >4.75mm material. The slurry from the trommel, which consists of <4.75mm material and the water that was injected at high pressure into the trommel, is pumped to an vibrating screen deck. The >50 mesh material will be removed in the screen deck and the slurry with the <50 mesh material will be collected in a mixing tank.

The slurry in the mixing tank will be processed through a continuous centrifuge that is installed on the SDPP. Before feeding the centrifuge, water will be added to the slurry, if necessary, to adjust the liquid/soil weight ratio to 4/1 (the lowest ratio recommended for operation of the centrifuge). Depending upon the test to be run, the centrifuge will be operated either to dewater and collect all the <50 mesh material as a wet cake, or to separate the <50 mesh material into a coarse fraction and a fine fraction. For dewatering, a polymer will be added to the feed slurry to flocculate the fines and maximize the amount of soil recovered in the wet cake. The centrifuge wet cake, which will consist of either the <50 mesh material or the coarse soil fraction, will be combined with the >50 mesh material from the screen deck and then treated in the attrition scrubber. Samples will be taken to determine the effect of attrition scrubbing on soil particle size distribution and uranium concentration as a function of soil particle size.

After attrition scrubbing, the centrifuge wet cake and the >50 mesh material will be combined with the centrate (the solids-depleted stream produced by the centrifuge) and transferred to the leaching reactor into which a leaching agent will be added. The leaching agent may be added directly in concentrated or pure form, or from a 100 gallon mixing tank where it has been diluted to the appropriate concentration. All feedstock chemicals will be transferred to the holding tanks from carboys or totes supplied by the chemical supplier. Feedstock chemicals will be stored in a diked area for spill containment.*

After addition of chemicals, the soil will be leached in the reactor for a specified time. During this leaching step, samples of the slurry will be taken periodically so that

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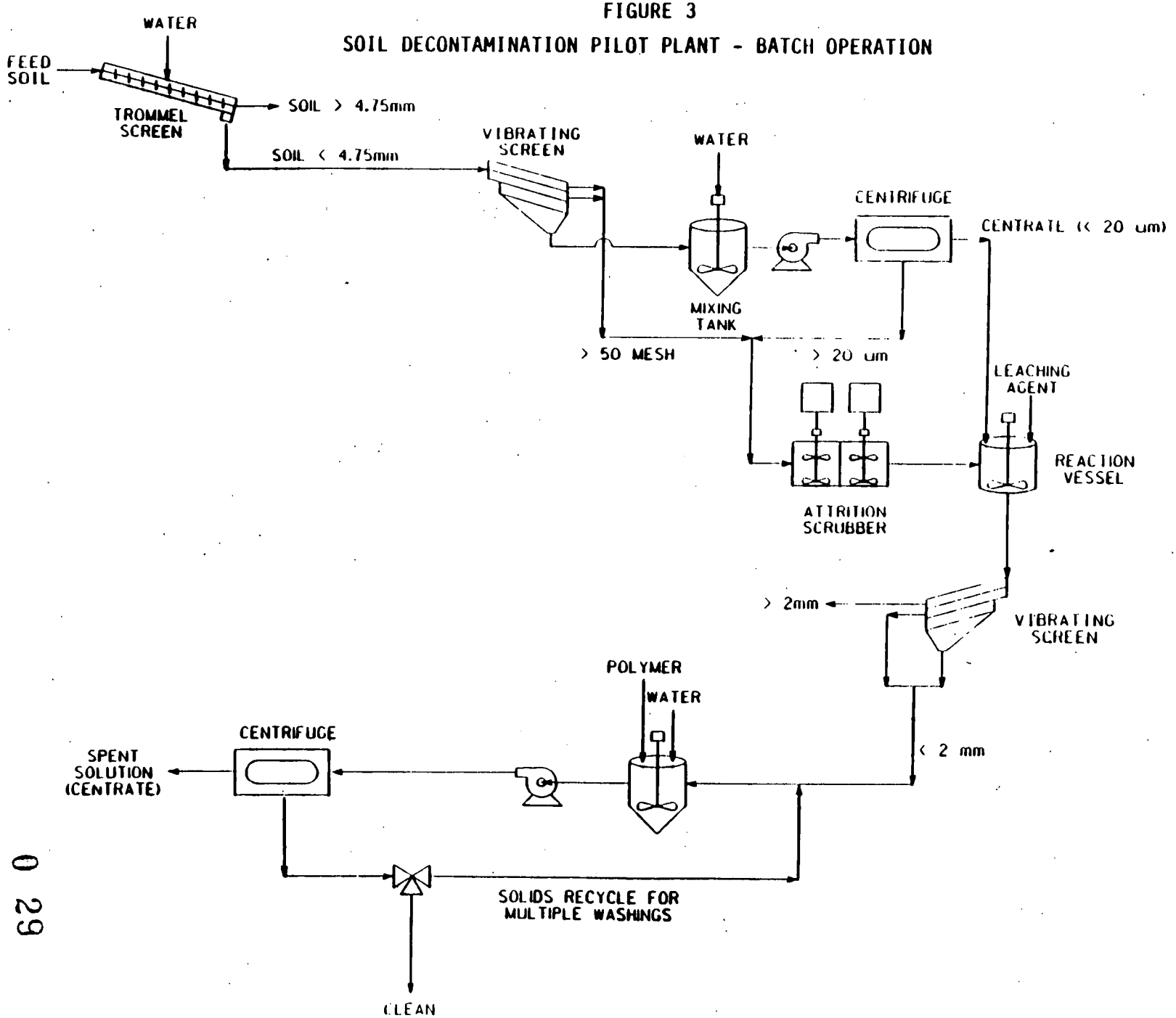
*Reference: Safety Assessment for the Operable Unit 5/Integrated Demonstration Soil Treatability Studies, January 1993, Rev. 1

FIGURE 3

SOIL DECONTAMINATION PILOT PLANT - BATCH OPERATION



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the uranium leaching efficiency can be determined as a function of time.

Upon completion of the leaching step, the soil will be rinsed. First, the soil slurry will be pumped from the reactor to the vibrating screen to remove the >10 mesh material. The slurry passing through the screen will be collected in a mixing tank. If necessary, water will be added to the soil slurry to lower the solids concentration to less than 20 weight per cent. The slurry will then be pumped from the mixing tank through the centrifuge to separate the solids from the leaching solution. The solids will then be recovered and returned to the mixing tank. Clean water will be added to the mixing tank, the slurry will be mixed, and the slurry will again be fed to the centrifuge for solids/liquid separation (SLS). If desired, this rinse cycle can be repeated by returning the solids to the mixing tank, mixing with clean water, and separating the solids in the centrifuge.

The procedure described above will be modified for some of the runs that will be made. These runs are described in Section 5.2.

3.2 Continuous Operation

The design for continuous operation of the SDPP will be based on the results of the bench-scale and the batch pilot-scale studies. The intent of the design will be to simulate a full-scale system. Thus, the SDPP will use equipment expected to be included in a full-scale design. Additional equipment that may be installed for continuous operation of the SDPP are a belt filter, a clarifier, and a spiral classifier.

4. TREATABILITY SOIL DESCRIPTION

Two soils were selected based on characterization for utilization in this demonstration. The soils were collected from the Sewage Treatment Plant/Waste Incinerator area and the Plant 1 Pad area.

4.1 Sewage Treatment Plant/Waste Incinerator Area

4.1.1 Background

The Sewage Treatment Plant Area is located on the eastern property line of the FEMP reservation. The Sewage Treatment Plant, associated facilities and the abandoned incinerator are contained within a six foot chain link fenced area on FEMP property where access is restricted by security officers. The Sewage Treatment Plant became operational in 1952 for the treatment of FEMP sanitary waste water. The system was later transitioned to receive both sanitary and process related waste waters. The practice of employing the Sewage Treatment Plant to treat process related waste water flows was discontinued recently with the installation and start-up of a biodenitrification effluent treatment system.

The solid waste incinerator is located in the northwest corner of the Sewage Treatment Area. This incinerator was operated from November 1954 through December 1979 at which time a new solid waste incinerator at Building 39 was put into service. The incinerator was used to burn contaminated and uncontaminated combustible trash during its period of operation.

4.1.2 Characterization

Surface radiological measurements and limited soil samples collected in the vicinity of these facilities indicate the presence of localized elevated concentrations of radionuclides.[5] As a result of the RI/FS sampling activities, the concentration of U-238 in surface soils was found to range from 1.8 to 25,670 pCi/g. In addition to surface soil samples, there was a limited number of core samples taken in this area as part of the RI/FS. The results indicated that uranium contamination is limited to the upper reaches of the soil column; in fact, only one sample exceeded 100 pCi/g, which was taken from a depth of 0.45-0.91 m. FEMP RCRA determination procedures have established that the USID sewage treatment plant soils are non-RCRA.

4.2 Plant 1 Pad

4.2.1 Background

During the production years at the FEMP, the Plant 1 Pad was used to temporarily store various residues from the production plants, raw ore, and material from other DOE sites. This material was then processed through Plant 2/3 and Plant 8 to recover the useable uranium present. After Plants 2/3 and 8 were shut down, the pad was used as a permanent storage area for the residues. The area from which the soils in question came was never used for production processes. However, the Plant 1 Pad did not have a containment barrier until the late 1980's. Numerous releases of material from deteriorated drums and accidental spills occurred throughout operational history of the pad. The lack of containment allowed released material to wash off the pad during rain storms and routine water-spraying of the pad, thereby contaminating the soil on the perimeter of the pad.

The Plant 1 Pad has been determined to be a Hazardous Waste Management Unit as a result of hazardous wastes having been stored there. Characterizations conducted under the purview of the Proposed Amended Consent Decree show that approximately 10% of the material which at some time was stored on the pad has been classified as hazardous waste under RCRA. Most of the identified RCRA wastes have been removed from the pad as a result of ongoing characterization efforts.

4.2.2 Characterization

The Plant 1 Pad soil has been determined to be a low level radioactive waste. FEMP RCRA determination procedures have established that the USID Plant 1 Pad soil is neither a RCRA listed waste nor a RCRA characteristic waste.

4.3 Soil Preparation

Soils have been removed from the Sewage Treatment Plant and the Plant 1 Pad areas and stored as containerized material. This material consists of unmodified soils and blended soils. The blended soils were sifted using a 3/4 inch screen to remove gravel fragments and unwanted debris. The sifted soils were then processed in a 5 cubic yard concrete mixer to obtain homogeneity. Containers include drums of blended soils and metal boxes of unmodified soils, blended soils, and residues from the blending process.

Soil for the batch operation portion of this demonstration will be taken from the unmodified soils in the metal boxes. Drums of each soil will be made up by screening the excavated soil from the metal boxes through a 3/4 inch screen. The screened soil will be processed through a ribbon blender to obtain homogeneity and then will be

drummed. This blended soil will be used in this demonstration for optimization of the process. When the optimum process parameters have been established, one drum of the original blended soil (excavated and drummed in 1991) will be processed through the SDPP as a demonstration of the best processing method developed in the batch testing. The original blended soil will be a reference soil that will be tested by all USID investigators of soil decontamination technologies. The soil to be used for the continuous operation of the SDPP is to be determined.

5. EXPERIMENTAL TEST PLAN

5.1 Overall Approach

The SDPP will be operated in a batch and then in a continuous mode to demonstrate the removal of uranium from two contaminated soils from the Fernald site. These soils were taken from near the Fernald waste incinerator and near the Fernald plant 1 storage pad.

In Phase II, as discussed in Section 2.3, leaching tests will be run with up to four different leaching agents on each of the two Fernald soils. These leachants, which were used in previous bench-scale studies, are sodium carbonate, CBD, citric acid, and sulfuric acid. For the CBD tests, modifications will be made to the SDPP to treat the gas produced by the reaction of sodium dithionite. It is anticipated that an absorption unit will be installed to remove the sulfur oxide gas that is generated. Testing with sulfuric acid will depend on the results obtained in Phase I pilot-scale testing. The leaching solution molarities to be used in the Phase II pilot- scale tests will be selected based on the ORNL bench-scale and the CRU5 Phase I pilot-scale leaching results. The carbonate runs will be made at a total carbonate concentration of 0.25M.

In Phase III, the SDPP will be run in the continuous mode, using the most effective leachant as determined from these batch tests, to demonstrate the operability of the soil leaching process and to obtain additional data on process efficiency.

The primary independent parameters in the pilot-scale tests are the residence times in the attrition scrubber and in the leaching reactor. In batch operation, residence time simply depends on the elapsed time for either the attrition scrubbing or the leaching operation. In continuous operation, residence time is determined by the ratio of total vessel volume to soil slurry feed rate.

5.2 Experimental Design

In Phase II, a total of eight different runs on the SDPP would be required to test all possible combinations of the four different leaching agents and the two Fernald soils. To determine if the data quality objective (DQO) developed for this demonstration (see Section 6.2) was satisfied, the variance of the results must be established. This will be accomplished by replication of samples and runs. Rather than replicating all runs, two of the eight runs will be done in triplicate. The results from these six runs will be used to calculate a pooled variance.[11] This pooled

variance will then be used for the comparison of the results from all the runs (i.e., the replicated and the non-replicated runs) with the technology screening level. The pooled variance will also be used to compare the effectiveness of the different leaching agents with one another. The total number of runs required by this experimental design is less than that for a design involving duplication of all runs.

The processing sequence and conditions for these twelve runs are based on the results of the bench-scale studies and a conceptual design for the full-scale soil leaching process. Specifically, the bench-scale studies showed attrition scrubbing of a slurry with a high solids content was beneficial. In a full-scale system, equipment to produce such a slurry could likely be a spiral classifier. In the operation of this classifier, fine material would be removed as a dilute slurry and the high solids content stream would contain the coarse material . The operating procedure for the twelve runs is summarized in the following.

1. Process the feed soil through the trommel and the vibrating screen and collect the slurry (containing the <50 mesh material) in the mixing tank.

2. Process the slurry through the centrifuge. Operate the centrifuge to obtain a size cut of approximately 20 μm. Collect the centrifuge wet cake (which will have a liquid/soil weight ratio of about 1/1) in a drum and the centrate in a mixing tank.

3. Turn off the centrifuge and collect the centrifuge heel in a drum. Add the centrifuge heel to the centrate in the mixing tank.

4. Combine the centrifuge wet cake with the oversize material (>50 mesh) from the vibrating screen. Feed this high solids content mixture through the attrition scrubber at a rate that produces a 15 minute residence time.

5. Combine the attrition scrubber product with the centrate (which contains the <20 μm material) and transfer the slurry to the leaching reactor.

6. Add leachant to the slurry and carry out the leaching reaction for 2 hours. Take samples at specified times in order to determine leaching efficiency as a function of time.

Before any of the runs in the experimental design are made, an initial run will be made to check out the system operation. This run will involve carbonate leaching of incinerator site soil and will follow the procedure outlined above, with the exception that the attrition scrubber residence time will be one hour. During the attrition scrubber operation, samples will be collected after each 15 minute period. These samples will be analyzed to determine the affect of attrition scrubbing on particle size

distribution. Also, the samples will be used in bench-scale studies to determine the effect of attrition scrubbing residence time on carbonate leaching efficiency.

Three other runs will be made for a total of sixteen runs in Phase II. The purpose of the runs is to determine the effect of the attrition scrubber on leaching efficiency. All three runs will be made with sodium carbonate. The conditions for these runs are described in the following.

Run 1. Attrition scrubbing of a coarse soil fraction in the presence of sodium carbonate: The procedure for this run is the same as that outlined above with the exception that sodium carbonate will be added to the attrition scrubber feed. This run will simulate a full-scale process in which the leaching solution would be recycled.

Run 2. Attrition scrubbing of the total soil in the presence of sodium carbonate: The procedure for this run is the same as that for Run 1, with the exception that the centrate will be reprocessed through the centrifuge with the addition of polymer to maximize the recovery of solids. The centrifuge wet cakes will then be combined with the vibrating screen oversize material to produce the attrition scrubber feed. Sodium carbonate will be added to this attrition scrubber feed as in Run 1.

Run 3. Leaching reactor only: The slurry from the trommel will be transferred directly to the leaching reactor. Leachant will be added and the reaction carried out for two hours with samples taken as specified in the outline above.

The conditions for all of the batch tests to be carried out in Phase II are summarized in Table 2. The notation in the table will be used to identify the conditions under which each run is made. For example, 05BNA2FW indicates test number 5 in the experimental design, which is the second replicate of a treatment of storage pad soil with sodium carbonate. Before leaching, the centrifuge separates the soil into a coarse and a fine fraction and the coarse fraction is attrition scrubbed without the addition of any chemicals.

Table 2. Summary of Conditions for Phase II Batch Tests

TEST	SOIL [a]	REAGENT [b]	REPLICATE	SIZE SEPARATION [c]	ATTRITION [d]
00	A	NA	1	F	W [e]
01	A	NA	1	F	W
02	A	NA	2	F	W
03	A	NA	3	F	W
04	B	NA	1	F	W
05	B	NA	2	F	W
06	B	NA	3	F	W
07	A	CI	1	F	W
08	B	CI	1	F	W
09	A	CB	1	F	W
10	B	CB	1	F	W
11	A	SU	1	F	W
12	B	SU	1	F	W
13	A	NA	1	F	C
14	A	NA	1	T	C
15	A	NA	1	T	N

a A - incinerator soil; B - storage pad soil.

b NA - sodium carbonate; CI - citric acid; CB - citrate/bicarbonate/dithionite; SU - sulfuric acid.

c F - soil separated into two size fractions with centrifuge; T - total soil is treated (no size separation)

d W - attrition with water only; C - attrition with chemicals added; N - no attrition.

e Collect samples after each 15 minute period of attrition scrubbing. Samples to be used in bench-scale studies.

In one of the runs with sodium carbonate treatment of the incinerator soil (Test

01, 02, or 03), the leaching reaction will be carried out for 24 hours. Samples will be taken during the first two hours according to the standard procedure and then midway and at the end of the 24 hour period. The results of this test will be an indication of the maximum leaching efficiency that can be expected with sodium carbonate.

The pooled variance determined as described above is a combination of the analytical variance, the sampling variance, and the run-to-run variance. The analytical variance will be determined by laboratory QA procedures. The sampling variance will be determined by taking replicate samples for analysis. The run-to-run variance may then be calculated by difference. If problems in meeting the DQO are encountered, examination of these variances may indicate where corrective actions may be taken.

As discussed previously, in each SDPP test with a particular soil and leaching agent combination, the primary independent parameters will be the residence times in the attrition scrubber and the leaching reactor. In the batch tests, the effect of residence time will be studied by taking slurry samples from the attrition scrubber and the leaching reactor at time intervals during each operation. Samples will also be taken of the feed soil, the decontaminated soil, and the spent leaching and rinse solutions. These samples will be analyzed for uranium content. The Sampling and Analysis Plan for the Phase II batch experiments is presented in Appendix A.

In addition to the sampling and analysis of various process streams, data acquisition will include the measurement of several process parameters. For batch operation, these parameters are (1) weights of feed and product streams such as feed soil, leaching chemicals, trommel oversize material, centrifuge wet cake, etc., (2) time duration of attrition and leaching operations, and (3) number of rinse cycles. Similar measurements will be made in the continuous mode operations.

The experimental design for the continuous operation tests in Phase III will be developed later.

5.3 Experimental Procedures and Details

Section 3.1 discussed the Phase II operation of the SDPP in the batch mode. Detailed procedures will be written for this batch operation and for the sampling activities planned for each batch test. The operating and sampling procedures developed by CRU5 for the Phase I tests will be adapted for the Phase II operation.

One addition to the sampling procedure will be the use of laboratory filters at the SDPP site to filter the slurry samples taken from the mixing tank and leaching reactor and to rinse the filter cake that is produced. The liquid and solid samples from this filtration/rinsing operation may then be submitted for analyses.

In the operating procedure for the SDPP, emphasis will be placed on the conditioning step used to clean the equipment between each run. The equipment will be flushed with water and samples taken of the effluent water either to confirm the absence of contamination or to obtain baseline contamination levels for the following run.

For operation in the batch mode, one drum of contaminated soil will be treated in each experimental run. For the experimental design described in Section 5.2, six drums of the storage pad soil and ten drums of the waste incinerator area soil will be required. Table 3 gives the quantities of soil and chemicals required for all sixteen runs. A total of ten runs will be made with sodium carbonate/sodium bicarbonate and two runs with each of the other three leachants. The material quantities in Table 3 were calculated based on a liquid/soil weight ratio of 4/1 (the lowest ratio recommended for feed to the centrifuge) and molarities used in the bench-scale studies.

Table 3. Materials Required for Batch Tests

Material	Weight (pounds)[a]
Storage Pad Soil	3600
Incinerator Area Soil	6000
$Na_2CO_3/NaHCO_3$[b]	204/344
Citric acid	230[c]
Sodium citrate/sodium dithionite/sodium carbonate[d]	370/45/120
Potassium permanganate	88
Sulfuric acid[e]	232

[a] Weight required for all batch tests with each material.
[b] For sodium carbonate tests.
[c] 0.25M.
[d] For CBD tests.
[e] Requirement for storage pad soil to maintain pH <2. Incinerator soil requirement, which will be less, will be determined by titration.

The amounts of materials needed for the SDPP tests in the continuous mode will be determined when the continuous system is designed.

Data sheets will be provided that list samples to be taken, analyses to be obtained, and process parameters to be measured. The information on the data sheets, along with the results of the analyses, will be used for data evaluation.

5.4 Data Evaluation

For each test, material balances will be made for the soil and uranium. This will include overall balances and balances around individual operations such as leaching and rinsing. Also, the uranium leaching efficiency will be calculated as a function of time. Statistical analyses will be performed to determine the variances of the calculated results.

The overall efficiency of the leaching process will be measured by comparing the uranium concentrations of the feed soil and the soil that is produced from the filtration/rinsing treatment. Thus, this measured efficiency will depend not only on the extent to which the leachant solubilizes uranium, but also on the extent to which the spent leaching solution that is retained by the soil after filtration is removed by rinsing.

Depending upon the feed rate for the system and the duration of each run, blending of the amount of material required for a test in the continuous mode operation of the SDPP in Phase III may be impractical. Thus, variation with time in the feed soil composition, and consequently in the product soil composition, may occur (which is to be expected in full-scale operation). If necessary, a statistical sampling plan for taking feed and product soil samples will be developed that will provide an acceptable measure of leaching efficiency.

The performance objective for leaching is to reduce the uranium concentration in Fernald soils to an acceptable level. As discussed previously, soil decontamination technologies will be evaluated with respect to a preliminary screening level of 52 ppm. The experimental uranium concentrations in the leached and rinsed soil will be used for comparison with the screening level. The leaching efficiencies of the different leaching agents will also be compared against one another.

6. QUALITY ASSURANCE PROJECT PLAN (QAPjP) FOR THE FERNALD INTEGRATED DEMONSTRATION

6.1 Purpose And Scope

It is essential to ensure that the data resulting from this demonstration are of known quality and that a sufficient number of critical measurements are taken. According to the description of ASLs specified in "Data Quality Objectives For Remedial Response Activities, Volume 1, Development Process" (USEPA 1987) this demonstration is categorized an ASL level II. This level is characterized by the use of portable analytical instruments which can be used on-site, or in mobile laboratories stationed near a site. Depending upon the types of contaminants, sample matrices, and personnel skills, qualitative and quantitative data can be obtained.

The FEMP Sitewide CERCLA Quality Assurance Project Plan (SCQ) will be referenced as a guideline during the implementation of this project. Applicable portions of the SCQ will be adopted to maintain comparability with other site activities.

6.2 Data Quality Objectives

Data quality objectives are qualitative and quantitative statements describing the level of uncertainty that project managers are willing to accept in making a decision using a decision rule. The acceptable level of uncertainty should be used as the basis for the design specifications for project data collection and data assessment. The QA component of this demonstration is designed to maximize the probability that the quality of the resulting data will be sufficient for the intended use.

Data generation efforts involve three parts: establishment of the DQOs and design of the project plan to meet the DQOs; implementation of the project plan; and assessment of the data to determine if the DQOs were met. Data quality indicators are used in the data assessment step. Data quality indicators are qualitative and quantitative parameters used to measure the attainment of the necessary quality for a particular environmental decision. Indicators of quality include precision, accuracy, representativeness, completeness, and comparability:[12]

1) Precision: the degree of mutual agreement among individual measurements. Provides an estimate of random error. Precision is determined by the measurement of replicates. Frequently expressed by standard deviation, relative standard deviation, variance, and confidence

interval.

2) Accuracy: refers to the difference between the population mean and the established concentration value for any analyte. Sampling accuracy can be assessed by evaluating the results of field blanks, while analytical accuracy can be assessed through the use of matrix spike and laboratory control samples. Net bias, the difference between a measured value and the true or expected value, provides an estimate of systematic error.

3) Representativeness: the degree to which the data accurately and precisely represent a characteristic of a parameter, variation of a property, a process characteristic, or an operation condition. Representativeness can be assessed by the use of collocated samples. Collocated samples are samples collected such that the samples are equally representative of a given point in space and time.

4) Completeness: measure of the amount of data collected from a measurement process compared to the amount expected to be obtained under the conditions of the measurement.

5) Comparability: a qualitative parameter expressing the confidence with which one data set can be compared to another. Comparability is assessed by determining whether the standard techniques stated in the work plan were used and that the analytical results were reported in the appropriate units. Data sets can only be compared with confidence when the precision and accuracy are known.

The final output from the DQO process is the development of a quantitative decision rule with uncertainty constraints. These uncertainty constraints are based on statistical sampling and analysis criteria. The DQO process is iterative. As more information is gathered, it may be appropriate to reevaluate the decision rule and uncertainty constraints limiting the decision.

As stated previously, the purpose of the tests on the SDPP is to assess the performance of selected soil leaching technologies for removal of uranium using CRU5 soils. One measure of performance of the leaching technologies is the uranium concentration that can be attained in the treated soil. The decision rule developed for this measure of performance, as described in the project DQO document,[13] may be stated as - if the uranium concentration in the treated soil is less than 80% of the technology screening level, then, at a confidence level of 95% or greater, treatment with the particular leaching agent is considered effective. That is, if 52 mg U/kg of soil is the screening level, then 41.6 mg U/kg of soil is the level that must be achieved for a treatment to be considered effective. Whereas 52 mg U/kg soil is the initial

technology screening level adopted by the Uranium Soils ID, the experimental results may be compared with any other screening level.

6.3 Performance And Systems Audits

Quality audits are considered an essential part of a QA program. Audits are of two types: systems audits are conducted to qualitatively evaluate the operational details of a QA program and performance audits are conducted to quantitatively evaluate the outputs of a measurement system. Both may be external independent evaluations or internal self evaluations, denoting whether the activity is conducted by the operating group or by someone not connected with it.

A performance audit (surveillance) will be conducted twice at a minimum during the demonstration. The audit will involve the development of a check list of critical operations extracted from this test plan and determining whether the operation was completed satisfactorily. The persons performing these performance audits are specified in QA Responsibilities section. The IDC will inform the FEMP QA organization of scheduled activities and make available valid copies of the test plan to be used in the field during project activities.

A system audit to verify that the instrument(s) are operating within expected parameters will be done as applicable by operational personnel. This information will be documented in log books.

6.4 Data Reporting And Verification

All raw data will be written into controlled log books or the raw data instrument print outs will be attached with appropriate notes into specified log books. All data entries into log books will include the following minimum information:

- Date of entry,
- Subject of entry,
- Signature of person entering data, and
- Signature of another individual who has cross checked the entries for accuracy.

The following information related to QC will be accumulate as part of the project's data files:

a. Identification of analytical methods used.
b. Chain of custody records, request for analysis forms, field sample variances, and sample location identifications.
c. A copy of this plan and the project DQO.

d. Initial and follow-on calibration information.
e. Identification of certified standards used by the lab and their expiration dates.
f. Background and blank information.
g. Identification of the counting/analysis system used to make analyses.
h. Statement of instrument detection limits and minimum detection activity.
i. Statement of percent yields for spikes, tracers, and/or carriers.
j. Run logs showing which samples and controls were run as a unit and their times of analysis.
k. Sample preparation logs which show aliquots taken and time they were done.
l. For isotopic results, the lab must supply relative error ratios.

Prior to submission, all data must be reviewed by a person (other than the analyst) who is experienced in all aspects of the technique being employed. Hard copy and electronic results as applicable must be accompanied by a case narrative which at a minimum includes the following:

- Any deviations from Sampling/Analysis Plan
- Any analytical problems encountered,
- Data interpretation difficulties,
- Any corrective actions taken as result of initial data taken.

A summary sheet which details the experimental conditions for the current test will be posted at the SDPP site. The operating status of the SDPP and the experimental data being obtained may be compared with the experimental conditions on the summary sheet for audit purposes.

6.5 Quality Assurance Reports To Management

QA reports provide management with the information to monitor data quality effectively. The only report which will be required for this project is the "Demonstration Evaluation Report". This report will be the responsibility of ORNL and ID personnel. This report will include all pertinent information/data and the conclusions to be reached about utilization of this technique for screening activities. The final report will be subject to both QA and technical review and will be revised and submitted for management approval. The QA review will be done by FEMP QA personnel, while the technical review will be done by the ID Decontamination Task Group and ORNL technical staff. The distribution of this report will be the responsibility of the IDC.

7. DATA MANAGEMENT

The elements of verifiability, traceability, and accessibility are the primary concerns which dictate that a data management process be implemented for any project which is concerned with QA.

Verifiability is the ability of the data user to determine that indeed the data being utilized were generated by the processes defined by this demonstration plan and other associated documents. This would include references to the project, dates, and analysts signatures.

Traceability is defined as the process of being able to identify the source of any piece of data generated from a project, why it was generated, and all those who had a part in the specific data generation. This is generally very important in regulatory analyses which will require chain-of-custody processes to track sampling/analysis activities.

Accessibility refers to the ability of the data user to easily retrieve data generated while at the same time assuring that the data will not receive irreparable damage by the data user.

7.1 Data Responsibilities

The project engineer is required to do the following with all data generated from this project:

- Maintain controlled log book with hard copies of all raw data entered or attach the raw data to pages of the log book with the subject for entry, the date, and signature of person making he log entry,

Note: Copies of all pages and data attached to the log book should be made for backup purposes in case original log book is lost or damaged.

- If applicable, maintain electronic media copy (e.g., flexible disk) of all raw data with a minimum of at least one back up stored in secured and fire proof area,
- Hard copies of all raw data, electronic media copy of all raw data if applicable, and a case narrative must be submitted to the ID personnel identified in the Data Review, Reporting, and Verification section of this document.
- Maintain index of all data generated from this project which

includes the following information about each entry:

1) accurate description of what datum involves (e.g., analyte, location, etc.),
2) date data taken,
3) storage location of original data, and
4) responsible party that generated data.

The IDC has the following responsibilities for management of all data submitted to them under the directions of this demonstration plan:

- Maintain a file of hard copies of all data submitted by ORNL.
- Submit electronic data media as applicable for entry into FEMP data base system under descriptive titles,
- Maintain index of all data received from ORNL with the following information for each entry:

  1) accurate description of what datum involves (e.g., ID Demo Plan, field screening data, technique employed, etc.),
  2) date the data was submitted,
  3) storage location of original data, and
  4) responsible party for generating the data (name, business, affiliation, phone number)

This system will adequately address the elements of verifiability, traceability, and accessibility when implemented.

8. HEALTH AND SAFETY PLAN

This demonstration program will be conducted in accordance with the provisions of the FERMCO CRU5 health and safety plan. Consistent with this program and 29 CFR 1910.120, a project specific health and safety plan will be prepared addressing the proposed work activities and will be included in Appendix B of this demonstration plan. A copy of the project specific Health and Safety Plan will be completed and approved by FEMP management prior to field mobilization. The project specific Health and Safety Plan identifies, evaluates, and controls all safety and health hazards associated with this demonstration program. In addition, it provides for emergency response for hazardous operations. Appendix C of this plan is a copy of the Uranium Soils Integrated Demonstration: Phase II Operating Procedures. These procedures cover the operation of the individual pieces of equipment used in the CRU5/ID soil decontamination pilot plant.

9. REFERENCES

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13. Fernald Site DQO Number TS-001i.

APPENDIX A: SAMPLING AND ANALYSIS PLAN

1. Logistics, Equipment, and Communications

It is the responsibility of the project engineer and FEMP personnel to identify all logistical activities, to ensure that the necessary equipment and other resources are available on-site, and to ensure that the proper lines of communication and the principle contacts have been identified.

The Uranium Soils ID will coordinate the communication network and ensure that the primary points of contact have been identified, including names, telephone numbers, and addresses. This network will ensure that sample and data flow are proper; that all logistical, technical, and QA issues are quickly and properly addressed; that any necessary corrective actions are addressed and approved; and that deviations from the demonstration plan and corrective actions are documented and communicated to the principle party responsible for overseeing each operation.

The demonstration program does not require any non-routine field equipment to be provided by FEMP. Samples of soil and leaching and rinse solutions will be taken from the SDPP by operational personnel. Radiological analysis services for collected soil samples and leaching solutions will be required.

Photographs will be taken as appropriate to document on-site procedures and operations and will be maintained in a photographic log. This log will include date, time, subject, frame, roll number, and photographer.

2. Sample Locations and Quantity

In this section, the sampling location and quantity are described for the batch tests. The sampling description for the continuous tests will be developed later.

The sampling plan for each batch test is summarized in Table A1. Figure A1 shows the location of the sample points described in Table A1. Table A1 and Figure A1 are included at the end of this appendix. As discussed in Section 5.2, a total of 16 batch tests will be run. This total includes (1) a start-up run (2) twelve tests to include all eight combinations of leaching agents and Fernald soils and to run two of these eight combinations in triplicate, and (3) three runs to assess the effectiveness of the attrition scrubber. Additional sampling details are described in the following.

Before the addition of chemicals to the leaching reactor, samples will be taken of the agitated slurry in the upper, middle, and lower sections of the tank (by inserting

the sample probe through the manhole) and of the slurry in the recycle line (the slurry is continuously pumped from the bottom to the top of the mixing tank). The samples will be analyzed for percent solids and uranium concentration in the soil. The results from these samples will be compared with one another (using the sample variance determined as discussed later). If no significant difference exists, the reactor vessel will be considered to be completely mixed. The measured uranium will then be used to quantify the uranium concentration in the feed soil. This measured uranium concentration will be compared with the uranium concentration measured from sampling the feed soil drum.

Samples will be taken from the reactor during leaching to determine the efficiency of uranium removal from the soil. Samples will be taken at times 0, 5, 15, 30, 60, and 120 minutes. The samples will be filtered and the filtrates submitted for analysis. The results from the analyses of the samples will be compared to ascertain that, by the end of the leaching period, little or no change in the uranium concentration in the soil is occurring. Also, leaching efficiency as a function of time will be determined. The solids from the samples at 0 and 120 minutes will be analyzed to determine if any change in particle size distribution has occurred during leaching.

Isotopic analyses of samples of the treated and rinsed soil will be done to determine if partitioning of uranium isotopes between the different soil fractions occurs. Such partitioning is not expected.

In addition to the sampling for test purposes, the process water discharged from the pilot plant will be sampled and analyzed for various metals. The water will be analyzed for CWA Section 307 toxics: antimony, arsenic, beryllium, cadmium, mercury, selenium, thallium, and zinc. Also, the water will be analyzed for the following NPDES regulated metals; chromium, nickel, lead, silver, and copper.

Samples of soil from the last rinse cycle will be sent to ORNL for analysis to determine the effect of the leaching process on the soil characteristics. The mineralogical characteristics of the soil will be determined and the uranium content will be measured by gamma spectroscopy and by neutron activation. Also, the soil samples will be used for phytotoxicity tests. The intent of these tests is to demonstrate that the soil treatments do not seriously curtail use of the cleaned soils agronomically or their use as fill materials for landscaping grasses, shrubs, and trees around buildings, etc.

As described above and in Table A1, many of the samples submitted for analyses are composite samples. That is, during a sampling event, samples are taken from several sections of, for example, the mixing tank, the leaching reactor, or the centrifuge wet cake drum. These samples are then composited to produce the sample

to be analyzed for the sampling event. For several purposes, it is necessary to determine the sampling variance. This will be done for several selected sampling events by separately analyzing the different samples that are combined to make the composite sample. The sampling variance will be determined for both soil and liquid samples.

Table A2 summarizes the number of samples specified in Table A1 for the sixteen batch tests. The table lists the number of samples required for each type of analysis.

Table A2. Samples Required for Phase II Batch Tests

ANALYSIS	NUMBER OF SAMPLES[a]
Per cent solids (or moisture content)	118
Uranium in soil	150
Uranium in liquid[b]	251
Metals	32
Uranium isotopic distribution	16
Gross alpha/beta	16

[a] In addition to the numbers shown, approximately 100 QC samples will be required.

[b] The pH of all liquid samples will be measured.

For data validation purposes, the following QC samples will be taken and analyzed:

Uranium total: laboratory control sample, method blank, matrix spike, and duplicate.

Uranium isotopic: reagent blank, tracer, and matrix spike.

3. *Sample Collection and Handling Procedures*

Procedures to be employed by this demonstration program have been adopted from FEMP Standard Operating Procedures. As such, details of these procedures may be found in the FEMP sitewide Quality Assurance Project Plan (SCQ). In particular, the following sections of the SCQ apply to this demonstration program:

5.1	▸	Daily Logs
6.7	▸	Field Storage and Shipment of Samples
6.8	▸	Decontamination Procedures
7.	▸	Sample Custody
9.2	▸	Radiological Analysis
10.	▸	Internal Quality Control Checks and Frequency
11.	▸	Data Reduction, Validation, and Reporting
14.	▸	Specific Routine Procedures to Assess Data Precision, Accuracy, and Completeness

The specific sample collection and handling procedures to be used are essentially those as described in the Environmental Monitoring/Site Media Sampling Sample Plan, 93-432, Soil Washing Pilot Plant (EM-SMPLN-93-432, REV-0). This plan was used for the CRU5 Phase I work. The plan will be modified to be consistent with the Phase II batch operation as described in Section 3.1.

One addition to the Phase II sample plan will be a procedure for filtering slurry samples and rinsing the filter cake that is produced. A system consisting of Buchner funnels, filter flasks, and a vacuum pump will be set up at the SDPP for this operation.

Table A1

Sampling Plan for Batch Soil Leaching Tests

TEST NUMBER 00

Sample Point	Sample Location	Description of Sample	Quantity of Sample Required	Sampling Methodology	Sample Preparation	Parameters for Analysis	Analytical Methodology
S-1	Contaminated soil in feed drum	Soil	TBD	Take 3 grab samples with scoop from drum while feeding conveyor. Sample at 3/4, 1/2, and 1/4 levels in the drum.	Composite the 3 grab samples for analysis.	Per cent solids Uranium in solids [a]	Method No. 3002 [b]
S-2	Trommel oversize	>4.75mm material from soil (small rocks and pebbles)	TBD	After completion of trommel operation, take representative sample of >4.75mm material.	None	Per cent solids Uranium in solids [a]	Method No. 3002
S-3	Vibrating screen oversize (after trommel step)	None					
S-4	Mixing tank (before size fractionation step)	None					

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Sample Point	Sample Location	Description of Sample	Quantity of Sample Required	Sampling Methodology	Sample Preparation	Parameters for Analysis	Analytical Methodology
S-5	Centrifuge centrate (from size fractionation step)	Slurry - <20 μm soil in water	TBD	After completion of the centrifuge operation, obtain samples of the centrifuge centrate collection tank. Sample with a coliwasa tube (or equivalent).	For per cent solids sample: None For Uranium samples: Filter or centrifuge the sample. Sample filtrate for analysis. Rinse filter cake & sample filter cake for analysis.	Per cent solids in centrate Uranium in filter cake • Uranium in filtrate pH of filtrate	 Method No. 3002 Method No. 3002
S-6	Centrifuge wet cake + vibrating screen oversize	Wet cake (high solids content) - >20 μm soil	TBD	After completion of the centrifuge operation, sample the drum containing the centrifuge wet cake and the >50 mesh material from the screen. Take 3 representative samples with a scoop.	Combine the 3 samples and homogenize the mixture. Collect a sample of the mixture for analysis.	Per cent solids Uranium in solids •	 Method No. 3002
S-7	Attrition scrubber effluent	Wet cake (high solids content) - >20 μm soil	TBD	After each 15 min. period, collect a sample of the attrition scrubber product.	None.	Uranium in solids. Particle size distribution.	Samples to be analyzed at ORNL.

Sample Point	Sample Location	Description of Sample	Quantity of Sample Required	Sampling Methodology	Sample Preparation	Parameters for Analysis	Analytical Methodology
S-8	Leaching reactor	Slurry - soil in carbonate solution	TBD	Sample at 0 (before chemicals addition), 5, 15, 30, 60, and 120 minutes. At 0 and 120 minutes, take 4 samples - one sample from the recycle line using a dipper and three samples at different depths in the reactor with a coliwasa tube (or equivalent). At 5, 15, 30, and 60 minutes, obtain duplicate samples by coliwasa tube (or equivalent) from the top of the reactor.	For 0 and 120 minute samples: Record sample weight. Add polymer to sample and filter sample. Collect filtrate for analysis. Rinse filter cake and collect filter cake for analysis. For 5, 15, 30, and 60 minute samples: Add polymer to sample and filter sample. Collect filtrate for analysis.	<u>0 & 120 min. samples</u> Dry solids weight Uranium in solids * Uranium in liquid pH of liquid <u>5, 15, 30, & 60 min. samples</u> Uranium in liquid pH of liquid	 Method No. 3002 Method No. 3002 Method No. 3002
S-9	Vibrating screen oversize (after leaching step)	>2 mm material from soil	TBD	After completion of screening operation, take representative sample of >2mm material.	None.	Per cent solids Uranium in solids *	 Method No. 3002

0 56

7 5 8 3 7 8

Sample Point	Sample Location	Description of Sample	Quantity of Sample Required	Sampling Methodology	Sample Preparation	Parameters for Analysis	Analytical Methodology
S-10	Rinse cycle solids	Wet cake (high solids content) - total soil	TBD	After completion of rinsing the treated soil, sample the drum containing the centrifuge wet cake. Take 3 representative samples with a scoop.	Composite the 3 grab samples for analysis.	Per cent solids. Uranium in solids [a] Uranium isotopic distribution. Gross alpha/beta.	 Method No. 3002 Thermal mass spec.
S-11	Rinse cycle rinsate	Slurry (low solids content) - soil fines in spent carbonate solution and rinse water	TBD	After completion of rinsing the treated soil, obtain a sample of the centrifuge centrate collection tank. Sample with a coliwasa tube (or equivalent). Note: The rinsate will be contained in at least two mixing tanks.	For per cent solids: None For U analysis: Filter or centrifuge the sample. Sample the filtrate for analysis. Rinse filter cake and sample filter cake for analysis.	Per cent solids Uranium in solids [a] Uranium in liquid pH of liquid Metals [c]	 Method No. 3002 Method No. 3002