DOE/MC/31189 -- 5035
(DE96000557)

Recovery and Removal of Mercury from Mixed Wastes

Final Report
September 1994 - June 1995

William F. Sutton
Thomas E. Weyand
Casimir J. Koshinski

June 1995

Work Performed Under Contract No.: DE-AR21-94MC31189

For

U.S. Department of Energy
Office of Environmental Management
Office of Technology Development
Washington, DC

U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Mercury Recovery Services
New Brighton, Pennsylvania

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Dle

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, 175 Oak Ridge Turnpike, Oak Ridge, TN 37831; prices available at (615) 576-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161; phone orders accepted at (703) 487-4650.

DOE/MC/31189 -- 5035
(DE96000557)

Recovery and Removal of Mercury from Mixed Wastes

Final Report
September 1994 - June 1995

William F. Sutton
Thomas E. Weyand
Casimir J. Koshinski

Work Performed Under Contract No.: DE-AR21-94MC31189

For

U.S. Department of Energy
Office of Environmental Management
Office of Technology Development
1000 Independence Avenue
Washington, DC 20585

U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By
Mercury Recovery Services
700 Fifth Avenue
New Brighton, Pennsylvania 15066

June 1995

TABLE OF CONTENTS

APPENDICES

LIST OF FIGURES

LIST OF TABLES

APPENDICES

EXECUTIVE SUMMARY

This report summarizes the results of a pilot-scale process demonstration program, **Removal and Recovery of Mercury from Mixed Wastes**, performed by Mercury Recovery Services, Inc. (MRS) in accordance with U.S. Department of Energy contract DE-AR21-94MC31189. The objectives of this program were to:

- demonstrate the capability of MRS's patented, thermal technology to remove and recover mercury from typical DOE waste streams,
- demonstrate the technical and economic feasibility of the MRS process to successfully remove and recover mercury from low-level mixed waste containing mercury compounds (HgO, HgS, $HgCl_2$) and selected heavy metal compounds (PbO, CdO),
- determine optimum processing conditions required to consistently reduce the residual total mercury content to 1 mg/kg while rendering the treated product nontoxic as determined by TCLP methods, and
- provide an accurate estimate of the capital and operating costs for a commercial processing facility designed specifically to remove and recover mercury from various waste streams of interest at DOE facilities.

The above objectives were achieved in a four-stage demonstration program.

Based upon the results obtained, it has been determined that,

- the base soil selected by MRS for use in this program was typical of soils found in the flood plain of East Fork Poplar Creek, Oak Ridge, Tennessee that contain mercury and low-level radioactive contamination as a result of releases from DOE's Oak Ridge facility,
- the optimization test work successfully removed mercury added to the soil in elemental and compound forms,
- the cerium oxide (CeO_2) added to the soil as a surrogate tracer to model the behavior of radioisotopes was retained in the soil and not carried over to the process gas handling system where mercury recovery takes place,
- the addition of lead and cadmium compounds to the soil did not have any adverse effect on the removal or recovery of mercury from the soil,
- the Naturally Occurring Radioactive Materials (NORM) added to the soil were retained in the soil and not carried over to the process gas handling system where mercury recovery takes place, and
- no secondary wastes were generated in the demonstration. Products were metallic mercury and soil with the NORM addition retained.

The capital investment required to construct a facility capable of processing 110 tons of waste per day is approximately $10.5 million dollars. Operating costs for the same facility are estimated at $107 per ton of waste processed.

The MRS technology offers a viable means of removing and recovering the mercury from low-level mixed wastes, obtaining an effective separation of the mercury from the radioisotopes by the retention of the radioisotopes in the solid residuals, and economically processing low-level mixed wastes of volumes generated at DOE facilities.

OBJECTIVES

The objectives of the project were to use a model sample material to:

1. demonstrate the capability of the MRS process to remove and recover mercury from typical DOE waste streams,

2. demonstrate the technical and economic capability of the MRS process to successfully remove mercury from low-level radioactive waste containing mercury oxide, mercury sulfide, mercury chloride and selected heavy metals (i.e., lead, cadmium, etc.),

3. determine the optimum processing conditions required to consistently reduce the residual mercury content in "typical" DOE waste streams to 1 mg/kg, or less, and render the treated waste nonhazardous as defined by TCLP leach testing, and

4. provide an accurate estimate of the capital and operating cost of a commercial treatment facility designed specifically to remove and recover mercury from the various waste streams of interest to DOE.

EXPECTED PROJECT RESULTS

The results that were expected at the conclusion of this project were:

- a residual mercury content in any treated sample of 1 mg/kg or less,
- all treated samples passing the TCLP test for mercury,
- a net treatment time of less than 12 hours per furnace cycle,
- recovery of the mercury in a form that can easily be refined,
- successful separation of the mercury from the radioactive components and other heavy metals contained in the waste, and
- capital and operating costs that are competitive with alternative waste remediation techniques operating at equivalent performance levels of mercury removal.

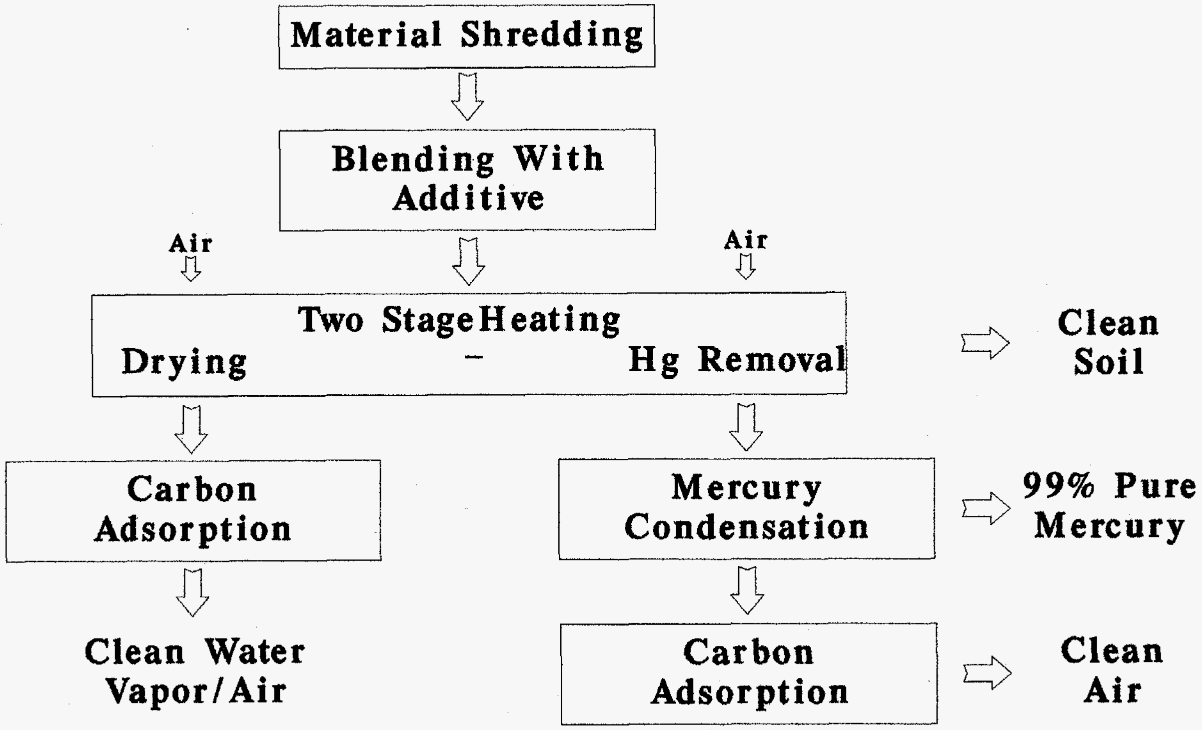
PROJECT BACKGROUND

In recognition of the major environmental problem created by mercury contamination of wastes and soils at an estimated 200,000 sites along U.S. natural gas and oil pipelines and at a number of government facilities, including Oak Ridge, Savannah River, Hanford, and Rocky Flats, the U.S. Department of Energy (DOE) is seeking an effective and economical process for removing mercury from various DOE waste streams in order to allow the base waste streams to be treated by means of conventional technologies.

In response to the need for improved mercury decontamination technology, Mercury Recovery Services (MRS), in conjunction with Pittsburgh Mineral & Environmental Technology, Inc. (PMET), has developed and commercialized a thermal treatment process for the recovery of mercury from contaminated soils and industrial wastes. The MRS/PMET Mercury Removal/Recovery Process whose flow sheet is illustrated in Figure 1:

- has consistently achieved residual mercury contents of <1 mg/kg in simulated soils doped with up to 3,000 mg/kg of mercury and mercury compounds,
- has been used to reduce the mercury content of contaminated soils excavated from sites along natural gas pipelines to a level at, or close to, background level (<1 mg/kg) such that the soil could be returned to its original location,
- produces a high metallic mercury product suitable for triple refining to high purity metal,
- produces no liquid effluent,
- generates a gaseous effluent having a mercury content that is normally below detection limits and consistently below OSHA respirator limits,
- involves technology that releases mercury from refractory compounds and eliminates gaseous sulfur and debris in order to achieve a preferred agglomerate size and desired surface area/volume ratio,
- combines the contaminated soil with additives that facilitate the decomposition of specific mercury compounds and/or reduce the content of gaseous sulfur and chlorine compounds in the process effluent,

Figure 1
Mercury Recovery Services, Inc.
Process Flow Sheet



- heats the material to be treated in a low-volume, low-velocity air stream in two stages: (i) a low-temperature stage during which low-temperature volatiles (i.e., water vapor, etc.) are vaporized without vaporizing significant quantities of mercury and (ii) a second stage at a temperature sufficient to vaporize the mercury (1000°F to 1200°F) from the dry material,

- exhausts the water vapor generated during the first heating stage to the atmosphere in gaseous form after passage through a gas purification system formulated to remove all traces of mercury and other impurities,

- condenses the mercury vaporized during the second heating stage to metallic mercury suitable for refining and recycling.

Tests performed in MRS's batch pilot system (250-pound capacity) using samples of three soils excavated from sites along the U.S. natural gas pipeline system produced the following results:

- the mercury content of three samples of sandy and clay soils containing 15,000 mg/kg, 900 mg/kg, and 255 mg/kg mercury was reduced to less than 1 mg/kg in each case,

- the bulk of the mercury removed from the soil was recovered in metallic form,

- the highest moisture content in the three soils, 14.7%, was easily removed without creating a liquid effluent or interfering with the effectiveness of the process,

- an oil and grease content of 2,400 mg/kg did not interfere with mercury removal and recovery,

- the sulfur contained in the as-received sample was retained in the treated sample,

- treated samples easily passed present EPA TCLP levels as well as the more demanding limits recently proposed by the US EPA, and

- the mineralogical structure of the soil was basically unchanged by the process as determined by X-ray diffraction analysis.

During the above pilot studies, the equipment operated consistently without measurable concentrations of mercury in the gaseous process exhaust.

The New Mexico Environmental Department (NMED) (1) and the U.S. Environmental Protection Agency (EPA) Region III (2) have classified the MRS/PMET Mercury Removal/Recovery Process as "Recycling" rather than "Waste Treatment" thereby simplifying permitting requirements, and the U.S. Environmental Protection Agency - National has confirmed the recycling classification and has declared the MRS/PMET process to be Best Available Demonstrated Technology (BDAT) for mercury removal and recovery.

Based upon the above results and favorable findings of the two environmental agencies, MRS, a joint venture, was formed by PMET, McCarl's Process Systems, Inc., and McCarl's, Inc. to commercialize the MRS/PMET process.

APPLICATION OF MRS TECHNOLOGY

There is little doubt that the MRS process will be able to successfully treat mixed wastes consisting of mercury-contaminated soils or industrial wastes containing low-level radioactive materials and other heavy metals (i.e., lead, cadmium, etc.). It is anticipated that the mercury will be removed, as described above, leaving the radioactive components in the treated soil or waste. The low carrier gas flow rates employed, along with the fixed beds of material being treated, result in very low dust carry-over to the condenser and carbon columns. In the unlikely event that radioactive material is carried over into the air purification system, it will be trapped in the carbon absorption columns and recirculated in the system when the mercury-contaminated carbon is blended with fresh waste for treatment at the end of the useful life of the carbon.

The MRS process has also been successfully demonstrated as a technique to remove mercury from the polymer sorbant used for in-situ removal of heavy metals from soil via electrokinetic migration at DOE's Savannah River site. In laboratory tests performed for the Isotron Company, the MRS process successfully reduced the mercury content of contaminated polymer sorbant from 500 mg/kg to 0.42 mg/kg and recovered the mercury in metallic form while retaining lead and cadmium in the treated material.

Utilization of the MRS technology to remove and recover mercury from soils and industrial wastes offers many technical, environmental, and economic advantages.

- By reducing the residual mercury content in treated wastes and soils to below 1 mg/kg and recovering and recycling the removed mercury, the MRS process generally eliminates public health risks and relieves the generator of any future liability associated with the mercury contamination.
- By maintaining all process equipment under negative pressure and placing the equipment in a secondary containment chamber, the MRS/PMET process utilizes redundant means to protect the environment and workers from mercury exposure in case of equipment failure.
- By applying proprietary technologies that (a) eliminate sulfur and chlorine compounds from the gaseous process effluent, (b) maintain mercury levels in the gaseous effluent well below OSHA respirator limits, and (c) recover and recycle the removed mercury, the MRS process is in compliance with existing air, water, and waste disposal regulations and dramatically reduces the environmental impact of the remediation process.
- Because of its energy efficiency, modular design, flexibility with respect to furnace size and capacity and its ability to be operated in a fixed or mobile mode, the MRS process is a relatively low-cost operation, and it is estimated that the cost of remediation using this technology will be comparable to or below costs of existing thermal processes.
- By operating without a liquid effluent, reducing the residual mercury content in the treated waste to less than 1 mg/kg, and by eliminating sulfur and chlorine compounds from the gaseous effluent, the MRS process produces no secondary hazardous wastes.

To date, the MRS technology has been successfully used to treat a variety of soils and K106 and D009 wastes and it is anticipated that this technology will find broad application in treating contaminated soils associated with natural gas and oil production and transmission, K106 and D009 wastes associated with chlor-alkali production, and a wide variety of soils, sludges, and sediments associated with industrial processes that produce or utilize, or have previously produced or utilized, mercury.

By contrast with the above advantages of the MRS process for mercury removal and recovery:

- Physical treatment technologies (a) normally produce at least two products ... a clean soil fraction and one or more fractions contaminated with low volumes of mercury that require further treatment, (b) cannot remove organic or inorganic mercury compounds or metallic mercury sorbed onto clay, and (c) normally produce a contaminated liquid effluent.

- Chemical treatment technologies normally utilize strong leaching solutions such as nitric or hydrochloric acids that (a) must be collected and treated to recover the contained mercury, (b) must be decontaminated and neutralized prior to disposal, and (c) have various degrees of effectiveness depending upon the alkalinity and nature of the soil or waste being treated.

- Immobilization, which involves several steps including comminution of the waste, reagent addition, and mixing with cement, greatly increases waste volume, normally is suited solely for low levels of contamination, does not remove mercury from the waste, and does not end generator liability.

- Biological treatment, in which bacteria are used to assimilate organic mercury compounds, is a very slow process in which the waste normally requires secondary treatment to remove nonassimilated mercury compounds.

- Electrolytic treatments, which are normally used to treat mercury-contaminated liquid wastes and sludges, usually involve physical separation or chemical leach steps prior to electrolysis and generally generate both solid and liquid secondary wastes.

METHODOLOGY

To achieve the previously defined objectives and results, MRS assembled and executed the scope of work described below:

- consulted with DOE personnel to select a base material to serve as a model waste stream that is deemed typical of mercury-contaminated wastes that must eventually be treated by DOE,
- completely characterized the base material with regard to the chemistry and structure of the waste matrix,
- modelled "typical" DOE waste streams by blending various quantities of mercury, mercury compounds, and select heavy metals into the typical waste material,
- optimized treatment process parameters for the base material and each additional waste type prepared,
- determined the concentrations of residual total and leachable mercury that can be reasonably expected for treated wastes,
- determined the amount of the surrogate tracer(s) in the treated material and elsewhere in the system including in the process piping, the recovered mercury, the process exhaust, and the various components of the processing system, and
- determined the type of equipment that best meets the specific needs of DOE and projected the operating and capital costs pertaining thereto.

The specific methodology within and among tasks performed over the course of this project is described below under "Tasks Performed."

TASKS PERFORMED

Task 1: Modelling of DOE Waste Streams

For this task, the intent of MRS was to identify and select a waste stream containing mercury and low-level radioactive contaminations produced by a DOE facility that was regarded as a typical mixed waste from a number of DOE facilities. As the process of selecting the waste developed, it became apparent that a typical waste could not be made available to MRS for testing by primary contractors at DOE sites due to difficulties in obtaining the release of material from DOE sites, regardless of whether the level of radioactivity was below regulatory limits, to a facility that did not possess an NRC permit.

For this reason, an alterative strategy was proposed by MRS and accepted by DOE-METC by which MRS would select an uncontaminated base material similar in matrix to a typical DOE waste stream to which mercury compounds and a nonradioactive surrogate tracer compound would be added to model a mixed waste. In addition, in selected tests, additional heavy metal compounds often found as cocontaminants with mercury would be added to evaluate their effect on removal and recovery of the mercury.

As a final demonstration, MRS would obtain and blend Naturally Occurring Radioactive Material (NORM) with the base waste matrix and mercury compounds in deminimis concentrations below regulatory concerns but significant enough to model actual waste behavior and to enable reliable analysis to be performed such that a radiochemical balance could be prepared and subject these blended materials to process conditions determined optimum by the previous suite of tests.

Task 2. Detailed Speciation Analysis

Since the key to successful removal and recovery of mercury from soils and industrial wastes is detailed knowledge of the form in which the mercury is present, the characteristics of the surrogate waste matrix in which it is contained and the potential interactions between the contained mercury and the matrix, the base soil sample obtained during Task 1 was subjected to a complete mineralogical analysis including:

a. chemical and physical analyses (soil characterization and TCLP testing),

b. particle size determination,

c. optical microscopy,

d. scanning electron microscopy,

e. X-ray diffraction, and

f. Radio-chemical analysis by gamma spectroscopy.

The mercury compounds that were added to the test samples were selected to simulate those species that are most often contained within primary and secondary waste streams as well as those that may be formed as a result of processing.

Task 3: Optimization of Processing Parameters

Task 3 was divided into three stages:

1. determination of optimum process conditions for treatment of each of the samples described in the scope for Task 1,

2. determination of the residual total and leachable (TCLP) mercury contents in samples processed under optimum conditions, and

3. confirmation of optimum nonradioactive, surrogate test parameters through tests using Naturally Occurring Radioactive Materials (NORM).

During this task, MRS evaluated the effects of a range of process variables on the removal and recovery of mercury from each sample listed in the description for Task 1. The process variables that were investigated included temperature, time at temperature, and, to a lesser extent, carrier gas flow rate. All test batches were processed in MRS's 200- to 250-pound capacity pilot treatment facility. The preferred processing regime and optimum processing conditions for each type were determined based upon the total retained mercury in the treated sample and the behavior of nonmercury impurities added to the waste stream.

The final stage of Task 3 involved treatment of samples containing mercury compounds and Naturally Occurring Radioactive Material (NORM) using process conditions deemed optimum by surrogate tests.

The goal of this task was to achieve a residual mercury content of 1 ppm or less, mercury TCLP leach values below regulatory limits, and greater than 90% retention of NORM.

During this task, special emphasis was placed upon the sampling and analytical procedures employed. In order to compensate for the "nugget effect" caused by inhomogeneity of the mercury-containing globules and particles in the sample matrix, all samples were thoroughly blended prior to splitting. Small-lot samples were obtained using a riffle splitter. Large samples, wet samples, and samples containing high clay contents were blended by shoveling on an oxidized steel plate followed by coning and quartering until the desired sample size was achieved. All chemical analyses were run in batches using a spiked control sample to check for extraction and instrument errors in the analytical procedure. In some cases a blind replicate sample was included for an additional measure of quality control. Splits of all samples were retained for use as blind replicates and to check the quality of analyses through a second laboratory, as necessary.

In addition to the above, in the tests utilizing radioactive compounds, all process feeds, treated materials, recovered mercury, and process exhaust were monitored for radioactivity in accordance with procedures that were established and carried out in conjunction with MRS by Central Environmental, Inc. (CEI). Throughout the project tasks leading to the use of the NORM, CEI worked closely with MRS to adapt the MRS safety and health and quality assurance programs to attain Nuclear Quality Assurance (NQA-1) standards.

Upon completion of Task 3:

1. the final samples treated under optimum conditions were subjected to soil characterization and total chemical analyses to determine the effect of soil treatment on the soil structure, and the results compared to those obtained on the starting materials analyzed during Task 2,

2. a mercury mass balance was prepared by summing the residual mercury contents of all treated samples, the total amount of metallic mercury recovered, and the amount of mercury contained in the air purification carbon columns, and comparing the result with the total amount of mercury processed during the program,

3. the pilot test equipment was disassembled and inspected for radioactive contamination. This inspection consisted of comparison of wipe samples collected from process equipment prior to and after completion of testing with NORM, and

4. all untreated base waste materials and treated materials containing NORM, at the conclusion of the program, were disposed at a properly licensed and secure waste management facility. All carbon will be desorbed in the MRS unit upon completion of its useful life. Recovered mercury will be retained by MRS as a reagent material.

The process regime and optimum processing conditions determined during Task 3 are the bases for the capital and operating cost estimates developed in Task 4.

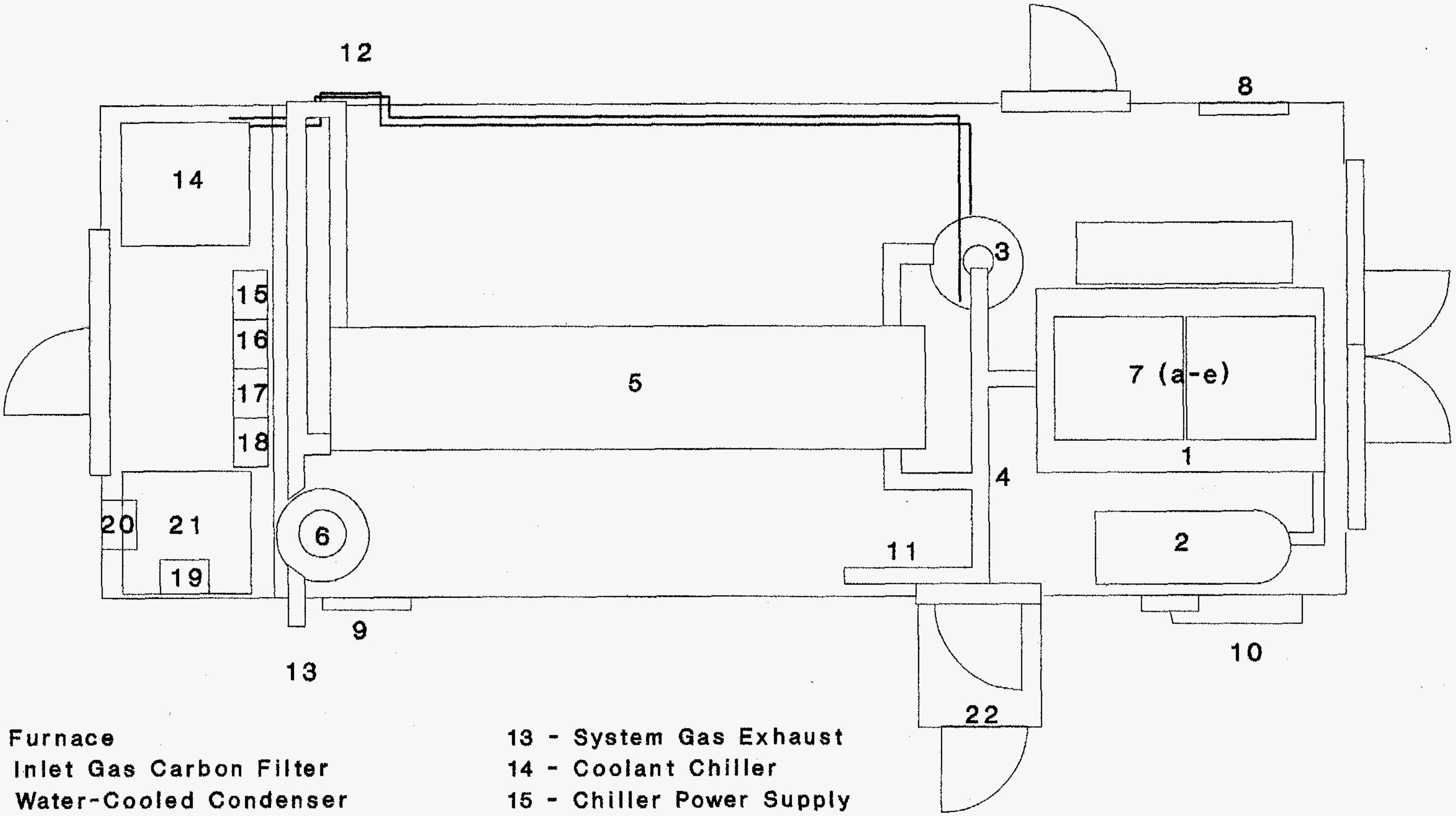
DESCRIPTION OF MRS PILOT-SCALE DEMONSTRATION UNIT

The pilot-scale demonstration unit used in this test program, illustrated in Figure 2, was designed and constructed to assure worker and environmental safety and the containment of hazardous materials in the event of a process upset, as follows:

a) The entire process operates under negative pressure to insure that failure of any sealing mechanism to perform results in air flow into the system rather than a release of mercury vapors into the surrounding area.

b) All processing equipment is maintained within an enclosure that provides secondary containment. Features of this enclosure include provisions to maintain a slight negative pressure within the enclosure, air lock access to its interior, and carbon filtration media on all air inlet and exhaust vents.

c) All system and containment chamber gas inlet and exhaust lines have carbon filtration canisters to prevent the release of mercury vapor from either the process or the containment system in the event of a process upset.



1 - Furnace
2 - Inlet Gas Carbon Filter
3 - Water-Cooled Condenser
4 - Condenser Bypass Line
5 - Carbon Columns
6 - Vacuum Pump
7 (a-e) - SS Trays
8 - Containment Air Inlet
9 - Containment Air Exhaust
10 - Process Gas Inlet/ Air Flowmeter
11 - Bypass Gas Inlet/ Air Flowmeter
12 - Condenser Line Air Flowmeter
13 - System Gas Exhaust
14 - Coolant Chiller
15 - Chiller Power Supply
16 - Furnace Power Supply
17 - Vacuum Pump Power Supply
18 - 110 Power Supply
19 - 110 Circuit Box
20 - Main Power Breaker
21 - Transformer
22 - System Air Lock/Entrance

Figure 2
MRS Pilot-Scale Demonstration Unit

The major components making up the pilot unit are the furnace, the heat exchanger, gas-purifying carbon adsorption columns, a vacuum pump, valves and piping, and instrumentation for the measurement of process temperatures, air flows, and exhaust mercury vapor concentration. The entire pilot system, including its protective enclosure, is self-contained and can be set upon a trailer by crane or fork-lift for transportation to a field operation. The only service requirement is approximately 30 kW power. System utilities which include the power transformer, all electrical switch gear, and the recirculating coolant chiller for the process heat exchanger, are contained in a compartment at the end of the skid and are separate from the processing equipment.

A brief description of the primary components of the process unit, shown in Figures 3, 4 and 5, and the general operation of the unit follows.

Figure 3. Exterior View of Demonstration Unit

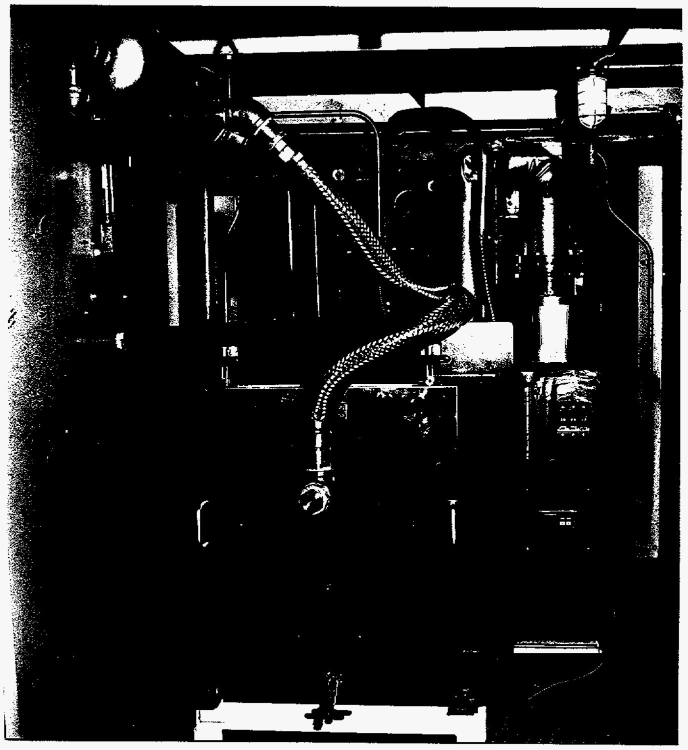


Figure 4. View of Furnace

Figure 5. Interior View of Demonstration Unit

Furance

The furnace is a standard heat-treating box type furnace that contains a solid, gas-tight liner or retort with a maximum temperature of 2050°F which has been modified to allow carrier gas (air) to pass through the furnace and into the system's carrier gas handling equipment.

The contaminated feed is charged in five (5) trays, each having a capacity of approximately one-half (0.5) cubic foot. The dimensions of each tray are 17" W X 16" L X 4" D. In the furnace, the trays are supported by angle that is welded to the retort walls.

Because the resistance heaters for the furnace are located within the walls of the heating chamber, the furnace charge is heated by both convective (hot process air) and radiant (retort shell) means.

Furnace air temperature is measured through a thermowell in the upper rear of the retort. Feed temperature is measured in the center of the tray located at the lower front of the furnace, which was determined through previous experimentation to be the lowest temperature point in the charge.

Heat Exchanger

The heat exchanger for the high-temperature process gases is approximately 34" high and 8" in diameter. Hot gases entering the top of the heat exchanger are distributed through parallel tubes which are surrounded by a coolant composed of water and ethylene glycol. The coolant is chilled by means of a 30,000-Btu/Hr recirculating chiller. In the event the recirculating chiller malfunctions, an arrangement is available by which water from a municipality or another source can be used to supply the system until it is brought back to equilibrium.

Mercury resulting from the condensation of vapors in the heat exchanger drops into a 7" high and 8" diameter collection pot attached to the bottom of the heat exchanger via a flange connection. A valve at the bottom of the collection pot is used to drain recovered mercury from the pot without need for total disassembly of the apparatus.

Carbon Adsorption Columns

All process gas leaving the system passes through a gas purification system that consists of carbon columns arranged in series. This system is capable of (a) cleaning the bypass air generated during the low-temperature drying cycle and (b) cleaning the gas exiting the heat exchanger during the high temperature segment of the cycle. The columns are designed to distribute process gases uniformly through the bed of sulfur-impregnated, activated carbon for efficient removal of the gas's mercury content.

Vacuum Pump

Air flow through the system is controlled and maintained by means of a vacuum pump.

Instrumentation

Process temperatures that are typically monitored by thermocouples are the furnace charge, furnace air, and furnace outlet air. Signals from these devices are routed to either digital readouts or a computer equipped with an automatic data collection system that logs and stores temperature data for future reference. Dial temperature indicators are used to monitor temperature of the air entering the bypass carbon columns, exiting the heat exchanger, and exiting the process unit at the exhaust.

Flow rates of furnace inlet air, flow rate of gas through the heat exchanger/carbon columns, and the total process exhaust from the vacuum pump are measured via turbine meters equipped with digital readouts.

A Jerome™ meter is used as a general survey meter to monitor ambient concentrations of mercury vapor in the work area and the containment system. The meter is also equipped with an automatic sampler and data logger that monitors the process gas at regular intervals throughout normal operations. In addition to the Jerome™ meter, Sensidyne™ gas sampling tubes are used as required to detect mercury vapor and other gases in the gas handling system and general work area.

General Operation

General operation of the pilot-scale demonstration unit consists of the following sequence:

- Arrangement of Process Gas Flows (Bypass or Heat Exchanger).
- Preparation of Feeds by Blending With Additives.
- Charging of Trays into the Furnace.
- Insertion of Thermocouple in the Charge and Sealing of the Furnace Door.
- Programming Furnace Controller (2 Stage - Drying and High Temperature Furnace Air Set Points).
- Monitoring of Process Temperatures and Exhaust Mercury Concentration.
- Change in Direction of the Gas Flows from the Bypass to the High Temperature Handling System.
- Shutdown and Cool Down
- Removal and Sampling of the Processed Charge.

After completion of a test program, the heat exchanger and process gas handling system are disassembled, the metallic mercury is removed, and the carbon filtration media is sampled and analyzed to determine its mercury content.

NQA - 1 QUALITY ASSURANCE PROGRAM

As part of the overall scope of this project, MRS, in conjunction with Central Environmental, Inc. (CEI), updated its standard quality assurance and safety and health plan in developing a Nuclear Quality Assurance Program as presented in American National Standards Institute (ANSI) and American Society of Mechanical Engineers (ASME) standard document; ASME/ANSI-NQA-1, 1989, "Quality Assurance Program Requirements for Nuclear Facilities" and its related forms in 10CFR50 Appendix B; "Quality Assurance Criteria for Nuclear Power Plants and Fuel Reprocessing Plants"; and DOE Order 5700.5C; "Quality Assurance." A summary of this document is presented in Appendix 3.

With regard to performance of the scope described in this report, the plan detailed specific guidelines regarding the training of personnel, record keeping requirements, and reporting requirements related to the receipt, storage, handling, and return of soils containing NORM.

Although the soils containing NORM were below regulatory concern, the test program involving these materials was run as though the soils were regulated, low-level, mixed wastes. Specific activities that occurred within the time frame covered by this project included:

- A one-day, eight-hour training session for MRS personnel involved in this and future projects dealing with radioactive materials on handling NORM which was conducted by MRS's Radiation Safety Officer (RSO),
- Establishment of a Controlled Area and a Personnel Survey Area around the area in which the soils were stored and handled. Personnel entering these areas were required to wear two layers of personal protective equipment (gloves, tyvek suits, etc.), respirators, and personal radiation monitoring devices (thermoluminescent dosimeters). In addition, upon leaving the controlled area, each person was surveyed using a contamination meter,
- Personnel participating in this project conducted surveys of the incoming material, general work area on a daily basis, and drums of soil upon shipment for disposal using survey and contamination meters. These data were logged and reviewed by the RSO on a daily basis, and
- Performance of wipe tests at the conclusion of the test program to determine whether any contamination had resulted from these activities.

At no time during this program did either of the meters detect radiation above local background levels.

Task 4: Scale-up Methodology & Estimation of Capital and Operating Costs

Task 4 consisted of preparation of (a) a preliminary design for a commercial processing facility capable of treating DOE wastes that fit within the envelope of the samples treated during the program and (b) a detailed projection of the capital and operating costs inherent in the commercial treatment of representative wastes in quantities pertinent to DOE's remediation projects.

The scale-up methodology set by MRS was designed to:

- minimize the technical risk involved in scale-up,
- provide an operating system that assures maximum efficiency and minimum downtime,
- offer a flexible approach to remediation of the soil in order to allow DOE to optimize operations with regard to labor cost and/or soil transport, and
- dramatically reduce the time between completion of the demonstration/test programs and initiation of commercial cleanup operations.

Based on the relatively large volumes of low-level mixed wastes containing mercury known to exist at a number of DOE sites, the approach used to prepare the scale-up and cost estimates was a modular system capable of processing approximately 110 tons per day or 40,000 tons per year. This approach offers a degree of flexibility in that the system can operate at one site for a period ranging from a few months to several years, be disassembled upon completion, and transported and reassembled for operation at another site therefore minimizing capital costs associated with construction of several fixed-site facilities while maintaining the lower operating costs that typically accompany larger-scale operations.

LICENSING AND PERMIT REQUIREMENTS

All test work described in this report was performed at the MRS/PMET facility in New Brighton, Pennsylvania which is authorized to perform the program without obtaining specific permits for the following reasons:

- MRS received verbal notification from Ms. B.R. Heath of the NRC stating that an NRC license is not required to receive, handle, or process soils with concentrations of NORM as added to the base soil material as part of this test program (3),

- MRS received a statement from Pennsylvania Department of Environmental Resources (PA DER) that no permits were necessary to perform the testing described herein with regard to processing soils containing the heavy metal and radiochemical additions described herein (4),

- During the latter part of the test program in which soils containing NORM additions were being evaluated, several local and regional representatives from PA DER's Bureau of Radiation Protection and Bureau of Waste Management - Hazardous and Residual Waste Divisions visited MRS to review the scope of the project, the specific test procedures being employed, and to tour the pilot-scale demonstration unit. All comments were constructive and confirmed the regulatory compliance of the test program,

- Since (a) the mercury used in the program is the result of additions made by MRS, recovered mercury will either be maintained by MRS or sent to Bethlehem Apparatus, an EPA-approved recycler of mercury, for recycling and (b) there are no secondary solid wastes generated by the MRS process, no solid waste permits are required, and

- MRS did not require a specific air quality permit in order to perform the pilot study because the pilot unit exhausts air within MRS's lab structure. No exhaust gases are vented to the atmosphere outside the facility structure.

RESULTS

The following sections describe results obtained over the course of the scope performed, as obtained by task.

Task 1: Modelling of DOE Waste Streams

BASE SOIL MATERIAL

During Task 1, MRS consulted with DOE personnel in the identification, selection, and preparation of a surrogate uncontaminated material that is representative of the mercury-contaminated DOE waste streams that must eventually be treated to remove mercury prior to final management as a low-level radioactive waste. After several contacts, it was determined that the material which MRS would attempt to model through this program was contaminated soil found in the floodplain of Lower East Fork Poplar Creek (LEFPC) near Oak Ridge, Tennessee. The contamination found throughout this area is the result of discharges from the Y-12 plant of DOE's Oak Ridge facility. Having this material serve as the basis for the model base matrix to be used in MRS's test program contained several advantages: (a) nearly all DOE facilities have, to at least some degree, soil containing low-level radioactive and mercury contaminations, (b) the material was well characterized in numerous reports from studies performed on the contaminated material, and (c) the high clay and carbon content are especially challenging processwise, thereby adding a degree of conservatism to the process conditions and results.

In order to obtain a sample material having properties as closely similar as possible to the soil and sediment found in the floodplain of the LEFPC, MRS contracted a professional geologist to (a) research the LEFPC material to identify potential locations from which a nearly identical soil/sediment matrix could be obtained and (b) obtain approximately 12,000 pounds of material deemed most similar in matrix to that found in the LEFPC flood plain.

Data provided in three major sources:

> "An Investigation of Shallow Ground-Water Quality Near East Fork Poplar Creek, Oak Ridge, Tennessee" (5),
>
> "Mercury Assessment for Water and Sediment in Oak Ridge National Laboratory Streams" (6), and
>
> "Draft - Treatability Study Report for Mercury in East Fork Poplar Creek, Oak Ridge, Tennessee" (7)

enabled the identification and selection of soils in an area just north of LEFPC, in the floodplain of Poplar Creek, as the best available model for those found in the floodplain of LEFPC that contain low-level radioactive and mercury contaminations. This area was selected as the best available because a common ridge between the two streams provides runoff/drainage to both waterways. This provides for common silt/sediment components. Another characteristic of the soil found in the selected area that was strikingly similar to characteristics described in the first source listed above (5) was the presence of fine coal particles throughout the soil matrix. While the presence of coal in the LEFPC soils is attributed to discharges from the Y-12 plant, it is believed that the coal in the model soil is the result of runoff from mining operations conducted upstream from the selected area.

The specific location from which the model soil sample was collected was from the floodplain of the Poplar Creek drainage, north of Airport Road, approximately two and one-half (2.5) miles northwest of

Oak Ridge, Tennessee. The site is on the Windrock 7.5 minute topographic quadrangle, contained in Appendix 4, and 300 feet north of the Mile 20 marker on Poplar Creek. The sample was collected on private property, with the owner's release, from the floodplain just west of the creek using a rubber tired backhoe. In collecting the sample, less than six (6) inches of organic A horizon soil was scraped prior to excavation. The sample material then was taken from the next two layers of B horizon consisting of a layer of interlayered clayey silt and sand and layers of coal fragments, and light brown clayey silt and clayey sand found below the initial horizon. Sample soil was collected at depths as great as five (5) feet where a sticky brown clay layer was encountered. Since the intent was to focus on the clay-rich silt and sand layers, the heavy clay layer was left undisturbed.

The soil was delivered to MRS September 14, 1995 and assigned sample identification number 1577-1 upon its receipt. The sample was thoroughly blended and three 15- to 20-pound splits were separated and prepared for soil characterization, TCLP testing, SEM, XRD, size distribution, and optical mineralogy analysis performed in Task 2. The remainder of the bulk sample was split in two. One of the two splits was divided further into approximately 40-pound batch fractions that were sealed to retain moisture until processed in test work, while the second split was held as a reserve fraction.

SURROGATE TRACER

In order to evaluate behavior and response of radioactive isotopes to processing without actually using radioisotopes, a surrogate tracer compound, cerium (IV) oxide (CeO_2) was blended with the base soil material at a targeted concentration of approximately 5,000 mg/kg prior to each process test.

Cerium oxide was selected through consultation with a DOE representative based upon data provided in "Surrogate Formulation for Thermal Treatment of Low-Level Mixed Waste, Part III Plasma Hearth Testing" (8). This publication described cerium oxide's use as a conservative representative for both uranium and plutonium in thermal treatment applications. The use of cerium oxide in this demonstration was preferable because it is not considered to be a common soil mineral, thus eliminating the possibility of discrepancies due to naturally occurring quantities found in the base material.

For each test, five split fractions were blended with the surrogate tracer compound, mercury and/or mercury compounds, and/or other heavy metal compounds, depending upon the conditions specified for the particular test. By blending each of the tracer, mercury, and other heavy metal compounds with the soil immediately prior to each test, the potential for variance in feed concentrations was minimized while maximizing the ability to prevent spills and contamination.

NATURALLY OCCURRING RADIOACTIVE MATERIALS (NORM)

Although the surrogate tracer compound, cerium oxide, was designated and used in the majority of the process optimization testwork the final suite of process testing was performed on soils that were blended with Naturally Occurring Radioactive Material (NORM) as well as cerium oxide and mercury compounds. The purpose of adding NORM to the soils in the final stages of testing was to add a degree of confidence to the program by demonstrating process performance in the presence of radioactive isotopes while staying below concentrations of concern in state and federal regulations.

NORM used in this program was obtained from an oil production operation located in the Southern United States. It has been found that NORM often occurs as scale deposits along the internal walls of piping used to draw oil up from beneath the earth's surface. The scale forms as NORM and other soluble materials precipitate from water, found along with the oil, when the mixture cools as it is brought to the surface.

NORM removed from internal surfaces of oil production tubulars was collected and processed into a fine particulate form to provide a homogeneous concentration of the radioisotopes contained within the scale. Appendix 5 shows a typical gamma spectrum analysis of such a sample. The highest atomic weight isotope of interest is radium 226. This isotope, with a half-life of 1620 years, is a parent isotope of the NORM daughter isotopes which are produced by the decay of the radium 226.

The exempt concentration of radium 226 is any value less than 5 picoCuries per gram (pCi/g). Radiochemical analysis of the base soil material found that it already contained NORM with a radium 226 concentration of 1.68 pCi/g. Hence, this natural content had to be taken into account when calculating the amount of NORM material that could be added to the base soil as a tracer. It was also necessary to consider both the moisture and organic content of the soil, since the thermal treatment to remove the mercury would also remove the moisture and any component in the soil which would be volatilized at or below the maximum treatment temperature of 1200°F.

Blending of the NORM with the base soil material took place at a facility licensed in compliance with NRC regulations to handle radioisotopes at concentrations higher than the exempt concentration of 5 pCi/g. Before addition of the NORM, the soil was air dried to remove excess moisture and ground to facilitate uniform blending of the NORM throughout the soil. After blending, the soil was repackaged in sealed plastic bags, placed in two steel drums and shipped to MRS by common carrier. Because the concentrations in the soil were below the exempt levels, no permits were required to transport or receive the blended soil. Upon receipt at MRS, the blended soil was assigned sample identification number 1678-1.

ADDITIVE

The MRS technology utilizes a mineral additive that is blended with the feed prior to processing. The additive serves to fulfill many purposes within the process including (a) acting as a catalyst to decompose stable mercury phases at lower than equilibrium temperatures, (b) converting mercury found in mercury compounds to its elemental state prior to vaporization, enabling the mercury to vaporize and condense as metallic mercury, (c) retaining chloride and/or sulfide components, freed by the decomposition of mercury compounds, within the solid process residue as stable mineral phases, and (d) preventing the release of chloride or sulfur compounds in gaseous form, thus eliminating problems with corrosion and exhaust emissions.

SAMPLE BLENDS DESIGNATED FOR TESTING

The suite of samples designated for process testing included:

a. the base soil material (1577-1) and 5,000 mg/kg cerium as cerium oxide, CeO_2 (4 test batches),

b. the base soil material (1577-1) and 1,500 mg/kg mercury as mercury metal and 5,000 mg/kg cerium as cerium oxide (3 test batches),

c. the base soil material (1577-1) and 1,500 mg/kg mercury as mercury oxide, HgO, and 5,000 mg/kg cerium as cerium oxide (3 test batches),

d. the base soil material (1577-1) and 1,500 mg/kg mercury as mercury sulfide, HgS, and 5,000 mg/kg cerium as cerium oxide (3 test batches),

e. the base soil material (1577-1) and 1,500 mg/kg mercury as mercury chloride, $HgCl_2$, and 5,000 mg/kg cerium as cerium oxide (3 test batches),

f. the base soil material (1577-1) and metallic mercury, mercury oxide, mercury sulfide, and mercury chloride totalling 3,000 mg/kg mercury, 5,000 mg/kg cerium as cerium oxide, lead oxide (PbO) and cadmium oxide (CdO) at approximately 500 mg/kg each (4 test batches), and

g. the base soil material blended with approximately 3.25 pCi/g NORM (1678-1) and metallic mercury, mercury oxide, mercury sulfide, and mercury chloride in which total mercury contained will be 3,000 mg/kg, and 5,000 mg/kg cerium as cerium oxide (4 test batches).

Concentrations of cerium oxide, mercury and mercury compounds, and heavy metal compounds listed above were achieved by addition of the following amounts to each of the trays containing base soil material (5/test). For estimating purposes, a base soil weight of 40 lbs. (18,143.7 g) was used.

Table 1
Reagent Concentrations in Prepared Process Samples
(Based on 40 lb. soil weight)

REAGENT	CHEMICAL FORMULA	TARGET CONCENTRATION (mg/kg)	MASS ADDED/TRAY (g)
Cerium Oxide	CeO_2	5,000	111.4
Mercury	Hg	1,500	27.2
Mercury Oxide	HgO	1,500	29.4
Mercury Sulfide	HgS	1,500	31.6
Mercury Chloride	$HgCl_2$	1,500	36.8
Additive	Proprietary	10,000	180.0
Lead Oxide	PbO	500	9.8
Cadmium Oxide	CdO	500	10.4

In tests in which all of the mercury compounds listed above were used to obtain a total concentration of 3,000 mg/kg, one-half (1/2) the mass of each listed above was added to each tray.

All cerium, mercury, lead, and cadmium compounds added were reagent grade. Descriptions for each reagent used are presented in Appendix 6.

NORM Blending

NORM was blended with the base soil material based upon the following calculation:

Note: Specific Activity of Materials given in picoCuries per gram (pCi/g) of Radium 226.

NORM Material used as a tracer for tests 21, 22, 23, and 24, specific activity = 1433 pCi/g.

Soil received for tracing with NORM =1.68 pCi/g of Radium 226
Weight of soil =772.5 lbs.
Moisture content by weight =10%
Volatile Organic content =2.3%
Estimated total weight lost in processing =12.0%

Weight of soil after processing to remove all moisture and volatile organics
=772.5 X 0.88 lbs.
=679.8 lbs.

Total activity of NORM tracer required to give a maximum concentration of residual radium 226 after processing of 4.95 pCi/g
=(4.95-1.68)pCi/g x 679.8 lbs. x 453.6 g
=(3.27) x 679.8 x 453.6 pCi
=1008328.3 pCi

Weight of tracer material required =1008328.3/1433 grams
= 703.65 grams

Therefore, the NORM surrogate sample for treatment was made up of 772.5 lbs. of soil and had 703.65 grams of homogenized NORM with specific activity of 1433 pCi/g radium 226 added and the total was thoroughly mixed and analyzed.

Task 2: Detailed Speciation Analysis

After receipt and splitting of the bulk sample material, subsplits of 1577-1 were analyzed through a detailed characterization process consisting of the following:

a. Quantitative Chemical Analyses (including TCLP testing),

b. Optical Microscopic Analysis,

c. Particle Size Determination,

d. Scanning Electron Microscopy,

e. X-Ray Diffraction Analysis, and

f. Radio-Chemical Analysis by Gamma Spectroscopy.

Results from these analyses are presented below.

MINERALOGICAL SAMPLE DESCRIPTION

Optical Microscopy

The soil sample consists of a light to medium brown silty, clayey loam. Finely disseminated throughout the soil matrix are coarse (0.5 to 2.0 mm) to ultrafine (<0.03 mm) coal particles. There are minor to trace amounts of feldspar and fine agglomerations of hydrous iron oxides (goethite). The soil contains minor (<5%) amounts of organic debris consisting of wood, grass, and other plant remains.

Semiquantitative estimates of major soil constituents, as identified by optical microscopy, are presented in Table 2.

Table 2
Major Soil Constituents
Sample 1577-1, Base Soil Material
(Results Reported on Dry Basis)

SOIL MINERAL	CONCENTRATION (VOLUME %)
Quartz	49
Ferruginous, Micaceous Clay	30
Coal	10
Feldspar	8
Miscellaneous	3
Total	100

X-Ray Diffraction/Scanning Electron Microscopy

The mineralogical composition identified during the microscopic work has been confirmed through bulk X-ray diffraction analysis of the as-received soil sample. X-ray diffraction analysis reported that the sample primarily consisted of quartz with minor to moderate amounts of muscovite, chlorite (clinochlore), k-feldspar, and trace quantities of kaolinite.

The semiquantitative Scanning Electron Microscopy/Energy Dispersive X-Ray scan did not identify any chemical features beyond what would be considered typical - silicon, iron, and aluminum - for a soil of this type.

Clay Mineralogy

In order to determine the clay minerals present in the soil, a split of the as-received sample was screened at 0.002 mm to separate the clay fraction from the remainder of the soil components. Oriented, glycolated, and heat-treated slides of this material were then prepared and subjected to analysis by X-ray diffraction to determine species of clay present.

The results of this analysis showed that the sample's clay content is primarily made up of kaolinite. Other species of clay identified in lesser proportions included dioctahedral illite and chlorite.

Quantitative Chemical/TCLP Analysis

Results from quantitative chemical analyses performed on sample 1577-1 are presented in Table 3. Results of analyses to determine physical characteristics are presented in Table 4.

Table 3
Results of Quantitative Chemical Analyses
Sample 1577-1, Base Soil Material
(Results Reported on Dry Basis)

PARAMETER	METHOD	RESULT	UNITS
Ash (As-Received)	ASTM-D3174	94.59	percent
Cadmium	EPA 6010	5.1	mg/kg
Calcium	EPA 6010	178	mg/kg
Carbon, Total	ASTM D3176 (Modified)	22,300	mg/kg
Carbon, Total Organic	EPA 9060	22,300	mg/kg
Cerium	EPA 6010	70	mg/kg
Chloride	EPA 9252	150	mg/kg
Lead	EPA 6010	10.2	mg/kg
Mercury	EPA 7470	0.04	mg/kg
Pyridine	GC/MS Scan	<.05	mg/kg
Silicon	EPA 6010	115,000	mg/kg
Sulfur, Total	ASTM D-129	200	mg/kg
Sulfur, as Sulfide	EPA 9030	<1	mg/kg
Sulfur as Sulfate	EPA 9038	174	mg/kg

TCLP analysis for Metals, Volatiles, and Base Neutral Acids reported all but two items to be below limits of detection. The two constituents that were identified by TCLP were Barium, 0.92 mg/l, and Benzene, 0.05 mg/l, both of which are well below regulatory guidelines for each.

TABLE 4
Physical Properties of Sample 1577-1, Base Soil Material

PARAMETER	RESULT	UNITS
Moisture	22.73	weight percent
Loss on Ignition (After Dried)		
@ 600°C	4.73	weight percent
@ 1,000°C	5.25	weight percent
Cation Exchange Capacity	6.02	meq/100 g
Fuel Value (After Drying)	200	BTU/lb
Soil pH	6.22	

Particle Size Distribution

The particle size distribution for Sample 1577-1, as determined by wet screen analysis, is presented in Table 5.

Table 5
Results of Size Distribution Analyses
Base Soil Material, 1577-1

SIZE FRACTION (US No.)	WEIGHT PERCENT RETAINED
+10	0.02
-10+18	0.08
-18+30	0.30
-30+45	0.90
-45+70	9.50
-70+100	10.88
-100+200	21.88
-200+270	7.61
-270+500	11.61
-500	37.22
	100.00

These analyses indicate that the soil distribution is made up of approximately

10.8% Sand,
61.2% Silt, and
28.0% Clay.

Radio-Chemical Analysis

Radio-chemical analysis of the base soil material, 1577-1, found that the soil contained 1.68 pCi/g radium 226.

Analysis of the feed soil blended with NORM, 1678-1, reported a specific activity of 4.23 pCi/g. This result is within reasonable limits of the calculated target value for the specific activity, 4.36 pCi/g radium 226, which was determined based upon the amount of NORM added to the soil (703.65 g: 772.5 lbs) and the specific activity of the NORM, 1433 pCi/g Radium 226.

COMPARISON WITH LOWER EAST FORK POPLAR CREEK CONTAMINATED SOILS

After completion of the speciation program, data obtained from analysis of the base soil material were compared with data available on soils found within the floodplain of Lower East Fork Poplar Creek (LEFPC) that contain mercury and low-level mixed waste contaminations.

A comparison of common data for the samples is presented in Table 6.

Table 6
Comparison of Characteristics
Base Soil Material, 1577-1 vs. Oak Ridge LEFPC Soils

PARAMETER	MRS BASE SOIL MATERIAL 1577-1	LEFPC AVERAGE MERCURY SOIL	LEFPC HIGH MERCURY SOIL	LEFPC SEDIMENT	UNITS
Texture Class	Silty, Clay Loam	Loam	Silty, Clay Loam	Sandy Loam	
Moisture	22.9	36.9	48.3	29.8	weight %
Bulk Density	1.5	1.3	1.0	1.3	g/cc
Soil pH	6.1	7.5	7.5	7.3	
Loss on Ignition	@ 600°C 4.7				weight %
@ 1000°C	5.3	4.5	9.4	3.9	weight %
Cation Exchange Capacity	5.9	22.2	37.4	9.5	meq/100 g
Sand	10.8	31.0	12.3	69.6	weight %
Silt	61.2	49.2	58.7	17.1	weight %
Clay	28.0	19.8	29.1	13.3	weight %
Carbon, Total Organic	2.2	1.3	2.8	0.5	weight %

Data on the LEFPC soils were obtained from "Draft - Treatability Study Report for Mercury in East Fork Poplar Creek, Oak Ridge, Tennessee" (7)

Data presented in Table 6 show a strong similarity between the model soil material selected by MRS and the high mercury soils at Oak Ridge, particularly in textural class, carbon content, and particle size distributions. Differences in bulk density and cation exchange capacity may be the result of the higher moisture content and possible differences in characteristics of the water itself. Since the process performance is dependent more on thermodynamic than chemical properties, the difference in cation exchange capacity is expected to have little, if any, effect on mercury removal and recovery. Differences in moisture content have an effect on the time required to complete a furnace cycle but have no effect on mercury removal and recovery. After comparison with the Oak Ridge data, a determination was made that the model soil sample selected by MRS was representative of soils at Oak Ridge and suitable for use in process testing performed in Task 3.

Task 3. Optimization of Process Parameters

As described in the section describing Task 1, Modelling of DOE Waste Streams, Task 3, Optimization of Process Parameters, involved a series of primary process tests on a variety of prepared feeds consisting of combinations of base soil material, mercury and/or mercury compounds (-O, -Cl_2, and -S), heavy metal additives (PbO and CdO), and cerium oxide, the surrogate tracer compound. The objective of these tests was to evaluate the effects of variability in a number of different process variables including temperature, time at temperature, process air flow, etc. on the different feed combinations in order to determine optimum process conditions in terms of mercury removal, tracer compound retention, and behavior of additional heavy metal species. After determination of optimum parameters in the primary stage of testing, a second set of tests using soils blended with NORM in 'deminimis' proportions was performed to demonstrate the effectiveness of these same parameters in separating mercury from the radiochemical components. To confirm this separation, detailed analysis of product soils, recovered mercury, carbon filtration media, and process piping components were performed to construct material balances for mercury and NORM within the system.

PROCESS TEST RESULTS

Tables 7A through 7F present detailed summaries of process tests performed on soils that were blended with cerium oxide, mercury and/or mercury compounds, and lead and cadmium compounds.

Table 7A
Results from Process Testing
Soil (1577-1)/Cerium Oxide

Test ID	1	2	3	4
Process Time (hours)	2.8	2.0	1.5	2.3
Process Temperature (Degrees F)	1200	1100	1200	1000
Feed Soil- Mercury (mg/kg)	--	--	--	--
Feed Soil- Cerium (mg/kg)	4968	5069	5609	6201
Feed Soil- Pyridine (mg/kg)	<0.05	<0.05	<0.05	--
Product Soil- Mercury (mg/kg)	--	--	--	--
Product Soil- Cerium (mg/kg)	7400	6850	7950	5500
Product Soil- Pyridine (mg/kg)	<0.05	<0.05	<0.05	--
Product Soil- TCLP Mercury (mg/l)	--	--	--	--
Mercury Removal (wt%)	--	--	--	--

Table 7B
Results from Process Testing
Soil (1577-1)/Cerium Oxide/Mercury

Test ID	5	6	7
Process Time (hours)	2.2	2.0	1.5
Process Temperature (Degrees F)	1200	1100	1100
Feed Soil- Mercury (mg/kg)	1679.6	1728.0	1708.3
Product Soil- Mercury (mg/kg)	0.27	0.43	0.52
Product Soil- TCLP Mercury (mg/l)	<0.0002	<0.0002	<0.002
Mercury Removal (wt%)	99.99	99.98	99.98

Table 7C
Results from Process Testing
Soil (1577-1)/Cerium Oxide/Mercury Oxide

Test ID	8	9	10
Process Time (hours)	1.8	2.8	2.3
Process Temperature (Degrees F)	1200	1100	1100
Feed Soil- Mercury (mg/kg)	1671.7	1700.1	1729.5
Product Soil- Mercury (mg/kg)	0.42	0.99	1.10
Product Soil- TCLP Mercury (mg/l)	<0.0002	<0.0002	<0.002
Mercury Removal (wt%)	99.98	99.95	99.95

Table 7D
Results from Process Testing
Soil (1577-1)/Cerium Oxide/Mercury Sulfide

Test ID	11	12	13
Process Time (hours)	3.0	2.8	2.8
Process Temperature (Degrees F)	1200	1100	1100
Feed Soil- Mercury (mg/kg)	1672.6	1654.2	1781.7
Product Soil- Mercury (mg/kg)	0.81	0.15	0.14
Product Soil- TCLP Mercury (mg/l)	<0.0002	<0.0002	<0.002
Mercury Removal (wt%)	99.96	99.99	99.99

Table 7E
Results from Process Testing
Soil (1577-1)/Cerium Oxide/Mercury Chloride

Test ID	14	15	16
Process Time (hours)	2.0	2.0	2.5
Process Temperature (Degrees F)	1200	1100	1000
Feed Soil- Mercury (mg/kg)	1866.3	1736.5	1799.1
Product Soil- Mercury (mg/kg)	0.42	0.58	0.75
Product Soil- TCLP Mercury (mg/l)	<0.0002	0.0020	0.0012
Mercury Removal (wt%)	99.98	99.97	99.97

Table 7F
Results from Process Testing Soil (1577-1)/Cerium Oxide/Mercury/ Mercury Oxide/Mercury Sulfide/Mercury Chloride/ Lead Oxide/Cadmium Oxide

Test ID	17	18	19	20
Process Time (hours)	2.0	2.0	1.2	2.0
Process Temperature (Degrees F)	1200	1100	1100	1200
Feed Soil- Mercury (mg/kg)	3590.3	3747.3	3331.2	3139.5
Feed Soil- Lead (mg/kg)	600.0	626.3	556.7	524.7
Feed Soil- Cadmium (mg/kg)	600.5	626.7	557.1	525.1
Product Soil- Mercury (mg/kg)	0.29	0.58	0.88	0.20
Product Soil- Lead (mg/kg)	830.0	780.0	840.0	670.0
Product Soil- Cadmium (mg/kg)	1380.0	860.0	860.0	710.0
Product Soil- TCLP Mercury (mg/l)	<0.0002	<0.0002	<0.0002	<0.0002
Mercury Removal (wt %)	99.99	99.99	99.98	99.99

PROCESS DATA

For the test program,

- the average time required to dry the soil charge was 2 hours,
- the lowest average heating rate for the soil to go from 212°F to processing temperature was approximately four (4) degrees per minute. At this rate, approximately four (4) hours would be required to heat the soil from 212°F to 1200°F.

The two-hour drying period, four-hour heating period and a one- or two-hour hold at high temperature described here total to an eight-hour process cycle. This provides a 50% allowance for variability while maintaining the desired cycle time of eight hours.

Other pertinent data from this test program include

- the average airflow run through the furnace during the high temperature side of the process was 9 CFM,
- the average exhaust airflow was approximately 70 CFM, and
- the average mercury vapor concentration in the exhaust air was <0.01 mg/m^3, well below the OSHA standard of 0.05 mg/m^3 time-weighted average over an eight-hour period.

NORM TESTS

The final set of process tests performed in this program consisted of four tests run using the base soil blended with Naturally Occurring Radioactive Material (NORM) and mercury compounds which were conducted in accordance with procedures specified in MRS's NQA-1 Quality Assurance Procedures.

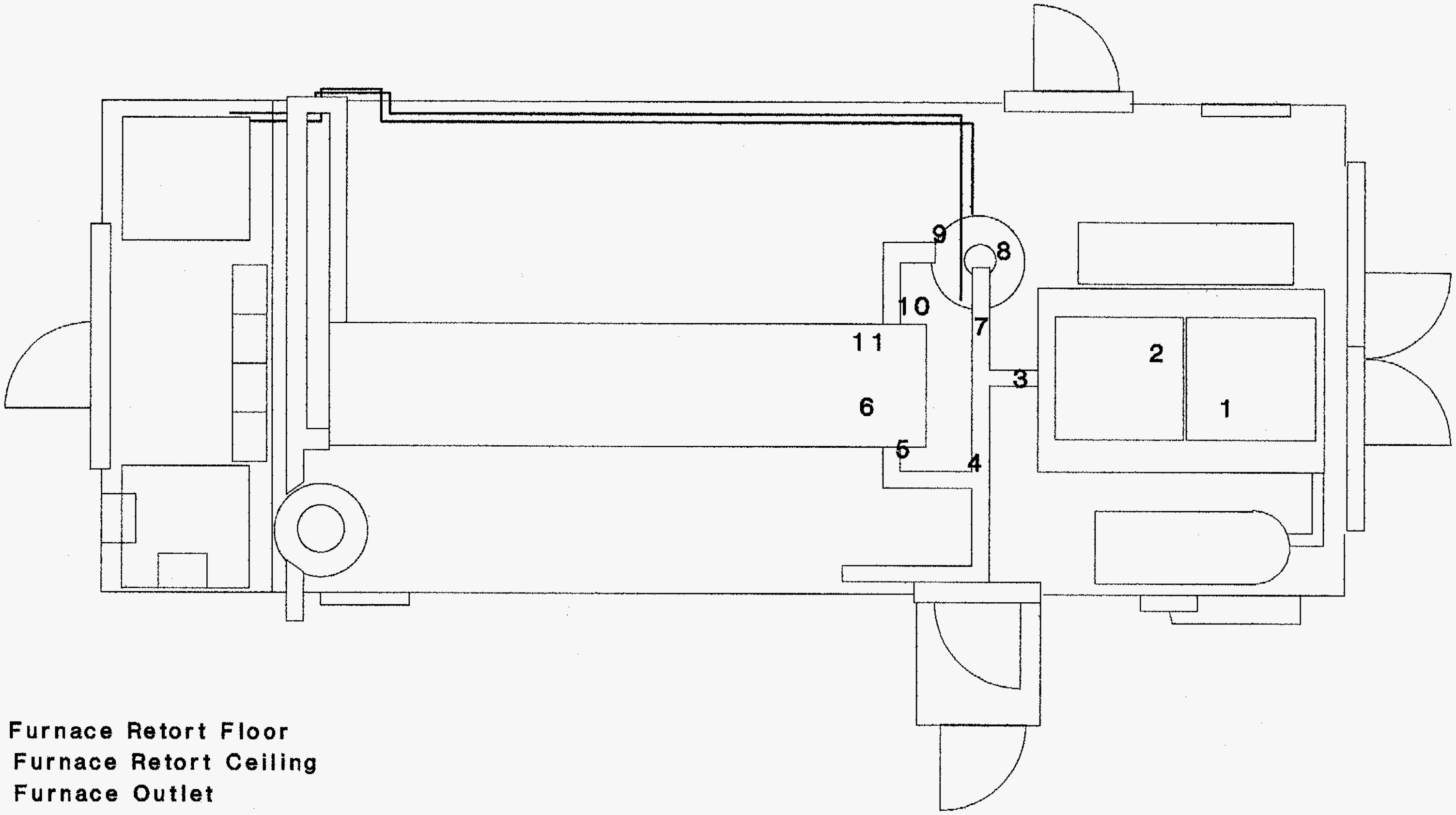
The base soil material used for these tests (1678-1) contained a specific activity of 4.23 pCi/g radium 226. As done in earlier tests, splits of this soil were loaded in process trays and blended with cerium oxide, mercury, mercury oxide, mercury sulfide, mercury chloride, and MRS's proprietary chemical additive and processed at different time periods at 1100°F and 1200°F. Results from these tests are presented in Tables 8 and 9 below.

Table 8
Results from Process Testing
Soil (1678-1)/Cerium Oxide/Mercury/Mercury Oxide/ Mercury Sulfide/Mercury Chloride

Test ID	21	22	23	24
Process Time (hours)	3.2	2.3	1.0	1.0
Process Temperature (Degrees F)	1200	1100	1200	1100
Feed Soil- Mercury (mg/kg)	3335.3	6913.1	3531.6	3552.5
Product Soil- Mercury (mg/kg)	0.44	1.92	1.07	1.45
Mercury Removal (wt%)	99.99	99.98	99.97	99.97

Table 9
Results of Radiochemical Analyses - Processed Soil Products

Test Number	Specific Activity Radium 226 (pCi/g)
1678-21	4.71
1678-22	4.88
1678-23	4.18
1678-24	4.15



1 - Furnace Retort Floor
2 - Furnace Retort Ceiling
3 - Furnace Outlet
4 - Fitting 1 - Bypass Piping
5 - Bypass Carbon 1 - Inlet
6 - Bypass Carbon 1 - Outlet
7 - Fitting 1 - Condenser Piping
8 - Heat Exchanger - Inlet
9 - Heat Exhanger - Outlet
10 - Condenser Carbon 1 - Inlet
11 - Condenser Carbon 1 - Outlet

Figure 6
Wipe Test Locations

WIPE TESTS

Primary Optimization Tests

After completion of the primary process optimization tests, process piping components were disassembled and wipe samples collected and analyzed for cerium, lead, and cadmium at the locations designated in Figure 6.

Wipe samples were collected by wiping a 10 cm x 10 cm section (100 cm^2) of the designated area with a square cloth patch, 7.62 cm to a side (58.0644 cm^2).

A comparison of results from these samples with results from samples collected at the start of the test program is presented below in Table 10. This comparison was generated to determine to what extent, if any, these compounds progressed beyond the retort in which the soils were housed during processing due to either thermal or dust migration.

Table 10
Comparison of Wipe Samples
Prior to Testing and After Completion of Base Process Tests

Sample Description	Cerium Start	Cerium End	Lead Start	Lead End	Cadmium Start	Cadmium End	Units
Retort Floor	< 5.0	< 5.0	2.8	6.0	< 0.5	3.8	$ug/100cm^2$
Retort Ceiling	< 5.0	< 5.0	3.2	< 2.5	< 0.5	16.2	$ug/100cm^2$
Furnace Outlet	< 5.0	< 5.0	2.5	53.7	< 0.5	98.2	$ug/100cm^2$
Fitting 1 - Bypass Piping	< 5.0	< 5.0	< 2.5	11.2	0.5	3.7	$ug/100cm^2$
Bypass Carbon 1 - Inlet	< 5.0	< 5.0	5.2	11.8	1.2	4.7	$ug/100cm^2$
Bypass Carbon 1 - Outlet	< 5.0	< 5.0	7.7	3.3	0.8	< 0.5	$ug/100cm^2$
Fitting 1 - Condenser Piping	< 5.0	< 5.0	< 2.5	133.0	0.5	125.0	$ug/100cm^2$
Heat Exchanger - Inlet	< 5.0	< 5.0	4.2	11.5	< 0.5	13.2	$ug/100cm^2$
Heat Exchanger - Outlet	< 5.0	< 5.0	4.2	< 2.5	2.0	< 0.5	$ug/100cm^2$
Condenser Carbon 1 - Inlet	< 5.0	< 5.0	5.0	3.2	2.5	< 0.5	$ug/100cm^2$
Condenser Carbon 1 - Outlet	< 5.0	< 5.0	< 2.5	< 2.5	0.5	< 0.5	$ug/100cm^2$

These results indicate that:

- the surrogate tracer compound, cerium oxide, did not exit the containment of the retort where the soil was housed during processing as a result of either thermal or dust migration, and
- lead and cadmium were detected in slight concentrations at the first pipe fitting, approximately one-third of the distance, between the furnace and the mercury condenser, but only in trace quantities at the point at which the pipe diameter expands at the inlet of the heat exchanger, where the mercury begins to condense.

Results from analyses performed on product samples from all the process tests as well as the above wipe tests, clearly show that an effective separation of the mercury from the soil as well as from the remainder of the soil's metal constituents had been accomplished in these tests.

Radio-Chemical Wipe Tests

Prior to the introduction of NORM into the soil matrix, the process pipework was disassembled and cleaned with wipe tests being taken during this operation. The disassembly, cleaning and wipe test sampling was repeated following completion of the NORM batch tests. No significant contamination was found on any of the wipes analyzed. Results from these analyses are presented in Table 11.

Table 11
Comparison of Radio-Chemical Wipe Samples Prior to Testing and After Completion of NORM Process Tests

Sample Description	Pre-NORM Gross Alpha (dpm)	Post-Norm Gross Alpha (dpm)	Pre-NORM Gross Beta (dpm)	Post-NORM Gross Beta (dpm)
Retort Floor	< MDA	0.07 +/- 1.02	< MDA	< MDA
Retort Ceiling	0.07 +/- 1.02	0.22 +/- 1.06	< MDA	< MDA
Furnace Outlet	4.04 +/- 1.84	4.04 +/- 1.84	< MDA	< MDA
Fitting 1 - Bypass Piping	0.51 +/- 1.14	0.96 +/- 1.25	< MDA	< MDA
Bypass Carbon 1 - Inlet	0.37 +/- 1.10	0.07 +/- 1.02	< MDA	< MDA
Bypass Carbon 1 - Outlet	0.37 +/- 1.10	< MDA	< MDA	< MDA
Fitting 1 - Condenser Piping	25.2 +/- 3.98	16.4 +/- 3.26	< MDA	< MDA
Heat Exchanger - Inlet	1.25 +/- 1.32	0.51 +/- 1.14	< MDA	< MDA
Heat Exchanger - Outlet	0.66 +/- 1.18	0.22 +/- 1.06	< MDA	< MDA
Condenser Carbon 1 - Inlet	0.37 +/- 1.10	0.81 +/- 1.21	< MDA	< MDA
Condenser Carbon 1 - Outlet	0.51 +/- 1.14	0.07 +/- 1.02	< MDA	< MDA

dpm = disintegrations per minute
Background Values:
Gross Alpha = 1.05 +/- 0.10 cpm
Gross Beta = 42.79 +/- 0.65 cpm
MDA - Minimum Detectable Activity
Gross Alpha = 0.702 dpm
Gross Beta = 3.910 dpm

The sensitivity of the NORM tracer detection level is illustrated by the consistency between the values reported from before and after the tests using NORM, and in many cases, this proximity of results caused the value reported for gross alpha prior to testing to be above the value reported after the tests with NORM.

In comparison to these results, the average Allowable Total Residual Surface Contamination (dpm/100 cm^2) from the DOE Order 5400.5 for Uranium 235, which is the parent isotope of radium 226, and other alpha emitters is 5,000 dpm average/100 cm^2.

CARBON COLUMN ANALYSES

After completion of the final process tests, carbon filtration columns were disassembled, emptied, and the contents weighed. During this process, samples were collected from the inlet, center, and outlet of each column. Results from these analyses are presented below in Table 12.

Table 12
Mercury Concentrations in Carbon Filtration Media
(After Completion of Tests)

Column	Inlet (wt %)	Center (wt %)	Outlet (wt %)	Average (wt %)
Bypass 1	9.67	5.34	1.09	5.36
Bypass 2	0.61	0.63	0.61	0.61
Bypass 3	0.71	0.62	0.57	0.63
Condenser 1	2.36	0.85	1.00	1.40
Condenser 2	0.81	0.79	0.78	0.79
Condenser 3	0.90	0.39	0.55	0.61

Analysis of carbon collected from the lead columns in each series for cerium reported that the cerium content was below limits of detection, <100 mg/kg. These results bolster the results of wipe samples reported in a previous section that did not identify the presence of any cerium beyond the retort chamber.

MASS BALANCE DATA

As part of this test program, mass balances were calculated for mercury and NORM to determine the distribution of each material in process products, equipment, etc. in order to firmly establish the quality of the separation obtained through processing by demonstrating in quantitative terms where each material is and isn't.

Mercury Balance

A mercury mass balance was developed for the entire test program based upon the amounts of mercury recovered as metallic product and analyzed contents of processed soils, carbon filtration media, exhaust air, and samples from process feeds in order to establish the distribution of mercury through the system after processing the contaminated feeds. This balance is summarized in Table 13.

Table 13
Test Program Mercury Balance

Material/Location	Mass Mercury (grams)	Distribution (%)
Metallic Hg (Condenser/Piping)	1898.4	46.2
Bypass Carbon	1588.1	38.7
Condenser Carbon	585.7	14.2
Exhaust Air	0.1	0.0
Product Soils	0.9	0.0
Samples	35.5	0.9
Total	4108.7	100.0

The total amount of mercury reported above, 4,108.7 grams, equals 100.6% of the total amount of mercury added to the system over the course of the test program, 4,082.5 grams.

NORM Balance

A balance for NORM material throughout the process system is presented below in Table 14.

Table 14
Radium 226 Balance, Tests 21-24

TEST #	1678-1 (lbs)	Reagents (lbs)	Samples (lbs)	Net Feed (lbs)	Post Process Weight (lbs)	Post Sample Weight (lbs)	Loss in Weight (wt %)
21	176	3.9	(2.8)	177.1	152.9	(2.3)	13.67
22	169	4.6	(1.5)	172.1	150.6	(2.6)	12.50
23	166	3.9	(1.7)	168.2	145.2	(2.2)	13.67
24	165	3.9	(1.7)	167.2	147.7	(2.7)	11.66
Totals	676			684.6	596.4		

Total Activity in Feed Material = (676 x 453.6 x 4.23) pCi
= 1297060.1 pCi

Residual Activity Calculation of Post Treatment Material

Test #	Calculation (lbs x g/lb x pCi/g)	Total Activity (pCi)
21	152.9 x 453.6 x 4.71	326664.12
22	150.6 x 453.6 x 4.88	333363.34
23	145.2 x 453.6 x 4.18	275306.16
24	147.7 x 453.6 x 4.15	278036.38
Total		1213370.00

Difference in pre- to posttreatment material = 83,690.1 pCi
(-6.4%)

Based upon the balance calculation presented above, the accountability for the NORM in the product soil is not only within the expected variability in analyses but also within the 90% target established in the project objectives.

TASK 4. Scale-up Methodology/Estimation of Capital and Operating Costs

The final task performed as part of this test program was the development of a preliminary design for a process plant of capacity suitable for processing quantities of low-level mixed wastes containing mercury at a single DOE location. The specific scope of this work included scaling the MRS process to a nominal capacity of 40,000 tons of waste per year (110 tons per day), designing a preliminary plant arrangement, and based upon these first two items, preparing capital and operating cost estimates for a plant of this capacity.

PLANT DESCRIPTION

The preliminary design MRS developed to process 40,000 tons per year (110 tons/day) of low-level mixed wastes containing mercury consists of three (3) independent process units.

Each process unit is made up of:

Two (2) Furnace Modules:

A furnace module consists of four (4) chambers, or muffles, that are connected to a single piping and control system. Four (4) trays, each containing 1,250 lbs. of waste, are placed into each muffle for each process cycle. Therefore, each furnace module has a capacity of ten (10) tons of waste per cycle. With an estimated process time of twelve (12) hours per cycle, each process unit has a capacity of 40 tons per day.

One (1) Air Handling System:

The first stage of the air handling system consists of a dehumidifier and inlet carbon filter through which carrier gas is passed prior to entering the furnaces. As it exits the furnace, (a) moisture-laden carrier gas is drawn through a series of carbon filtration columns to remove any traces of mercury vapor prior to being exhausted to the atmosphere and (b) mercury-laden carrier gas is passed through a heat exchanger to condense the mercury content of the gas and form a liquid metallic mercury product. After removal of the mercury, the stripped carrier gas is drawn through a series of carbon filtration columns to remove any traces of mercury vapor prior to being exhausted to the atmosphere.

Coolant for the heat exchanger is provided by a recirculating chiller. Carrier gas flow is maintained by a vacuum pump. Each air handling system is equipped with a spare vacuum pump and chiller for use in the event of failure of the primary process item.

Figure 7 illustrates the configuration of an individual process unit.

SITE ARRANGEMENT

The general arrangement for a site housing a plant with a capacity of 110 tons/day is shown in Figure 8. This particular site consists of a covered, 0.5 acre area that houses three complete process units described previously. The site is segmented into three primary areas:

a Waste/Soil Storage/Handling Area
an Air Handling Area, and
a Personnel/Office Area.

Segregation of the areas minimizes contamination throughout the site, particularly by segregating the waste/soil handling and preparation area at the furthest point from the administrative center.
The arrangement includes a security center through which all employees and visitors entering the area must pass, and a shower and decontamination area, or Contaminant Reduction Zone (CRZ), through which everyone entering and exiting the Contaminated Area must pass.

Capital cost estimates for the 110 ton/day plant described in the previous two sections are presented in Table 15.

Table 15
Capital Cost Estimate
110 Ton/Day Modular System

EQUIPMENT ITEM	INSTALLED COST (US $)
Furnaces	4,650,000
Chillers	180,000
Vacuum Pumps	72,000
Carbon Columns	132,000
Condensers	54,000
Additive Mixers	32,000
Additive Feeders	10,000
Charging Scales	13,000
Soil Shredders	14,000
Dehumidifiers	45,000
Tray Castings/ Fabrications	768,000
Structural Steel	85,000
Concrete/Foundations	280,000
Building Enclosure	1,125,000
HVAC/Ambient Air Cleaning	21,000
Instrumentation and Controls	230,000
Motor Control Centers	135,000
Piping and Valves	335,000
Electric Conduit/ Cable	160,000
Office/Lab/ Personnel Areas	110,000
Subtotal	8,451,000
Contingency	1,267,000
Engineering	780,000
Total	10,498,000

OPERATING ESTIMATES

Operating cost estimates for the 110 ton/day modular site described in the previous section are presented in Table 16. The bases for these estimates are described in further detail in the following paragraphs.

Supervision at the site will consist of a site manager, responsible for management of daily site operations, including scheduling of work, tracking of incoming and outgoing materials, general record management, etc. The site manager will be assisted by a technical supervisor who will provide technical oversight to site operations by monitoring process performance, designating changes in process parameters to accommodate different feeds, managing quality assurance program, etc.

Operating staff will be comprised of five (5) shift groups operating on a rotating basis. Each shift group will consist of one (1) foreman and three (3) operators. Operators will be responsible for preparation of soils, charging furnaces, recording relevant process data, collection and preparation for analysis of process batch samples, and other tasks associated with day-to-day unit operations. The foreman on each shift will be responsible for direct supervision of operators, interaction with other shift foremen to maintain continuity in processing, and implementation of tasks or assignments set forth by site management.

Electrical Power requirements for the system are estimated at 1.2 megawatts (MW) per furnace module at maximum draw. Average power for the system is expected to be approximately 30% of peak power or 2.16 MW for the six (6) furnace modules operating at the site. This estimate of power consumption is based upon actual consumption experience with a similar system and thermal data obtained from the pilot test program.

Activated Carbon, Chemical Consumption, Nonelectric Utilities, and Personal Protective Equipment estimates are based directly upon commercial experience and results from this pilot test program.

Laboratory Analyses factored into the estimate include analysis for total mercury on a composite sample collected from the four trays from a furnace chamber and a TCLP analysis for mercury on a composite sample representative of each batch from a furnace module (four chambers).

The estimate for Maintenance Parts, is based upon 5% of the total facility cost excluding costs for engineering. Maintenance Labor is based upon 2,000 man-hours per year of various service personnel including electricians, pipefitters, millwrights, and general technicians.

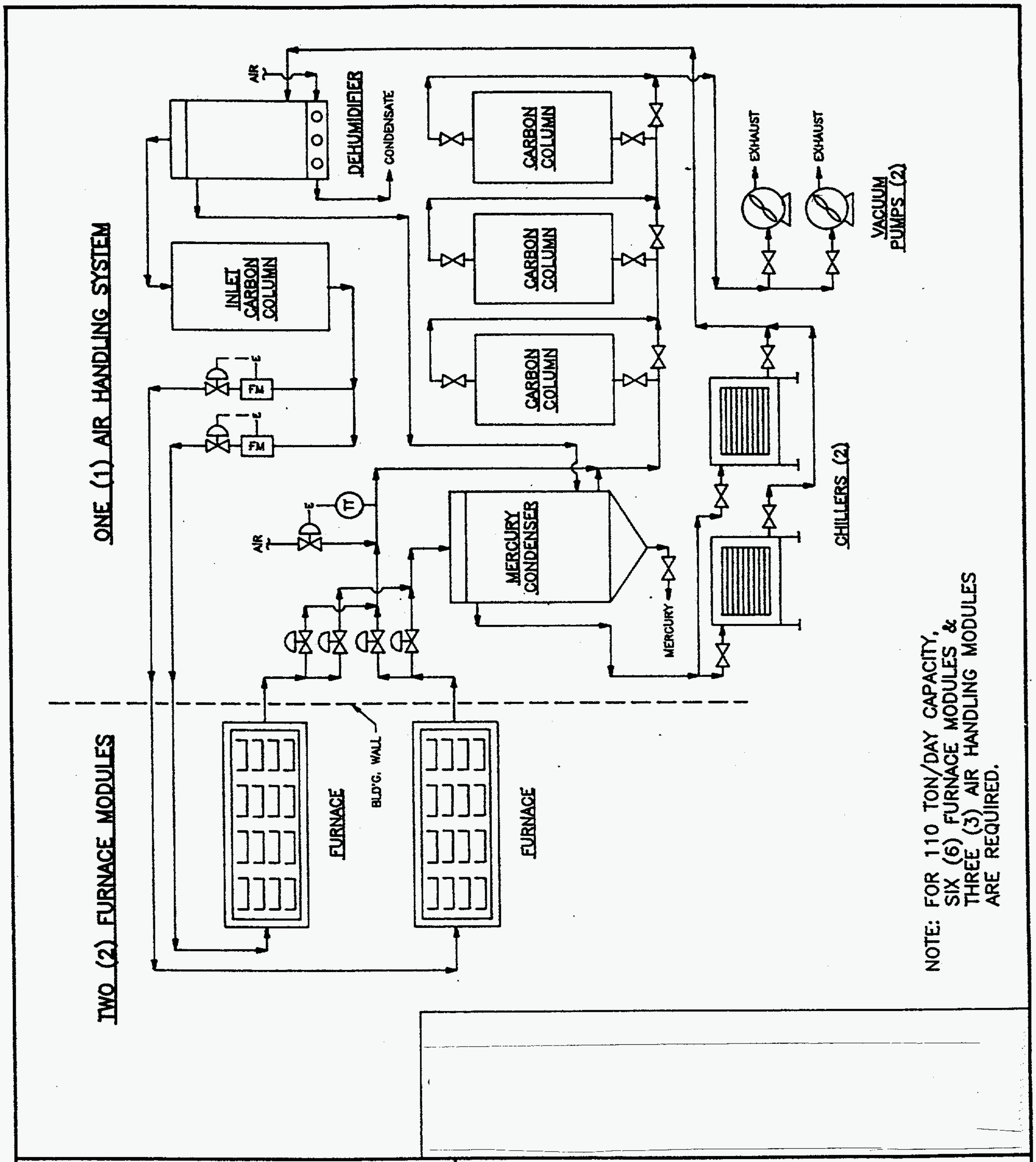
Table 16
Per Ton Operating Cost Estimate
110 Ton/Day Modular System

ITEM	$/TON (US)
Supervision	3
Operating Labor	14
Electric Power Consumption	48
Activated Carbon Consumption	9
Chemical Additive Consumption	2
Laboratory Analysis	15
Nonelectric Utilities	1
Maintenance Parts/ Consumables	12
Maintenance Labor	2
Personnel Protection Consumables	1
Total	107

Note: Operating costs do not include excavation, transportation to and from the recycling facility, and management of processed wastes/soils.

No value is placed on the recovered mercury.

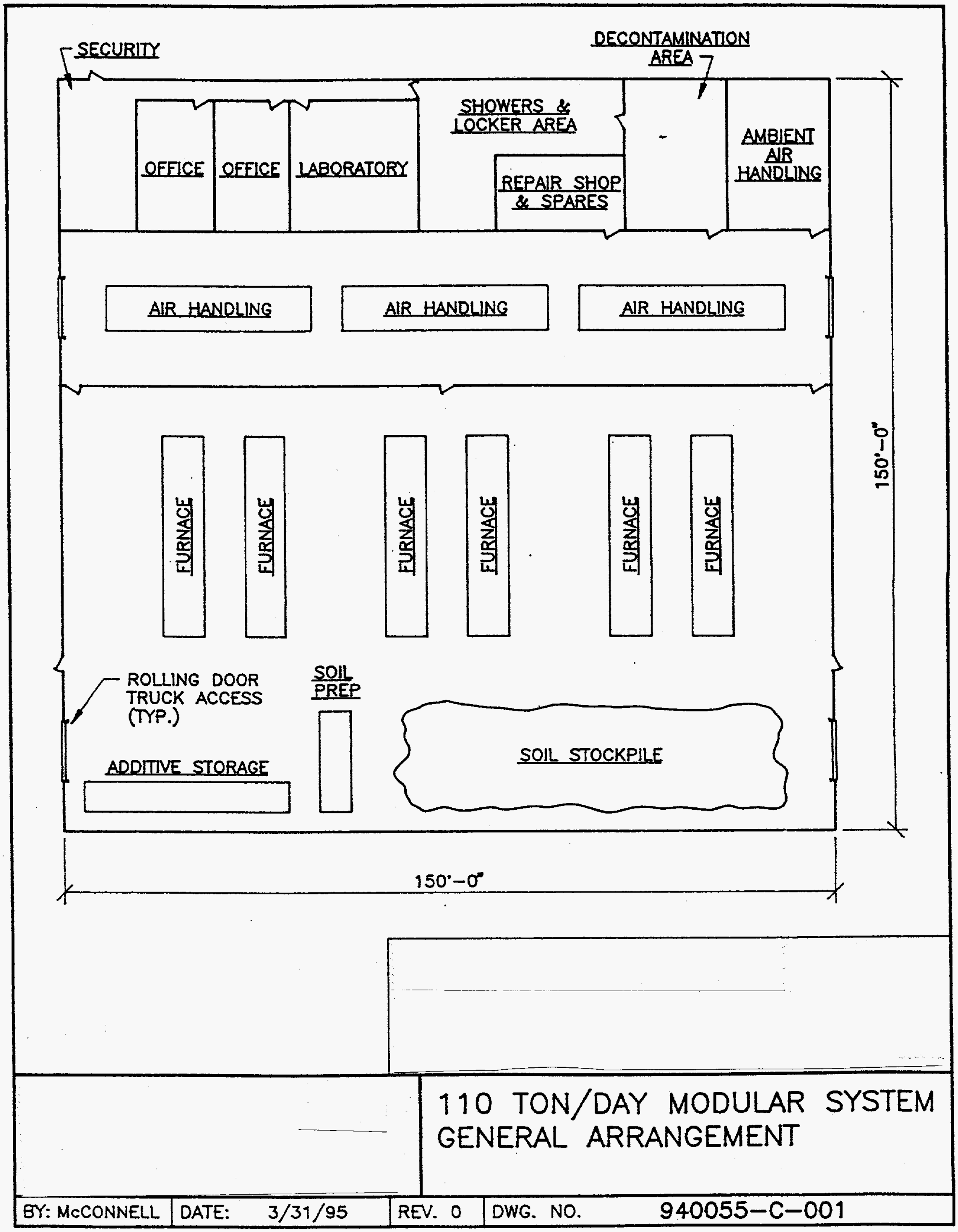
Figure 7



110 TON/DAY MODULAR SYSTEM
PROCESS FLOW DIAGRAM

BY: McCONNELL	DATE: 4/4/95	REV. 0	DWG. NO. 940055-F-001

Figure 8


SECURITY
DECONTAMINATION AREA
SHOWERS & LOCKER AREA
OFFICE
OFFICE
LABORATORY
REPAIR SHOP & SPARES
AMBIENT AIR HANDLING
AIR HANDLING
AIR HANDLING
AIR HANDLING
FURNACE
FURNACE
FURNACE
FURNACE
FURNACE
FURNACE
150'-0"
ROLLING DOOR TRUCK ACCESS (TYP.)
SOIL PREP
ADDITIVE STORAGE
SOIL STOCKPILE
150'-0"
110 TON/DAY MODULAR SYSTEM GENERAL ARRANGEMENT
BY: McCONNELL
DATE: 3/31/95
REV. 0
DWG. NO. 940055-C-001

DISCUSSION OF RESULTS

The results presented within this report clearly show that the MRS technology is an effective and efficient means of removing and recovering mercury from mixed wastes. Two of the primary objectives or goals of this project were to demonstrate that the technology was successful in removing and recovering mercury from the base model material and to demonstrate that an effective separation of the radiochemical isotope (or surrogate tracer) from the mercury species was possible.

PRODUCT SOIL ANALYSES

Results from analyses of processed soils show that the mercury content added to the feed soils as mercury and mercury compounds in concentrations ranging from 1,500 mg/kg to 7,000 mg/kg was reduced to less than 1 mg/kg in most tests and less than 2 mg/kg in all tests.

In most instances, but not all, the product analyses followed the pattern that longer times at higher temperatures resulted in lower product mercury contents. The reproducibility of these results is demonstrated quite well between tests 12 and 13 (Table 7D) where processing under the exact same conditions (time and temperature) resulted in nearly identical product mercury contents. Close replication of results was not observed between tests run on soils with and without NORM under nearly the same conditions, however. This is attributed to the more rigorous blending that occurred when adding the NORM to the soils to achieve an even distribution at the low concentration selected for this program. Due to this blending, the process feed was finer in nature than the soils that did not contain NORM. The finer texture affects the heat transfer within the soil bed as well as the diffusion of mercury vapor out of the bed. Despite the slight difference in performance observed, the mercury content of the processed soil was within acceptable limits.

In addition, no pyridine was detected in product soils. This is an important result because pyridine, as regulated, toxic substance, is often produced upon partial decomposition of coal. Due to the high coal content in the feed, there had been some concern that this toxic by-product may appear in the processed soils. These analyses show that this does not occur when processed by the MRS technology.

MERCURY BALANCE

The mass balance for mercury described in Table 13, shows that after being removed from the soil, the mercury was recovered by the process gas handling equipment. The final distribution of mercury within the gas handling system differed from previous demonstrations of the MRS technology where metallic mercury recoveries were significantly higher than the 46.2% reported in Table 13 and generally on the order of 75% to 90% of the total mercury added (9). The difference in metal recovery is attributed to the higher furnace air temperature used in the first part of the heating cycle to dry the soils. Although this approach reduces the overall cycle time, it results in increased vaporization of mercury during the first stage of the process cycle which then reports to the bypass carbon filtration media. The mercury contained in the carbon filtration media will eventually be recovered as metallic product, however, since spent carbon is processed through the same system utilized to process the contaminated waste/soil. Therefore, the difference in recoveries is temporary and only of economic concern as all mercury will eventually be recovered in metallic form.

NORM BALANCE

The mass balance for radioisotopes presented in Table 14 shows that the NORM materials added to the soil prior to testing were retained in the processed soil product, separate from the mercury. Wipe tests collected from the system pipework during the course of this program verified that neither the NORM or the cerium oxide used as a surrogate tracer compound for the majority of the tests were carried over to the process gas handling system over the course of this test program.

These results verify the quality of the separation and appear to place the MRS technology in the forefront of processes to recover mercury from mixed wastes. This assessment is based upon information provided by developers of competitive technologies, particularly those that utilize rotary kilns or similar devices, that acknowledge trace to significant (10% to 30% of feed weight) carryover of dust from the furnace to the gas handling system (10). Unlike these process technologies, the MRS technology does not promote the carryover of dust due to its batch operation and highly controlled gas handling system. Therefore, it is not susceptible to recontamination of the mercury product it generates as a result of contact with radioisotopes carried over with dust into the process gas handling system.

SCALE UP

Based upon all results presented herein, MRS is quite confident that the results achieved in this test program will be reproducible on a commercial-scale system, particularly since MRS has had previous success in successfully scaling results obtained with this pilot scale in the construction and operation of a commercial-scale facility to remove and recover mercury from soils contaminated with mercury. Therefore, there is little doubt regarding the ability to scale the results obtained here to a commercial system capable of meeting the requirements of DOE facilities.

REFERENCES

1. Correspondence: April 12, 1994; Ms. B. Hoditschek, RCRA Permits Program Manager, State of New Mexico Environmental Department, to Mr. W.F. Sutton (MRS).

2. Correspondence: November 29, 1993; Mr. M. Shapiro, Director - Office of Soild Waste, US Environmental Protection Agency, Washington, D.C. to Mr. W.F. Sutton (MRS).

3. Telephone Conversation: December 8, 1994; Mr. A. McArthur (CEI) and Ms. B. R. Heath, Nuclear Regulatory Commission, King of Prussia, Pennsylvania.

4. Correspondence: February 9, 1995; Mr. L. Kuchinski, Chief - Division of Hazardous Waste Management, Pennsylvania Department of Environmental Resources to Mr. W. F. Sutton (MRS).

5. Taylor, Fred G. Jr., "Mercury Assessment for Water and Sediment in Oak Ridge National Laboratory," (ORNL/M-713), 1989.

6. Carmichael, John K., "An Investigation of Shallow Ground-Water Quality Near East Fork Poplar Creek, Oak Ridge, Tennessee," U.S. Geological Survey, Water-Resources Investigations Report 88-4219, 1989.

7. Radian Corporation, Oak Ridge, TN, "DRAFT - Treatability Study Report for Mercury in East Fork Poplar Creek, Oak Ridge, Tennessee," Prepared for U.S. Department of Energy (DE-AC05-90OR21851), 1993.

8. "Surrogate Formulations for Thermal Treatment of Low-Level Mixed Waste, Part III Plasma Hearth Process Testing," Prepared for U.S. Department of Energy (DE-AC05-84OR21400), 1994.

9. Weyand, T. E., Rose, M. V., Koshinski, C. J., Pittsburgh Mineral & Environmental Technology, Inc., "Demonstration of Thermal Treatment for Mercury-Contaminated Soils," Prepared for Gas Research Institute, Chicago, IL, 1994.

10. Broadbent, C.P., Berkenhagen, F.P., Aurich, V.G., Rots, K.J., "High Temperature Oxidation Process for Decontamination of Mercury Containing Wastes", 'Aufbereitungs-Technik 35', (1994), Nr. 6

REFERENCES

1. Correspondence: April 24, 1994; Ms. B. Hoditschek, RCRA Permits Program Manager, State of New Mexico Environmental Department, to Mr. W.F. Sutton (MRS).

2. Correspondence: November 29, 1993; Mr. M. Shapiro, Director - Office of Soild Waste, US Environmental Protection Agency, Washington, D.C. to Mr. W.F. Sutton (MRS).

3. Telephone Conversation: December 8, 1994; Mr. A. McArthur (CEI) and Ms. B. R. Heath, Nuclear Regulatory Commission, King of Prussia, Pennsylvania.

4. Correspondence: February 9, 1995; Mr. L. Kuchinski, Chief - Division of Hazardous Waste Management, Pennsylvania Department of Environmental Resources to Mr. W. F. Sutton (MRS).

5. Taylor, Fred G. Jr., "Mercury Assessment for Water and Sediment in Oak Ridge National Laboratory," (ORNL/M-713), 1989.

6. Carmichael, John K., "An Investigation of Shallow Ground-Water Quality Near East Fork Poplar Creek, Oak Ridge, Tennessee," U.S. Geological Survey, Water-Resources Investigations Report 88-4219, 1989.

7. Radian Corporation, Oak Ridge, TN, "DRAFT - Treatability Study Report for Mercury in East Fork Poplar Creek, Oak Ridge, Tennessee," Prepared for U.S. Department of Energy (DE-AC05-90OR21851), 1993.

8. "Surrogate Formulations for Thermal Treatment of Low-Level Mixed Waste, Part III Plasma Hearth Process Testing," Prepared for U.S. Department of Energy (DE-AC05-84OR21400), 1994.

9. Weyand, T. E., Rose, M. V., Koshinski, C. J., Pittsburgh Mineral & Environmental Technology, Inc., "Demonstration of Thermal Treatment for Mercury-Contaminated Soils," Prepared for Gas Research Institute, Chicago, IL, 1994.

10. Broadbent, C.P., Berkenhagen, F.P., Aurich, V.G., Rots, K.J., "High Temperature Oxidation Process for Decontamination of Mercury Containing Wastes", 'Aufbereitungs-Technik 35', (1994), Nr. 6

Appendix 1

State of New Mexico
ENVIRONMENT DEPARTMENT
Harold Runnels Building
1190 St. Francis Drive, P.O. Box 26110
Santa Fe, New Mexico 87502
(505) 827-2850

BRUCE KING
GOVERNOR

JUDITH M. ESPINOSA
SECRETARY

RON CURRY
DEPUTY SECRETARY

April 12, 1994

Mr. William F. Sutton
President
Mercury Recovery Services
700 Fifth Ave.,
New Brighton, PA 15066

Dear Mr. Sutton:

RE: Mercury Recovery Services planned Mercury Reclamation Activities in the State of New Mexico

The New Mexico Environment Department (NMED) received your letter dated March 30, 1994, in response to our letter dated March 29, 1994, listing five main issues that your company, Mercury Recovery Services (MRS) had to address prior to the commencement of your planned mercury recovery activities in the State of New Mexico.

Your response to the issues raised concerning the operation of the mercury recovery system is satisfactory based on the following conditions as stated in your letter dated March 30, 1994, and in your work plan dated March 2, 1994.

(1) MRS expects the maximum holding period for as-received mercury contaminated soil in a covered roll-off to be "approximately 14 days", which is less than the maximum of 90 days allowable by New Mexico Hazardous Waste Management Regulations (HWMR-7), Part VIII, Section 268.50.

(2) the anticipated concentrations of toxic organic compound constituents and soil heating values are very low, compared to their regulatory thresholds of 500 ppm and 5,000 BTU/lb respectively;

(3) processed or spent carbon will be recycled continuously in and during the mercury recovery process.

NMED feels that if operated according to the information you provided, the mercury recovery process could meet the recycling exemption for recyclable materials, but subject to HWMR-7, Part II, Section 261.6(a)(1), and the acquisition of an EPA identification number in accordance with HWMR-7, Part II, Section 261.12. In addition, MRS must conduct the mercury recovery activities in a manner that protects human health and the environment.

If you have further questions on this matter, you may contact me or Cornelius Amindyas at (505) 827-4308.

Sincerely,

Robert S Dinwiddie

for Barbara Hoditschek, RCRA Permits Program Manager

cc: Benito Garcia, Chief, HRMB
Kathleen M. Sisneros, Director
Water and Waste Management Division
File 94

Appendix 2

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

NOV 29 1993

OFFICE OF
SOLID WASTE AND EMERGENCY
RESPONSE

Mr. William F. Sutton
President
Mercury Recovery Services
700 Fifth Ave.
New Brighton, PA 15066

Dear Mr. Sutton:

In your letter of August 27, 1993 to Sylvia Lowrance, you requested an EPA interpretation of the classification of your mobile mercury retorting process for mercury contaminated soils from natural gas pipeline meters as a recycling process. You also indicate in your letter an interest on the part of natural gas pipeline companies to understand permitting requirements for field operation of your process.

In response to your request, EPA has reviewed the written materials that you have submitted with your letter and during your meeting with EPA Headquarters on August 25, 1993. EPA recognizes that mercury roasting and retorting are two methods of reclamation, a type of recycling (40 CFR §261.1(c)(7)).

Under Federal RCRA Subtitle C regulations, recycling processes are generally not subject to regulation including permitting requirements (40 CFR §261.6(c)(1)). However, recycling in industrial furnaces or boilers represents an exception to this general rule (see 40 CFR Part 266 Subpart H generally). In particular, roasting and retorting operations meet the definition of an industrial furnace (40 CFR §260.10) since they are types of smelting, melting or refining furnaces.[1] However, if the retorting operation is burning solely for metal recovery, it is conditionally exempt from most of the boiler and industrial furnace (BIF) requirements including the requirement to obtain a permit (40 CFR §266.100(c)).

---

[1] Roasting involves the heating of a material such as an ore or metal compound in order to remove impurities such as sulfides from metal compounds such as mercury sulfides. Roasters are a type of smelting, melting and refining furnace listed in 40 CFR 260.10. A retorter is a furnace where metal compounds are refined from a metal oxide to metal form through distillation and condensation. EPA believes that retorters are also a type of pyrometallurgical device that meets the definition of smelting, melting or refinning furnace even through they are not specifically listed in 40 CFR 260.10 as an example of such devices.

To meet the conditions of the exemption, the owner/operator of a smelting, melting or refining device must submit a one-time written notice to the Director stating that: 1) the owner/operator claims the exemption, 2) the hazardous waste is being burned solely for metal recovery and contains recoverable levels of metals, and 3) the owner/operator will comply with sampling, analysis and recordkeeping requirements of 40 CFR § 266.100 (c)(1). (The owner/operator must also meet the management standards prior to burning (40 CFR 266.101) and the regulation of residues (40 CFR 266.112).)

In order to be considered burning solely for metal recovery, an owner/operator of an industrial furnace (e.g., smelting, melting or refining furnace) must meet two conditions. First, hazardous waste burned in the furnace, as fired, must have no more than a total of 500 ppm of organic compounds listed in 40 CFR Part 261 Appendix VIII. Second, the hazardous waste must have heating value of less than 5000 BTU/lb. 40 CFR §266.100(c)(2).

Note that if the hazardous waste as fired in the furnace exceeds a total of 500 ppm Appendix VIII organic constituents by weight, it is considered burning for destruction and therefore cannot qualify for the metal recovery exemption under the BIF rule. Alternatively, if the waste is greater than 5000 BTU/lb heating value, it is considered to be burned as a fuel. In either event, the industrial furnace would be subject to 40 CFR Part 266 Subpart H BIF requirements, including permit requirements. The hazardous waste to be burned in the furnace may be treated either below 500 ppm Appendix VIII organic constituents or less than 5000 BTU/lb by *bona fide treatment* that removes or destroys organic constituents. If this type of partial treatment were to occur prior to entry of the waste into an industrial furnace, then the industrial furnace could still be eligible for the metal recovery exemption under the BIF rule. However, blending to dilute below either of these levels is prohibited. 40 CFR §266.100(c)(2).

Based on information provided in your incoming, it appears that your operation is a roasting/retorting operation that may be exempt from RCRA permitting requirements. However, this regulatory determination will depend on situation specific factors such as the type of material fed into the retorting unit. Therefore, you should consult with the appropriate EPA Region or State for particular regulatory determinations regarding specific sites.

Please be aware that under Section 3006 of RCRA (42 U.S.C. Section 6926) individual States can be authorized to administer and enforce their own hazardous waste programs in lieu of the Federal program. When States are not authorized to administer their own program, the appropriate EPA Regional office administers the program and is the appropriate contact for any case-specific determinations. Please also note that under Section 3009 of RCRA (42 U.S.C. Section 6929) States retain authority to promulgate regulatory requirements that are more stringent than Federal regulatory requirements.

If you have any additional questions regarding this matter, please contact Mike Petruska of my staff at (202) 260-8551.

Sincerely,

Michael Shapiro, Director
Office of Solid Waste

cc: Waste Combustion Permit Writers Workgroup

Appendix 3

TABLE 1

Typical Table of Contents for Project Work Plan

		Page
INTRODUCTION		
PROJECT DATA	Section 1	
Location and Access		1-1
Project Personnel		1-2
Emergency Data		1-3
FACILITY DESCRIPTION	Section 2	
Site		2-1
Security		2-2
Material Staging Area		2-3
Process System		2-4
Air Monitoring		2-5
Area Plan		2-6
Process Flow Diagram		2-7
OPERATING PROCEDURES	Section 3	
Processing		3-1
Carbon		3-2
Mercury Removal from Condenser		3-3
Instrumentation		3-4
Record Keeping - Operations		3-5
Site Assessment & Decontamination		3-6
Spill Prevention Plan		3-7
SAMPLING PROCEDURES	Section 4	
Incoming Soil		4-1
Processed Soil		4-2
Sampling Diagram		4-4
Decontamination of Sampling Equipment		4-6
Sample Containers and Container Labels		4-7
Record Keeping and Chain of Custody		4-8
Report of Laboratory Analyses 4-9		
EQUIPMENT LIST	Section 5	5-1
EXHIBIT D HEALTH AND SAFETY	Section 6	

In addition to the review and development of individual Project Work Plans and associated Quality Assurance elements, the MRS QA Officer will call a meeting annually consisting of the President, VP Engineering, and Manager of Operations to review and update the Quality Assurance Program. The results of this review will be documented in meeting notes, as well as the revision --if required--of the general Quality Plan.

2.1 PURPOSE

The purpose of this section is to ensure that programmatic guidance for planning, implementation, and maintenance of the Quality Assurance Program, hereafter referred to as the "QA Program" or "Program", for Mercury Recovery Services is provided.

2.2 SCOPE

The focus of MRS business areas is limited to the recycle/recovery of mercury from materials which may be mixed wastes, soils, sludges, and etc., all of which contain recoverable quantities of mercury.

2.3 DEFINITIONS

Activity - A unit of work performed to meet a requirement either internal or external, regulatory or contractual.

Controlled Conditions - Use of appropriate equipment, suitable environment, and suitable environmental conditions, for accomplishing an activity and assuring the prerequisites for activity have been satisfied.

Qualification - The characteristics or abilities gained through education, training or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

Verification (Verify) - The act of reviewing, inspecting, testing, checking, auditing, or otherwise determining and documenting whether items, processes, services, or documents conform to specified requirements.

2.4 PROGRAM ELEMENTS

The MRS QA Program contains many elements. The specific considerations made in development of the Program are documented in this section.

2.4.1 Technical Considerations

Controls shall be established such that, sufficient consideration of the technical aspects which affect quality are maintained.

- Technical documents, procedures, test sequence procedures, procurements of special and control items or equipment and responses to Corrective Action Reports (CAR's), shall be developed by qualified Technical Personnel.

- Technical Personnel assigned to develop such items shall have achieved appropriate qualification and/or certification consistent with the technical disciplines(s) represented.

- Technical Personnel shall be as assigned by Line Management and each element shall be reviewed for consistency and must be approved by members of the Line and verified by a member of the QA Organization.

2.4.2 Control of Activities Affecting Quality

Controls of activities affecting quality shall be consistent with their importance.

- Corporate level documents affecting Health and Safety or Quality Assurance planning or procedures shall be under the direct control of the President or his designated alternate.

- Control of other activities undertaken by the corporation which could affect quality shall be under the direct control of a Senior member of Line Management designated by the President.

- Local Office documents which affect Health and Safety or Quality Assurance shall require the approval of the President or a designated alternate.

NOTE

Designations shall be identified in writing by the President. Corporate memorandum may be used to make such designation, however, distribution shall include Corporate Officers, the QA Manger, the Senior Office Management Representative in each office, and the local QA representative for each office.

- Control of other activities undertaken by the local offices which could affect quality shall be under the direct control of the Senior Office

Management Representative in each office. These activities shall be verified for quality considerations by the Local QA Representative.

Any deviations in quality practices shall be resolved locally or in accordance with Section 9.0, "Corrective Actions", of this Document. Any resolution shall be documented to the QA Manager.

NOTE

It is intended that each office develop a Local QA Plan and Procedures which meet the requirements of the Corporate QA Plan presented in this document.

- Control of activities on client jobsites shall be controlled either contractually through use of client QA Plans and Procedures or through development of QA Procedures specific to the job location.

  The Senior Management Representative on the jobsite shall assume the Line Management responsibility for the jobsite. Typically, the position would be designated as Project Manager or Job Superintendent. Any specific responsibilities beyond those found in this QA Plan shall be documented in contract documents for the job or job specific QA Plan or job specific QA Procedures.

  Unless documented in contractual documents, job specific QA Plans or job specific QA Procedures the local QA Representative, consistent with the office operation managing the job, shall maintain the responsibility for the QA functions associated with all activities at client jobsite(s).

2.4.3 Planning and Accomplishment of Activities

Planning and accomplishment of activities which affect quality shall be performed.

- It shall be the standard practice of each MRS office to develop detailed plans and schedules for each client job. These plans and schedules shall, typically, be developed using format identified in client bid request documents.

In the event such guidance is not presented the following elements shall be met as a minimum:

1. A listing of each major task or element of the job shall be developed. This listing may be contained in the body of the document.

NOTE

A detailed Table of Contents may be developed to meet this requirement.

2. Critical Path Management (CPM) style scheduling is the method preferred by the corporation. Any/all schedules developed must include estimated duration of each task, sub-task or element of all activities required to complete the entire job. These schedules shall indicate appropriate QA verification, as required.

- The QA Manager shall develop an annual QA Audit/Surveillance Plan for approval by the President, in accordance with Section 1.0, "Organization and Responsibility"; Part 1.4.2, "Quality Assurance Manger".

  The QA Audit/Surveillance Plan shall include a schedule for all planned Audits and Surveillances, in accordance with Section 11.0, "Audits/Surveillances".

- Any major activity undertaken by the corporation which will or might affect overall quality or specific quality functions shall be planned and reviewed for quality impact prior to implementation. Revisions to the QA Plan shall be made as required to maintain quality performance levels.

  These activities include addition of new business areas such as, manufacturing, purchase/resale of equipment or components, undertaking of major scope new construction projects and major, hard skills, engineering.

2.4.4 General Provisions for Special Controls

Provisions shall be made for any specials controls, processes, test equipment, tools, and skills to attain the required quality and for the verification of quality.

- Special controls, in the form of documents, verifications, and/or procedures, shall be established for processes, test equipment, tools, and skills consistent with Client QA Requirements or Sub-Contractor QA Plans and Procedures, if adequate and appropriate,. In such cases where requirements do not exist the provisions of this QA Plan shall be in effect.

  The specific requirements are documented in Section 5.0, "Processes"; Section 7.0, "Test Control"; and Section 3.0, "Training and Qualification".

- In the cases of work controlled by federal or state regulators specific procedures incorporating the regulatory requirements shall be developed to control all processes. Specifically this applies to decontamination (both radiological and chemical), environmental restoration/remediation and sampling (federal and state EPA and OSHA), and radiological hazards (NRC).

2.4.5 General Provisions for Training

Provisions shall be made for indoctrination and training, as required, of personnel performing activities to assure that suitable proficiency is achieved and maintained.

- It is the general policy of MRS to assist all employees in maintaining qualification. The specific responsibilities for scheduling training and achieving and maintaining qualifications is documented in Section 3.0, "Training and Qualification".

2.4.6 General Provisions for Program Reviews

Provisions shall be made to ensure that management of those organizations implementing the program regularly assess the adequacy of that part of the program for which they hold responsibility and assure its effective implementation.

- The President shall review the QA Program on an annual basis. This review may be accomplished through the approval of the annual QA report generated by the QA Manager, in accordance with Section 1.090, "Organization and Responsibilities", part 1.5.2, "Quality Assurance Manager".

- The QA Manager shall review the overall program annually.

- The QA Manager shall conduct a review of specific program elements on a periodic basis. The periodicity of this review shall not exceed quarterly.

- The local QA Representative shall review local QA Program elements routinely. This review shall be documented in writing the QA Manager on a monthly basis or as needed to assure proper implementation of the Quality Program.

- The Senior Office Management Representative shall review Quality issues and Program implementation with the local QA Representative monthly or as needed to assure proper implementation of the Quality Program.

2.5 PROGRAM ORGANIZATION

The QA Program is designed in three (3) major elements. These elements are: management, performance, and assessment. In any root cause analysis of a quality issue, concern or finding the major element(s) which directly affected or facilitated the problem should be identified.

- **Management** - Line and Quality Management hold primary responsibility for the major program components of a quality program, personnel training and qualification, documents and document control and Quality Assurance records.

  Corporate Line and QA Management shall maintain the responsibility for establishing and maintaining the identified quality components such that all quality concerns are addressed. In the event issues arise in these areas the principle topic to be addressed in any corrective actin must include the assessment of management problems or systems which created or failed to identify and correct the problem(s) which generated the quality issue.

- **Performance** - Corporate Line at the Local Office Level and designated Site Management hold primary responsibility for the major program components of processes, inspection, test control, procurement, and corrective actions.
  This component directly measures work practice on the Local Level. Local Line Management holds primary responsibility for issues related to the items listed.

- **Assessment** - QA Management holds the primary responsibility for Audits/Surveillances and collateral responsibility for Corrective Actions.

  It is the responsibility of the QA Organization to support Line Management goals while ensuring quality is maintained throughout the organization.

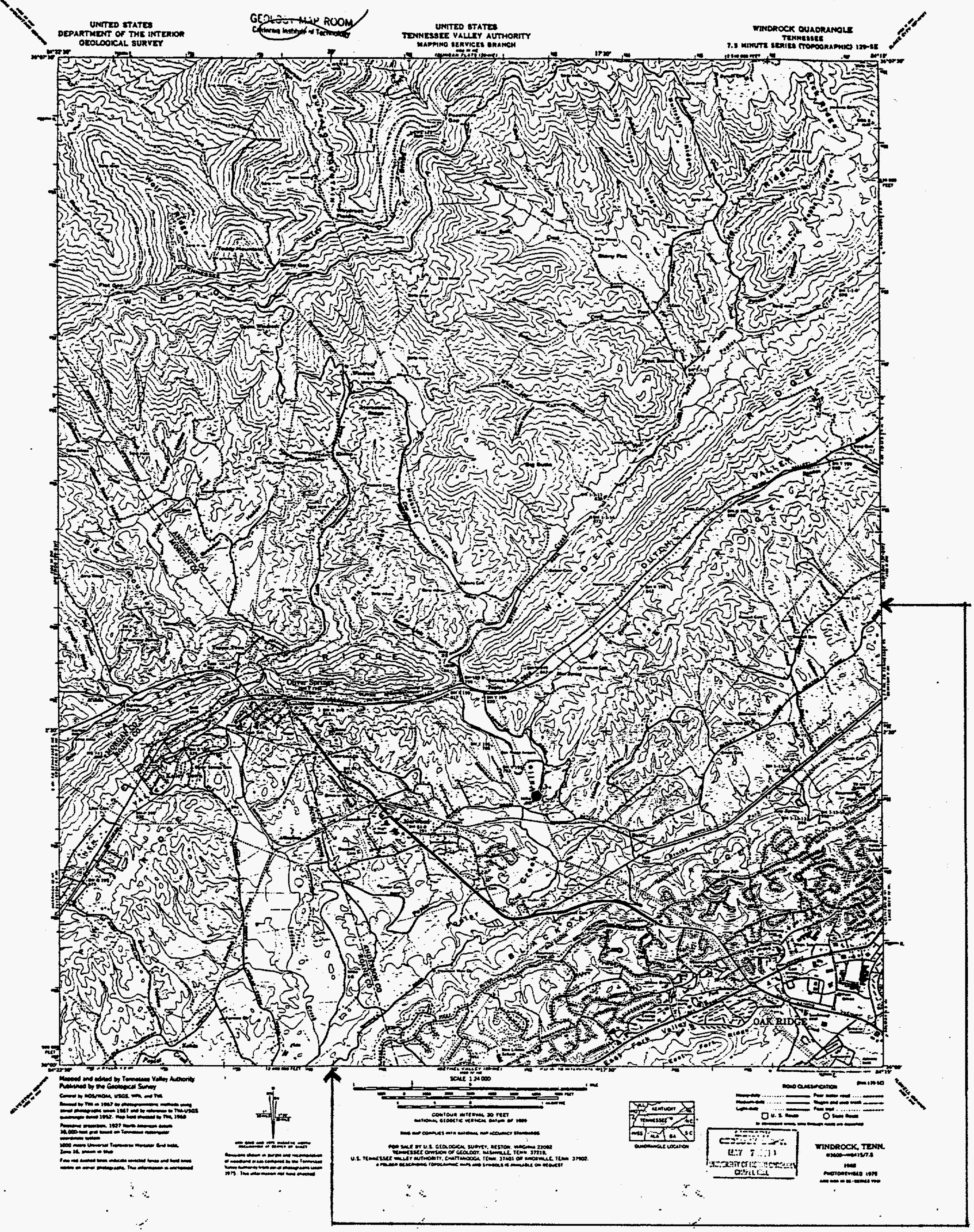
2.6 REFERENCES

ASME/ANSI - NQA-1 (1989) - "Quality Assurance Program Requirements for Nuclear Facilities".

DOE Order 5700.6C (8/21/91) - "Quality Assurance".

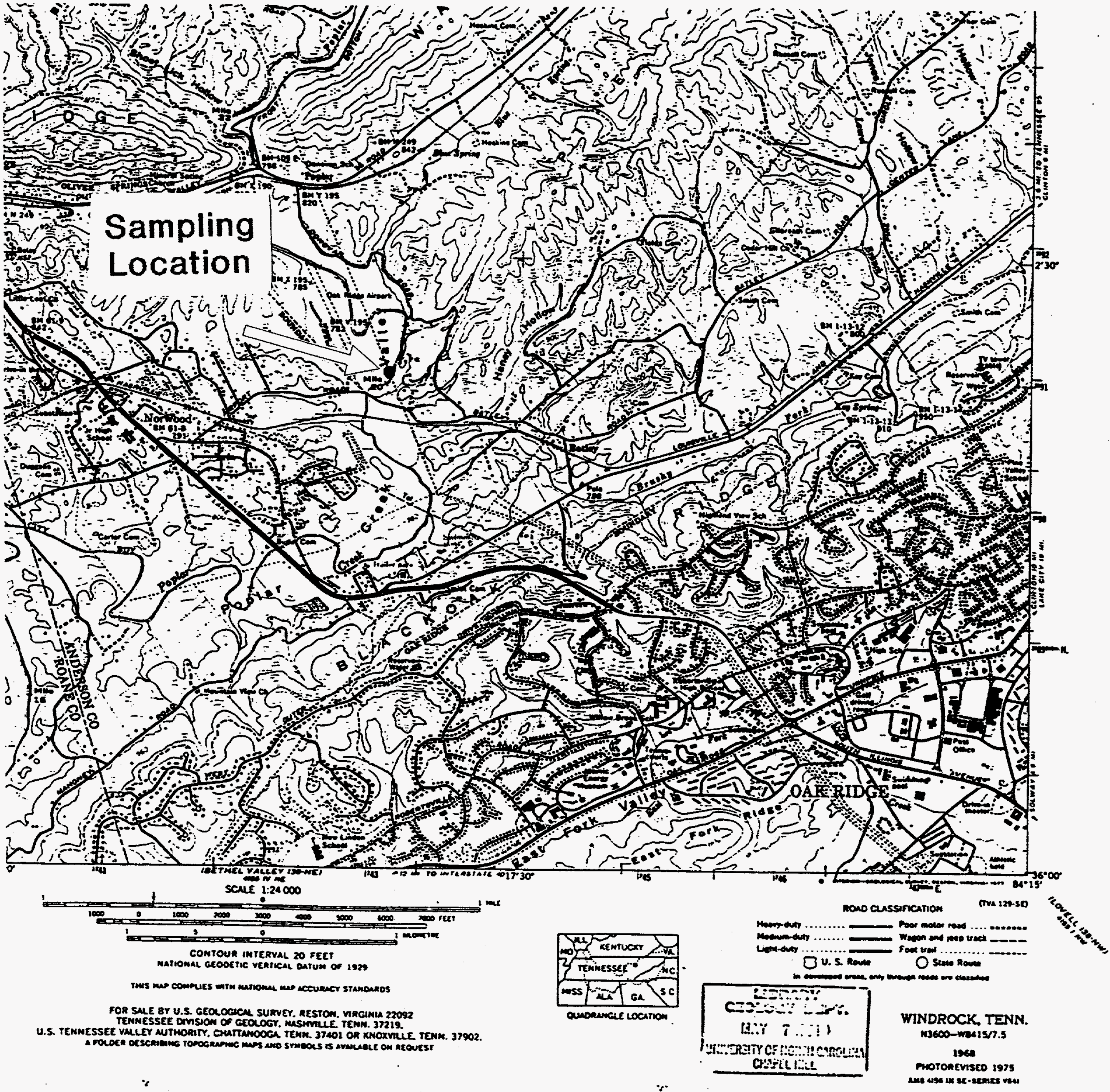
10CFR50 Appendix B - "Quality Assurance Criteria for Nuclear Power Plants and Fuel Reprocessing Plants".

Appendix 4



See Expanded View.

Expanded View of Windrock Quadrangle



Appendix 5

```
EG&G ORTEC OMNIGAM ( 191)          13.02.15  17-JAN-95 10:09:15   Page     1
                                   Spectrum name: TC617.SPC

Sample description
      COMPOSITE OF RAM  For MRS/DOE/METC Project      NORM

Spectrum Filename: TC617.SPC

Acquisition information
      Start time             16-JAN-95   15:39:00
      Live time                    3647
      Real time                    3773
      Dead time                   3.33%
      Detector/Geometry IDs          0 &          0

Detector system
      GEM 25185 SN: 29-TP20276B 3500 VOLTS. POS. BIAS
      671 AMP - 6US GAUSSIAN SHAPING - 919 MCB WITH 4K CONV. GAIN

Calibration
      Filename: TC618.CLB
      Created: 17-JAN-95 09:37:41 &
      QCY.44 ENERGY CALIBRATION FOR JANUARY 1995


      Zero offset       .293 keV;  Gain        .248 keV/channel

Library Files
      Main analysis library:            NATURALR.LIB

Analysis parameters
      Start channel               50 for an energy of    12.71keV
      Stop channel              8030 for an energy of  1995.23keV
      Peak rejection level        50.000%
      Activity scaling factor    1.0000E+00/  3.0000E+01 =  3.3333E-02
      Detection limit method:
            MDA - EG&G ORTEC method
      Additional random error:              0.0000000E+00
      Additional systematic error:          0.0000000E+00
      Background width: best method (based on spectrum).

Corrections                               Status          Comments
      Decay correct to date                YES        16-JAN-95 12:00:00
      Decay during acquisition             YES
      Peaked background correction         YES        001003.PBC
                                                      12-AUG-91 07:16:11
      Absorption (Internal)                NO
      Geometry correction                  NO
      Random summing                       NO

Energy calibration normalized difference:      .0808
The energy calibration was changed to fit the spectrum.
```

```
 EG&G ORTEC OMNIGAM ( 191)           13.02.15  17-JAN-95 10:09:15   Page    7
                                     Spectrum name: TC617.SPC

TL-208    3.10982E+00       583.19  3.1098E+00  ( P 1.5107E+00
                            510.77  3.0960E+01 +  P 4.2356E+00
                            860.56  0.0000E+00 ?  P 1.2304E+01
                            277.36  0.0000E+00 %  P 1.4047E+01
                            763.13  0.0000E+00 ?  P 4.3328E+01

K-40      5.86511E+01      1460.83  5.8651E+01  (    1.8325E+01
   ( - This peak used in the nuclide activity average.

   * - Peak is too wide, but only one peak in library.
   ! - Peak is part of a multiplet and this area went
       negative during deconvolution.
   ? - Peak is too narrow.
   @ - Peak is too wide at FW25M, but ok at FWHM.
   % - Peak fails sensitivity test.
   $ - Peak identified, but first peak of this nuclide
       failed one or more qualification tests.
   + - Peak activity higher than counting uncertainty range.
   - - Peak activity lower than counting uncertainty range.
   = - Peak outside analysis energy range.
   & - Calculated peak centroid is not close enough to the
       library energy centroid for positive identification.
   P - Peakbackground subtraction

*****   S U M M A R Y   O F   N U C L I D E S   I N   S A M P L E   *****
           TIME OF COUNT  TIME CORRECTED  UNCERTAINTY  2 SIGMA
NUCLIDE       ACTIVITY       ACTIVITY       COUNTING
            BECQUERELS     BECQUERELS
+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+-+
TH-234   <        5.32E-01      5.32E-01
PA-234   <        1.19E-01      1.19E-01
TH-230   <        6.08E+00      6.08E+00
RA-226          5.3073E+01    5.3073E+01          2.86%
PB-214          5.3839E+01    5.3839E+01           .69%
BI-214          4.8928E+01    4.8928E+01           .84%
PB-210   <        1.08E+00      1.08E+00
TH-231   <        3.17E-01      3.17E-01
PA-231   <        5.39E-01      5.39E-01
TH-227   <        7.31E-01      7.31E-01
RA-223   <        2.52E-01      2.52E-01
RN-219   <        2.82E-01      2.82E-01
AC-228          3.4112E-01    3.4112E-01         60.76%
TH-228   <        2.49E+00      2.49E+00
RA-224   <        1.87E+00      1.87E+00
PB-212          4.0330E-01    4.0330E-01         44.22%
BI-212   <        7.25E-01      7.25E-01
TL-208          1.0366E-01    1.0366E-01         49.20%
K-40            1.9550E+00    1.9550E+00         60.38%

--------------------------------  S U M M A R Y  --------------------------------
TOTAL ACTIVITY (    12.7 to   1995.2 keV) 1.5864260E+02     BECQUERELS

Analysis time  277.6 seconds.
------------------------------
```

Appendix 6

Reagent Specifications

MERCURY METAL:	Aldrich Chemical Company, triple distilled, 99.99+%, #7439-97-6
MERCURY OXIDE (HgO):	Aldrich Chemical Company, red, 99%, #21908-53-2
MERCURY SULFIDE (HgS):	Aldrich Chemical Company, 99%, #1344-48-5
MERCURY CHLORIDE ($HgCl_2$):	Aldrich Chemical Company, 99%, #7487-94-7
CERIUM OXIDE (CeO_2):	Aldrich Chemical Company, 99.9%, #1306-38-3
LEAD OXIDE (PbO):	Aldrich Chemical Company, 99.9+%, #1317-36-8
CADMIUM OXIDE (CdO):	Aldrich Chemical Company, 99.5%, #1306-19-0