

**Demonstration and Evaluation of the CORPEX TM Nuclear
Decontamination Process Technical Task Plan #SR152005 Final
Report**

by

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**DEMONSTRATION AND EVALUATION OF THE CORPEXTM NUCLEAR
DECONTAMINATION PROCESS (U)
TECHNICAL TASK PLAN NO. SR152005
FINAL REPORT**

**C. G. May
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April 30, 1997

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ABSTRACT:

In June, 1995, the Decontamination and Decommissioning Focus Area funded a demonstration of the CORPEX™ Nuclear Decontamination Process in an Old Metallography Laboratory glovebox at the Savannah River Site. The objective of the demonstration was to prove the effectiveness of a new and innovative technology that would reduce the risks associated with future cleanups of plutonium-238 contaminated equipment in the DOE complex. After facility and vendor preparations in support of the demonstration, Westinghouse Savannah River Company (WSRC) was informed by the vendor that the chemistry proposed for use in the decontamination process was not effective on sintered plutonium, which was the form of plutonium in the selected glovebox. After further technical evaluation, the demonstration was canceled. This report describes the work performed in support of the demonstration and the present status of the project.

INTRODUCTION:

The objective of this task was to demonstrate and evaluate the effectiveness of the CORPEX™ Nuclear Decontamination Process for decontaminating plutonium-238 facilities and equipment. The CORPEX™ chemical process is a nondestructive cleaning method that removes only the contaminant and the matrix that fixed the contaminant to the surface. It does not damage the substrate. The cleaning agent is destroyed by the addition of proprietary oxidizers, leaving water, carbon dioxide and nitrogen gases, and a sludge as waste.

Plutonium-238 contaminated facilities are located at Savannah River Site (SRS), Hanford, and Los Alamos, and are some of the worst in terms of risk to personnel and difficulty of decontamination. The glove box line in the Old Metallography Laboratory in Building 235-F at SRS was the selected site for the CORPEX™ demonstration. The surfaces have both transferable and fixed contamination. A successful demonstration would have lead to an alternative method for waste minimization, material recycling, and pollution prevention across the Department of Energy (DOE) complex.

BACKGROUND:

In May, 1995, the Department of Energy Savannah River Operations Office (DOE-SR) directed WSRC to prepare a Technical Task Plan (TTP) to secure D&D Focus Area funding for a decontamination demonstration in the Old Metallography Laboratory in Building 235-F at the Savannah River Site (SRS). The demonstration was intended to demonstrate the effectiveness of the CORPEX™ Nuclear Decontamination Process for the removal of Pu-238.

The TTP was prepared by the Savannah River Technology Center (SRTC) with assistance from Nuclear Materials Stabilization Program (NMSP) decontamination group personnel. The original intent of the TTP was to complete the demonstration before the end of Fiscal Year 1995 (FY95). The TTP, No. SR152005, was for \$490K which included \$290 for vendor services to provide and apply the CORPEX chemistry. The remaining \$200K was to provide support services and facility preparation.

The TTP was submitted on May 12, 1995, and approved by the Morgantown Energy Technology Center (METC) on June 12, 1995.

After the TTP was approved, a core team made up of individuals from key supporting WSRC organizations began developing a detailed Scope of Work (SOW), estimate, and schedule. Through this effort the full extent of facility support was realized resulting in the original estimate of \$490K increasing to approximately \$1800K (with contingency).

The work required by the detailed SOW prohibited the completion of the demonstration in FY95. A Baseline Change Proposal (BCP) was necessary to reprogram the FY96 235-F work scope to accommodate the demonstration in FY96. Approximately \$483K of EM50 funding was carried over to FY96 with the balance of funding (\$1300K) provided by NMSP Operations.

The core team also started discussions with the vendor during this planning stage. CORPEX Technologies Inc., manufacturer of the CORPEX™ chemistry, required a licensed applicator to apply the CORPEX™ chemistry. MELE Associates (MELE) was identified as the applicator for this technology. Discussions at this time involved the SOW, CORPEX processes, and MELE's proposal for meeting the demonstration requirements.

One of WSRC's early concerns was the effectiveness of the CORPEX chemistry on Pu-238. This was a major topic during several telephone conferences and meetings. CORPEX considers the chemical makeup of their products proprietary and was reluctant to divulge detailed information concerning the chemistries and how they work. WSRC technical personnel continued to question whether the chemistry would dissolve Pu-238; however, CORPEX's data indicated that it would.

A subcontract with MELE was awarded on November 27, 1995. A price and delivery schedule that identified milestones where payment would be made upon completion was established as part of the subcontract.

DESCRIPTION OF THE CORPEX™ NUCLEAR DECONTAMINATION PROCESS

The CORPEX™ Nuclear Decontamination Process consists of the application of a variety of chemicals depending upon specific conditions and contaminants. The chemistries required in the 235-F demonstration were CORPEX™ 921, CORPEX™ 960, and CORPEX SMEARAWAY™.

The application process involved the continuous spraying of the CORPEX™ 921 chemistry on the glovebox interior surface. The CORPEX™ 921 is a chelating chemistry that dissolves and holds the contamination in solution preventing recontamination of the material being cleaned. The effectiveness of solution is dependent on the time in contact with the material being cleaned and the temperature of the solution. The optimum temperature is 90° C (194° F). Reductions in temperature increase the time of exposure to the chemistry required to achieve comparable decontamination factors. At optimum

temperature, decontamination times may range from minutes to a few hours while at room temperature, times may be several days or the process not effective at all. Based on a predicted temperature of 60° C (140° F) for the SRS glovebox, exposure to the solution was estimated to be 16 hours for an effective decontamination cycle. The vendor estimated that this time and temperature would give an effective decontamination.

When the decontamination process is complete, a thorough rinse with deionized water is required. The chelants in the rinse water and spent CORPEX™ 921 are then destroyed by adding the CORPEX™ 960. The CORPEX™ 960 is an oxidizer that reacts with the CORPEX™ 921 to produce waste products of water, sludge, and a mixture of gases (nitrogen, carbon monoxide, and carbon dioxide). The sludge contains the contaminants removed from the material being cleaned and manganese dioxide. After the oxidation of the chelants, the waste contains no hazardous materials resulting from the CORPEX™ chemicals.

A sucrose solution is added to neutralize any oxidizer remaining from the destruction process.

The application of the SMEARAWAY™ was changed during the planning. In the original proposal, the entire glovebox was to be sprayed with SMEARAWAY™ as an initial decontamination step. SMEARAWAY™ is a surfactant and CORPEX considers its chemical makeup proprietary. After there were problems in obtaining sufficient chemical information for a waste analysis, MELE removed SMEARAWAY™ from the process. When the problem with the sintered plutonium surfaced, CORPEX revised the process to once again include SMEARAWAY™ on areas where CORPEX™ 921 was not effective. CORPEX provided chemical information for the waste analysis.

The equipment for applying the chemicals consisted of pumps, piping, holding tanks, sampling containers for waste characterization, waste transfer system, wet vacuum nozzles, and spray nozzles. All equipment, except the vacuum and spray nozzles, was contained in two vendor containment units designed for use in radiological conditions. The equipment was operated through glove ports in the units. The units were interconnectable and sized for moving through the building. The units were connected to the glovebox gloveports by a transfer sleeve.

FACILITY PREPARATION:

The preparation of the facility began in December, 1995, after the BCP was approved. The facility preparation covered three major areas: 1) Safety Analysis; 2) Waste Disposal; 3) Glovebox and Glovebox Room Preparation.

Safety Analysis

The Unreviewed Safety Question (USQ) process was positive and required a Safety Analysis Report (SAR) Addendum for the demonstration to proceed. The USQ and SAR Addendum required about twelve months for preparation and approval.

Waste Disposal

The demonstration was expected to produce approximately 100 to 125 gallons of liquid TRU waste. Approximately 30 gallons of CORPEX™ 921 were to be used and the rest of the liquid was from the rinse water. Building 235-F is not designed to handle liquids and

does not have a liquid waste stream. Also, WIPP, the DOE TRU waste repository, does not accept liquid TRU waste.

Two options for the liquid disposal were evaluated: 1) evaporation; and, 2) absorption. After many discussions and several different types of absorbents were evaluated, the decision to absorb the waste using an oil dry type absorbent was made because it was commonly used at SRS and would be accepted as a solid waste stream. The waste created by the demonstration was reviewed and approved by the WSRC Waste Characterization Board.

Glovebox and Glovebox Room Preparation

The glovebox in the Old Metallography Laboratory measures approximately 4 ft x 3 ft x 40 ft in length. The room where the glovebox is located has been out of service for several years. A major effort was required to bring the glovebox from standby to operating condition. The spread of contamination from the glovebox to other areas operating in the building was the primary concern during the demonstration since the room uses the same ventilation system as the rest of the building.

The glovebox is fabricated in sections and bolted together using a flanged joint and neoprene gasket. The glovebox is not designed to be liquid tight and the introduction of liquid inside the glovebox created the potential for leaks and the spread of contamination. A method of sealing the seams was designed to make the joints as water tight as possible. All reachable interior joints were first sealed with a sealant and then covered with a sealing tape. The outside joint flanges and bolts were sealed with the same sealant and a U-shaped rubber gasket. The outside flanges were then wrapped with an absorbent paper followed by a plastic covering to contain potential leaks.

The glovebox room walls and floors were lined with plastic and a false plastic ceiling installed to provide protection if there was a glovebox leak. Finally, the glovebox and room were completely covered with an application of stripcoat for further protection. The lined room served as a contamination hut for the vendor's containment unit and access to the glovebox. A temporary hut was erected outside the room in the hallway. This hut served as the step-off pad for the workers. These preparations provided three levels of radiological protection.

VENDOR ACTIVITIES:

MELE was responsible for the application of the CORPEX™ process. This included the design and fabrication of equipment necessary to spray the CORPEX™ 921 and protect the glovebox ventilation system against excessive moisture and liquids. MELE subcontracted the equipment design and fabrication to an outside company. MELE and CORPEX Technologies were also contractually obligated to provide chemistry and process information required for WSRC's safety analyses, waste evaluations, and overall evaluation of the decontamination process.

The first design submittal was unacceptable to both WSRC and MELE. MELE terminated the design agency's subcontract and hired another company for the equipment design and fabrication. This action delayed the May, 1996, demonstration to August, 1996. In July, 1996, the equipment fabrication was completed and demonstrated in a mockup glovebox at the fabricator's facilities. The equipment operated as designed and was acceptable with minor modifications.

The vendor and equipment arrived at SRS during the last week of July, 1996, for equipment setup and final preparations. WSRC completed design and fabrication of the mounting sleeve for the demister used to protect the glovebox HEPA filters and ventilation system. Four days before the demonstration was scheduled to start, CORPEX Technologies announced that the CORPEX™ 921 would not be effective on the sintered form of plutonium in the Old Met Lab glovebox.

The ultimate problem which caused the cancellation of the demonstration involved the sintered form of Pu-238. The 235-F process involves the heating or "firing" of the plutonium to temperatures of either 1100° C or 1600° C. This firing process produces a sintered form of plutonium. The sintered form is more insoluble than unsintered plutonium. During early conversations with MELE and CORPEX, WSRC used the term "high fired" plutonium rather than "sintered" plutonium. CORPEX claimed not to have understood this term and consequently did not equate it with insoluble plutonium. In the summer of 1996, when CORPEX attended a workshop and heard a presentation given by the Argentines about their decontamination problems with plutonium, they realized there was a problem. At this time, the demonstration was put on hold for further evaluation.

A technical evaluation was done by WSRC to decide a path forward after postponing the demonstration. The main issue was the CORPEX™ 921 would not dissolve sintered plutonium and the key step in the decontamination process would be the SMEARWAY™ chemistry application. The use of surfactants is not an innovative technology and requires the same labor intensive mechanical application of spraying, scrubbing, and wiping that has been used previously. No data was provided by the vendor which indicates SMEARWAY™ is more effective than other common surfactants. The CORPEX™ 921 application was considered innovative because of the chemical decontamination process and the remote application method that would reduce personnel exposure during the decontamination step.

CURRENT STATUS:

On January 13, 1997, WSRC advised MELE that their contract was being terminated under the Termination for Convenience clause. On March 7, 1997, MELE submitted their charges to terminate the subcontract. WSRC is presently evaluating those claims and will be working to complete the cancellation.

At the time of cancellation, MELE had delivered services that met four of seven payment milestones identified in the delivery schedule of the subcontract. The four milestones are valued at \$210,000 and have been paid by WSRC. Also paid is \$4470.00 for additional vendor travel. Total subcontract costs paid to the vendor are \$214,470.00.

The value of the remaining three milestones is \$80,162.

An additional charge of \$23,953.14 for delay charges was submitted by MELE. These charges will be settled during the cancellation of the subcontract.

The costs to date of the facility preparation and demonstration support are approximately \$1100K.

It was estimated that an additional \$750K - \$1M in FY97 would have been needed to complete the demonstration.

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