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Evaluation of the Graphite Electrode DC Arc Furnace for the Treatment of INEL Buried Wastes

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FOR THE TREATMENT OF INEL BURIED WASTES

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MASTER

SUMMARY

Across the U.S. Department of Energy (DOE) complex, the past practices of DOE and its predecessor agencies in burying radioactive and hazardous wastes have left DOE with the responsibility of remediating large volumes of buried wastes and contaminated soils. The Buried Waste Integrated Demonstration (BWID), a program within DOE's Office of Technology Development, has chosen to evaluate treatment of buried wastes at the Subsurface Disposal Area (SDA) of the Radioactive Waste Management Complex (RWMC) at the Idaho National Engineering Laboratory (INEL). Many scenarios are being evaluated for the treatment of buried wastes. Because of the characteristics of the buried wastes within INEL's SDA, the potential for using high-temperature thermal treatment technologies is being evaluated.

The soil-waste mixture at INEL, when melted or vitrified, produces a glass/ceramic referred to as iron-enriched basalt (IEB). The only waste form presently approved for the disposal of long-lived radionuclides is borosilicate glass (BSG). Studies have determined that large additions of additives would be required to adjust the INEL waste composition to produce a BSG form, which would lead to a substantial waste volume increase. Therefore, it would be advantageous if the INEL waste could be processed into an IEB waste form. One potential problem with producing the IEB material is the high melting temperature of the waste and soil (1400-1600°C). One technology that has demonstrated capabilities to process high melting point materials is the plasma arc heated furnace. Technology based on the graphite electrode DC arc furnace is particularly attractive because it could provide the advantages of simplicity, high availability, safety, a high processing rate, and versatility.

The Pacific Northwest Laboratory (PNL), Massachusetts Institute of Technology (MIT), and Electro-Pyrolysis, Inc. (EPI) have entered into a collaborative "National Laboratory-University-Industrial" three-party program to develop and demonstrate the technology using a graphite electrode DC arc furnace for the treatment of buried wastes. The program, which is funded through

BWID, has involved testing an engineering-scale DC arc furnace to gain preliminary operational and waste processability information. It has also included the design, fabrication, and evaluation of a second-generation, pilot-scale graphite electrode DC arc furnace.

The program was initiated in 1992, and 13 test melts were conducted at the MIT Plasma Fusion Center in the latter part of FY92 using EPI's engineering-scale (Mark I) furnace.

Widely ranging simulants of INEL buried waste were prepared and processed in the Mark I furnace. The tests included melting of soils with metals, sludges, combustibles, and simulated drums. Very promising results in terms of waste product quality, volume reduction, heating efficiency, and operational reliability and versatility were obtained. The results indicate that the graphite electrode DC arc technology would be very well suited for treating high melting point wastes such as those found at the INEL SDA. The graphite electrode DC arc furnace has been demonstrated to be very simple, yet effective, with excellent prospects for remote or semi-remote operation.

CONTENTS

SUMMARY	iii
1.0 INTRODUCTION	1.1
1.1 BACKGROUND	1.1
1.2 TECHNOLOGY DESCRIPTION	1.2
1.3 OBJECTIVE OF THIS WORK	1.3
2.0 EXPERIMENTAL PROGRAM USING THE MARK I FURNACE	2.1
2.1 DESCRIPTION OF MARK I EQUIPMENT	2.1
2.1.1 Furnace Description	2.1
2.1.2 Off-Gas System	2.7
2.1.3 DC Power Supply	2.9
2.1.4 N ₂ Purge System	2.11
2.1.5 Instrumentation	2.11
2.2 DEVELOPMENT OF THE INEL WASTE SIMULANT	2.14
2.2.1 INEL Soil	2.15
2.2.2 INEL Plus Metals	2.16
2.2.3 INEL Soil Plus Combustibles	2.16
2.2.4 INEL Soil Plus RFP 74 Series Sludges	2.17
2.2.5 INEL Soil Plus High Vapor Pressure Metals	2.18
2.3 MARK I EXPERIMENTAL METHCDOLOGY	2.18
3.0 RESULTS AND DISCUSSION	3.1
3.1 INEL SOIL	3.2
3.2 SOIL AND METALS	3.7
3.3 SOIL AND COMBUSTIBLES	3.12
3.4 SOIL AND RFP SLUDGES	3.16

3.5	INEL SOIL AND VOLATILE METALS	3.19
3.5.1	Off-Gas Sampling and Analysis	3.20
3.5.2	Sampling Methodology	3.21
3.5.3	Off-Gas Sampling Setup	3.22
3.5.4	Sampling Operation	3.22
3.5.5	Summary of Off-Gas Analysis	3.26
4.0	PERFORMANCE OF THE MARK I DC ARC FURNACE RELATIVE TO DESIGN AND FABRICATION OF MARK II FURNACE	4.1
5.0	CONCLUSIONS AND RECOMMENDATIONS	5.1
6.0	REFERENCES	6.1

FIGURES

2.1	Plan View of Mark I Installation	2.2
2.2	Elevation View of Mark I Installation	2.2
2.3	Schematic of Mark I Furnace	2.3
2.4	Photograph of Mark I Furnace	2.4
2.5	Thermocouple Locations on Mark I Furnace	2.6
2.6	Off-Gas Line Knockout Box	2.7
2.7	Venturi Scrubber	2.8
2.8	Mark I Power Supply Schematic	2.10
2.9	Nitrogen Purge System for Mark I Furnace	2.11
2.10	Mark I Instrumentation Diagram	2.12
3.1	Mark I Hearth Loaded with INEL Soil - Run 1	3.3
3.2	Vitrified INEL Soil	3.6
3.3	Photomicrograph of High Carbon Area in Vitrified INEL Soil	3.7
3.4	Metal Phase from High Metals Content Run	3.8
3.5	Glass/Slag Phase from High Metals Content Run	3.9
3.6	Glass Produced in Run 8	3.13
3.7	Slag Layer from Ash Run	3.14
3.8	Loading of Simulated RFP 743 Series Sludge Drums in Mark I Furnace Hearth	3.17
3.9	RFP 743 Series Sludge Containers with INEL Soil Added to Hearth	3.18
3.10	Partially Melted Slag and 743 Series Sludge Containers	3.20
3.11	Schematic Off-Gas Sampling System	3.22
3.12	Off-Gas Sampler Location	3.23
3.13	Photograph of Off-Gas Filters from Runs 12 and 13	3.27

3.14 Photomicrograph of Off-Gas Particulate from Run 13	3.28
3.15 Photomicrograph of Off-Gas Particulate from Run 13	3.28
3.16 Photomicrograph of Off-Gas Particulate from Run 13	3.29

TABLES

2.1	Composition of INEL Soil	2.16
2.2	RFP 74 Series Sludge Description	2.17
3.1	Waste Simulant Categorization	3.1
3.2	General Overall Material Balances for 13 Mark I Tests	3.2
3.3	Compositional Phase Analysis of Mark I Runs (Runs 1-3, 12, 13)	3.5
3.4	Compositional Phase Analysis of Mark I Runs 6 and 7	3.10
3.5	Redox Values for Mark I Runs	3.12
3.6	Compositional Phase Analysis of Ash Runs	3.15
3.7	RFP 74 Series Sludge	3.16
3.8	Compositional Phases Analysis of RFP 74 Series Sludge Runs	3.19
3.9	Off-Gas Particulate Composition for Run #12	3.24
3.10	Volatilized Material (Mass Percent) for Run #12	3.25
3.11	Off-Gas Particulate Composition for Run #13	3.25
3.12	Volatilized Material (Mass Percent) for Run #13	3.26

1.0 INTRODUCTION

The Pacific Northwest Laboratory (PNL),^(a) Massachusetts Institute of Technology (MIT), and Electro-Pyrolysis, Inc. (EPI) have entered into a collaborative "National Laboratory-University-Industrial" three-party program to develop and demonstrate the graphite electrode DC arc furnace technology for the treatment of buried wastes. The program, which is funded through the U.S. Department of Energy's (DOE) Buried Waste Integrated Demonstration (BWID), has involved tests of an engineering-scale DC arc furnace to gain preliminary information on operations and waste processability. The work described in this document involved the evaluation and demonstration of this technology at the engineering scale.

The program was initiated in 1992 and 13 test melts were conducted at the MIT Plasma Fusion Center in the latter part of FY92 using EPI's engineering-scale (Mark I) furnace. Widely ranging simulants of INEL buried waste were prepared and processed in the Mark I furnace. The tests included melting of soils with metals, sludges, combustibles, and simulated drums.

1.1 BACKGROUND

The DOE, through its Office of Technology Development (OTD), has initiated a comprehensive program to develop and demonstrate advanced remediation technologies to support DOE's environmental restoration objectives. The BWID has been established within the OTD to develop and demonstrate technologies applicable to the remediation of DOE sites containing buried wastes. Across the DOE complex, the past practices of DOE and its predecessor agencies in burying radioactive and hazardous wastes have left DOE with the responsibility for remediating large volumes of buried wastes and contaminated soils. The BWID program has chosen to evaluate treatment of buried wastes at the Subsurface Disposal Area (SDA) of the Radioactive Waste Management Complex (RWMC) at the Idaho National Engineering Laboratory (INEL). Many scenarios are being

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evaluated for the treatment of buried wastes. Because of the characteristics of the buried wastes within INEL's SDA, the potential for using high-temperature thermal treatment technologies is being evaluated.

The soil-waste mixture at INEL, when melted or vitrified, produces a glass/ceramic referred to as iron-enriched basalt (IEB). The only waste form presently approved for the disposal of long-lived radionuclides is borosilicate glass (BSG). Since studies have determined that large additions of additives would be required to adjust the INEL waste composition to produce a BSG form, which would lead to a substantial waste volume increase, it would be advantageous if the INEL waste could be processed into an IEB waste form. One potential problem with producing the IEB material is the high melting temperature of the waste and soil (1400-1600°C). One technology that has demonstrated capabilities to process high melting point materials is the graphite electrode or plasma arc heated furnace. Technology based on the graphite electrode DC arc furnace is particularly attractive because it could provide the advantages of simplicity, high availability, safety, a high processing rate, and versatility.

1.2 TECHNOLOGY DESCRIPTION

Since the early 1900s, AC-powered graphite electrode arc furnaces have been used for reprocessing scrap metal for the steel industry. Drawing upon steel-making experience and advances in solid-state power conversion equipment, EPI has developed and refined a DC arc furnace for treating waste materials.

The graphite electrode DC electric arc is used to deliver thermal energy into the material to be processed. The ionized gas (plasma) of the arc generally will be maintained between an electrode and the material to be processed (called the "transfer" arc mode). EPI has designed a graphite electrode assembly that allows the arc to be maintained between two concentric electrodes (called the "non-transferred" arc mode). When the electrode is operated in this manner, the energy supplied to the material being melted is the result of thermal energy transfer only and is not as efficient as operation in the transferred arc mode. Although future programmatic activities plan to use

the concentric electrode design, this technology was not used for the tests described in this report. Use of a current-controlled graphite electrode DC arc provides the high-temperature environment required for melting a range of heterogeneous waste forms (temperatures in excess of 1700°C can be produced in the material to be processed). In addition, the high-temperature region around the DC arc will destroy organic species and vapors that evolve from the material being processed. The volume of off-gas produced in the DC arc treatment of solid waste is greatly reduced relative to combustion treatment—typically by more than a factor of 10. Since the volume of gas is substantially reduced, there are fewer entrained particulates in the off-gas, and the size of the off-gas scrubbing equipment can be reduced substantially.

Graphite electrodes offer important advantages for buried waste treatment applications. Graphite electrodes are robust. These electrodes, as used in the simplified design of EPI devices, are well suited to the requirements of remote operation. Graphite electrodes do not have to be water cooled, in contrast to metal electrode plasma torches, thus eliminating any possibility of an accident that could cause pressurized water to contact molten slag or metal. Moreover, short DC arcs, which are highly effective in transferring thermal energy into the material to be processed, can be used. Graphite arc electrodes also permit the use of higher levels of DC arc current, providing the capability to transfer large amounts of the heat into the material to be processed. These factors make possible the high processing rates required for full-scale implementation of this technology at the INEL site.

1.3 OBJECTIVE OF THIS WORK

The primary objective of this work was to determine the suitability of the graphite electrode DC arc process to the treatment of INEL buried wastes. A secondary objective of this work was to obtain operational data for the design, fabrication, and testing of a larger pilot-scale (Mark II) DC arc furnace. The remaining sections of this report describe the engineering-scale work conducted using the EPI's graphite electrode DC arc furnace. Section 2.0 describes the equipment, simulant, and experimental methodology; Section 3.0

discusses the results of FY92 tests; Section 4.0 discusses the test results relative to the Mark II furnace design and fabrication; and Section 5.0 presents the conclusions and recommendations from this work.

2.0 EXPERIMENTAL PROGRAM USING THE MARK I FURNACE

This section describes the Mark I furnace system used in the course of this work. A brief description of the waste simulant development rationale is also presented, followed by a discussion of the methodology used in the test melt program.

2.1 DESCRIPTION OF MARK I EQUIPMENT

The furnace system used here, a Mark I graphite electrode DC arc furnace, consists of the furnace, off-gas handling components, a recirculating water system, a furnace shroud air exhaust system, DC power supply, nitrogen purge system, and instrumentation. These combined subsystems are located on two levels at the south end of the MIT Plasma Fusion Center building in Cambridge, Massachusetts.

This equipment occupies a space of approximately 14 ft x 12 ft with a height of 16 ft, not including the space occupied by MIT's existing power supply.

The furnace shell, exhaust blowers, circulating water pump, recirculating liquid tank, and DC power supply all are located on the bottom floor while the electrode control, scrubber venturi, N₂ bottles, and instrumentation are on the second floor balcony. Figures 2.1 and 2.2 show the plan and elevation views, respectively, of the equipment.

2.1.1 Furnace Description

The furnace consists of a graphite crucible machined from a 16-in.-dia x 24-in.-high solid graphite electrode to a top inside dimension of 13-1/2-in.-dia tapering to 12-1/2-in.-dia at the bottom. The inner height of the crucible is 21 in. This graphite crucible sits on a hot face high alumina brick placed on top of an insulating brick. The insulating brick is on top of a steel plate, and the steel plate is on top of 6 in. of SiO₂ sand. The entire configuration is contained in a square steel box with steel tangs welded to the bottom for connection to the positive terminals of the DC power supply (Figure 2.3 and 2.4).

FIGURE 2.1. Plan View of Mark I Installation

FIGURE 2.2. Elevation View of Mark I Installation

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FIGURE 2.3. Schematic of Mark I Furnace

FIGURE 2.4. Photograph of Mark I Furnace

Hot face brick and insulating brick surround the graphite crucible. Four 1-in.-dia graphite rods connect power through flexible connectors to the graphite crucible.

The original design of this furnace made provision for feed material to enter three ports located 120° apart. This configuration would allow containerized or low-density material to feed into the furnace before the furnace would reach its ultimate volume capacity as the feed material melts. However, because of safety concerns using this mode of operation at the Plasma Fusion Center, it was decided not to operate the furnace in this manner. Since the furnace would be charged only in a batch process, it was modified to place two shields made of high purity alumina over the graphite crucible. This alteration improved the efficiency of the furnace and protected the Duraboard insulation fastened to the roof of the furnace (Figure 2.5).

The inside of the furnace is encased in a rectangular concrete vault, which in turn was lined with 1/2-in.-thick Duraboard sheets made by Carborundum. Finally, a metal shroud was placed around the concrete vault. Four air exhaust ducts connected to blowers allowed air to be exhausted through the opening between the top of the concrete vault and the outside of the furnace roof. The furnace roof is sealed to the concrete vault through concentric silicon "O" rings and RTV sealant that is beaded around the lid. After material is charged into the furnace, the lid is clamped down to the concrete vault and secured with safety cable.

To prevent material from adhering to the graphite crucible after it melted, the following procedure was used when loading the crucible. A thin layer of graphite powder was placed on the bottom of the crucible. Next, a graphite disc was placed on top of this layer. A 1-in.-dia hole was drilled into the middle of the disc, allowing the insertion of variously sized graphite rods. The moveable 2-in.-dia electrode can strike an arc to these rods. A 12-1/2-in.-dia paper cylinder was inserted in the crucible and fine graphite powder was placed between the sides of the crucible and the paper cylinder.

FIGURE 2.5. Thermocouple Locations on Mark I Furnace

Depending on whether the furnace is operated in a "cold topping" or "hot topping" mode, a cardboard tube is placed over the positive graphite electrode. Care is taken when loading the crucible to prevent soil from falling into the cardboard tube. Soil is then carefully loaded around the cardboard tube and inside the paper cylinder.

Once the melt has cooled, the solidified material can be easily removed from the crucible without disturbing the crucible. The material is removed by lowering the 2-in.-dia moveable electrode into the molten mass at the end of a run and then picking up the solidified melt with the electrode attached to it after the melt has solidified.

2.1.2 Off-Gas System

The final addition to the furnace that helps minimize the amount of vaporized material that could clog the off-gas conduits is the gas knockout pot shown in Figure 2.6. The hot vapors condense on the cold sides of the knockout box before they exit through the vertical conduit. An internal baffle directs the gases to turn 180° before they can exit the knockout box.

FIGURE 2.6. Off-Gas Line Knockout Box

The off-gas conduit is made of 3-in. steel pipe. It rises vertically from the furnace and then makes two 90° bends before dropping into the venturi. Two fog nozzles spray the off-gases, and the residence time provided by a sufficient length of conduit following the fog nozzles allows the gases to cool significantly before they enter the venturi.

The venturi scrubber shown in Figure 2.7 can handle a gas inlet rate of up to 42 ACFM, with a removal efficiency of 90-95%. The system was originally designed as a once-through liquid flow system that would simply drain into a sewer pipe. MIT safety restrictions required that a liquid recirculating system be incorporated so the liquid could be analyzed before its disposal. This requirement made it necessary to incorporate a tank with a water-to-water heat exchanger, a water pump, and filters that prevent the fog nozzles and furnace cooling jackets from fouling. To prevent water from backing up and flowing back into the furnace through the off-gas conduit, a bypass water trap was incorporated in case clogging occurred in the venturi system.

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FIGURE 2.7. Venturi Scrubber

After every melt the liquid recirculating system is cleaned, the tank is drained, and the filters are replaced. The off-gases, after being scrubbed by the venturi, are then vented into the atmosphere through a centrifugal blower.

2.1.3 DC Power Supply

MIT has several DC power supplies of adjustable voltage that are permanently located near the Mark I furnace. These power supplies were designed to power a magnet and other loads used in the Plasma Fusion Center building. The one-line diagram shown in Figure 2.8 shows a typical magnet power supply used to power the Mark I DC arc furnace.

One DC power supply is connected to a 1 millihenry DC reactor located next to the power supply. The terminals of the power supply can be connected with either the positive or negative side grounded, or can be operated ungrounded, if desired. For this work it was decided to operate the power supply with the negative electrode at ground potential, allowing the furnace lid and all exposed metal parts to be grounded.

These adjustable voltage DC power supplies are water cooled SCR power supplies. The AC transformers feeding the power supplies have taps that can be connected to give 600V, 480V, 360V, and 240V AC line-to-line voltages. It was decided to operate the arc furnace on the 360V tap, which gives an open circuit DC voltage of approximately 510V DC.

These power supplies have very good current regulators, which can be remotely controlled. A special control module was built and mounted near the electrode hoist and lower winch. The module allows the current to be controlled easily by changing the current reference pot, and the arc voltage to be controlled by use of a hand-operated winch to raise and lower the negative electrode. An analogue voltmeter and DC ammeter were also incorporated into the remote control module. These meters were calibrated against a standard voltmeter and tracked better than 5% accuracy. To further smooth the DC voltage reading, a filter was incorporated into the voltage circuit. The filter allowed a digital DC voltmeter to be connected to the output, providing a reasonably accurate (within +5%) indication of arc voltage.

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FIGURE 2.8. Mark I Power Supply Schematic

2.1.4 N₂ Purge System

Nitrogen gas was used to purge the furnace before energizing the arc, during the run, and after the arc was de-energized (see Figure 2.9). The primary source of purge gas entered through dispersion tubes that surrounded the metal box containing the SiO₂ sand. Nitrogen tanks were located on the control platform and were monitored for gas flow using a floating ball-type flow-meter. Three N₂ tanks were connected in parallel so they could quickly be taken in and out of service as needed.

In addition to the main purge tanks, a single N₂ cylinder fed nitrogen around the moveable electrode bushing, a material loading tube, and the sight port tube.

2.1.5 Instrumentation

Figure 2.10 presents an overall instrumentation diagram for the Mark I furnace. The furnace temperature was monitored with strategically placed thermocouples as shown in Figures 2.5 and 2.6. The thermocouples within the

FIGURE 2.9. Nitrogen Purge System for Mark I Furnace

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FIGURE 2.10. Mark I Instrumentation Diagram

furnace shell were electrically isolated from each other and the metallic furnace shell. These thermocouples, as shown in Figure 2.5, were monitored by a TV camera and recorded on a VCR tape during the run, providing a continuous record of internal temperature readings of the furnace lining. Each thermocouple was connected to a digital voltmeter that was read by the TV camera. In addition to the thermocouples, a digital voltmeter was connected to the positive electrode and ground, which was also monitored by the TV camera.

Since the surface thermocouples shown in Figure 2.6 were connected to electrically-grounded material, they could be brought up to a terminal board on the balcony and were manually recorded approximately every 15 min during a run.

The off-gas furnace pressure was monitored at the top of the vertical gas conduit with a differential pressure gauge. Gas flow was monitored by measuring differential pressure across an orifice inserted in the venturi off-gas conduit. This differential pressure gauge was connected to a manometer that was calibrated against a flow meter in the N_2 gas circuit. Using N_2 as the off-gas inserted directly into the venturi gas conduit, a calibration curve between the manometer and the N_2 flow meter could be obtained.

Water pressure was measured at the output of the water pump and on the output side of the water filters. Previously, a curve of water flow versus water pressure had been obtained from the scrubber manufacturer. Thus, water flow could easily be determined by recording the water pressure.

Electrode movement was measured by placing a meter stick next to a mark on the cable used to raise and lower the electrode. A zero reference point was determined when the arc voltage went to near zero when the negative electrode was lowered to connect it to the positive electrode during the initial start of a run.

Floating ball-type flow meters calibrated in liters per minute were used to monitor the N_2 gas flow into the material-loading tubes, the moveable electrode bushing, and the sight gauge port. The main N_2 purge system was monitored with a floating ball-type flow meter calibrated in CFM.

During the last two runs (#13 and #14), an MM5 sampling train was used to monitor the off-gases by inserting the sampling tube into the top of the gas knockout box at the pipe plug shown in Figure 2.6. Gas samples also were taken from this location for analysis. Finally, volts and amps as previously discussed were periodically recorded along with the electrode movement.

2.2 DEVELOPMENT OF THE INEL WASTE SIMULANT

This section describes the approach and basis for the development of the waste simulants and test matrix used for evaluating the DC arc technology.

The DC arc technology is thought to be broadly applicable to the treatment of generic buried waste at many DOE complexes. However, because of the initial charter of this program, this work focused on evaluating the technology's applicability to a specific buried waste stream, that of the INEL SDA.

The SDA is 88 acres of land within a larger waste burial site known as the RWMC at the INEL site. Waste of various categories was disposed of in the SDA beginning in 1952 when the burial ground, or RWMC, was opened. Wastes disposed of at the SDA include organics, inorganics, toxic metals, and radio-nuclides. Much of the SDA waste was generated at many offsite locations and shipped to the INEL RWMC. The largest percentage of offsite waste buried at the SDA was derived from the shipment and subsequent burial of Rocky Flats Plant (RFP) waste. The waste simulant development effort for the DC arc testing program used all known characteristics to establish a full complement of waste simulants, thereby permitting a complete evaluation of the DC arc technology relative to its capabilities to treat this type of waste. The waste simulants developed and used in the Mark I testing program are grouped into five primary categories as shown below:

- INEL soil only
- INEL soil plus metals
- INEL soil plus combustibles

- INEL soil plus RFP 74 series sludges
- INEL soil plus high vapor pressure metals.

Although other waste categories exist, these five categories cover the major fraction of waste at the INEL SDA, and would be sufficient to complete a nearly comprehensive evaluation of the treatment of SDA waste using the DC arc process. The following sections describe the formulation of the various waste simulants.

The process of melting INEL soil produces a product that compositionally resembles natural basalt (Reimann 1992). The melted product of soil combined with the average waste composition at the SDA will produce a glass/ceramic product referred to as iron-enriched basalt (IEB). Due to the large quantities of contaminated soil at the RWMC, it is likely that the IEB or some variation closely resembling the IEB will serve as the waste form of choice for the stabilization of INEL RWMC wastes.

The primary objective of the waste simulant development effort was to establish what was considered to be the limiting compositions for the waste soil mixtures. Although it may be that a sufficient quantity of soil at the site exists to have higher soil-to-waste ratios, it presently is not known to what extent the surrounding soil is contaminated. It would be desirable not to add clean or uncontaminated soil to the waste for processing purposes to reduce the final volume of waste that ultimately will be permanently disposed of in deep geological repositories.

2.2.1 INEL Soil

The INEL soil used in all 13 of the Mark I tests was obtained from the INEL site. Table 2.1 gives an analysis of the soil's major constituents.

It has been reported that 8 to 12 million ft³ (227,000 to 340,000 m³) of contaminated soil plus waste exists at the SDA (Vigil 1989, Arrhenhoiz 1991). Of the total, roughly 63% is contaminated soil. For this reason, it is important to evaluate the potential for melting or treating the soil using only the DC arc technology. The INEL soil is a highly refractory material, melting in the range of 1400°C to 1500°C.

TABLE 2.1. Composition of INEL Soil^(a)

<u>Assumed Oxide</u>	<u>(Wt%)</u>
Al ₂ O ₃	12.34
CaO	9.54
Cr ₂ O ₃	0.10
Fe ₂ O ₃	5.41
K ₂ O	0.00
MgO	2.56
MnO ₂	0.10
Na ₂ O	2.89
NiO	0.10
PbO	0.10
SiO ₂	60.77
TiO ₂	0.71

(a) ICP analysis of INEL soil provided for this work.

2.2.2 INEL Plus Metals

The total quantity of metals buried in the SDA comprises over 22% of the waste on a mass basis. The process chosen to treat the SDA waste requires the capability to process large quantities of metals. Much of the buried wastes were disposed of in steel drums, and other metal objects were disposed of in their entirety at the SDA. To assess the effect of metals in the wastes on the processibility of the waste form, two metals containing simulants were developed for the Mark I tests. These simulants contained carbon steel, stainless steel, and aluminum.

2.2.3 INEL Soil Plus Combustibles

The SDA waste also has large quantities of combustible materials (42% volume basis, 20% weight fraction). A wide range of combustible wastes has been disposed of at the SDA, including combustible solids and organic liquids.

For the purpose of this evaluation, a waste simulant was prepared using waste surrogates comprising many of the solid combustible materials present in the SDA waste. These materials include paper, wood, plastic, and cloth. Consideration is being given to the possibility of incorporating many thermal treatment devices in the INEL buried waste treatment scenario (Mayberry 1991). Therefore, incinerator ash was included as a simulated waste for this program.

2.2.4 INEL Soil Plus RFP 74 Series Sludges

Waste descriptions for the INEL SDA include information on a variety of sludges that were buried or stored. The largest quantity of sludges buried at the SDA were shipped to INEL from RFP. The RFP sludges, referred to as the "74 series" sludges, were derived from waste stream and effluent processing at the RFP. Five primary sludges exist, as outlined in Table 2.2. The 741 and 742 sludges are similar compositionally; therefore, only one waste simulant was developed for both of these sludges. A simulant for the 743 sludge was also prepared and, because the similar nature of the 743 and 744 sludges and the likelihood for the 744 sludges to contain hazardous chemicals, one waste simulant was used for both the 743 and 744 sludges. The 745 sludge simulant was the final 74 series sludge simulant used in the Mark I tests.

The RFP 74 sludges were shipped to RFP in 55-gal drums. To simulate more realistically actual waste processing conditions, the waste simulants were prepared and packaged in scaled 55-gal drums. The containers used were 1/8 scale (on a linear scale, i.e. 3-in. x 2.25-in. dia). The basis for

TABLE 2.2. RFP 74 Series Sludge Description

<u>74 Series Sludge</u>	<u>Description</u>
741	First Stage
742	Second Stage
743	Organic Setups
744	Special Setups
745	Evaporator Slats

sizing the containers was the linear ratio of the Mark I furnace hearth diameter to that of the full-scale EPI furnace hearth located at Model City, New York. The volume of waste in the simulated containers was $(1/8)^3$ of the actual waste container.

2.2.5 INEL Soil Plus High Vapor Pressure Metals

Many thermal processes lead to concerns regarding the presence of metals in the waste that exhibit high vapor pressures at the temperature at which the waste is being treated. Metals of concern include lead, cesium, and to a lesser extent, plutonium. Cesium was evaluated here because of the heightened concern of the volatility of radioactive species in the melt. Cerium was also added to the melt to simulate actinides such as plutonium. A single waste simulant containing 1.4% Cs and 0.2% Ce introduced to the soil mixture in the form of nitrates was prepared for use in these tests.

2.3 MARK I EXPERIMENTAL METHODOLOGY

The typical experimental melt operation proceeded as follows:

- PNL designated the composition of the material to be melted and whether the melt should be conducted as a cold top or a hot top melt.
- EPI then determined the magnitude of current, arc voltage, rate of electric power input, total power input, and duration of each segment of the melt program for a particular melt type.
- After the furnace crucible was loaded with a particular type and weight of material, the furnace was purged with nitrogen, the melt program executed, the furnace cooled, and the ingot removed.
- Following ingot removal, all the dust vacuumed from the crucible and appropriate furnace areas was weighed. All solidified material not attached to the ingot was removed and weighed, the ingot was weighed, and electrode erosion was measured.
- During furnace operation, 4 CFM of nitrogen was injected into the furnace. During furnace cooling nitrogen was also continuously injected into the furnace and the venturi scrubber, and the stack gas fan continued to operate.

3.0 RESULTS AND DISCUSSION

Thirteen separate arc furnace runs were carried out to evaluate the processability of a wide range of waste simulant materials under a variety of operating conditions. These runs can be divided into five categories based on waste simulants. Table 3.1 lists these categories and their corresponding run numbers.

The following subsections provide detailed descriptions of each run, along with process data. For each category the material loaded into the furnace is described, the run conditions are given, and the processed material is described and characterized.

TABLE 3.1. Waste Simulant Categorization

<u>Waste Simulant Category</u>	<u>Run Number</u>	<u>Run Description</u>
Soil	1	INEL Soil - unsubmerged arc
	2	INEL Soil - submerged arc
	3	INEL Soil - submerged arc
Soil and Metals	6	INEL Soil and 25% metal mix
	7	INEL Soil and 50% metal mix
Soil and Combustibles	8	INEL Soil and wood, paper, plastic, cloth, and concrete
	4	Hospital Ash Incinerator
	5	Hospital Ash Incinerator
Soil and RFP Sludges	9	INEL Soil and RFP 745 sludge
	10	INEL Soil and RFP 741/742 sludge
	11	INEL Soil and RFP 743 sludge
Soil and Volatile Metals	12	INEL Soil and (CsNO ₃ and Ce(NO ₃) ₃) - submerged arc
	13	INEL Soil and (CsNO ₃ and Ce(NO ₃) ₃) - unsubmerged arc

In order to calculate weight change from processing, the feed stock weights, along with the carbon from the electrode and surrounding carbon, were logged before each run. After processing, the melt was removed from the furnace and the product was separated from the electrode. Weights of the electrode, slag, oxide layer, and metal layer (if any) were then individually logged. Table 3.2 lists a gross summary of this material balance information.

3.1 INEL SOIL

The first three runs processed 100% INEL soil in different processing modes. The primary objective of the first run was to determine how INEL soil processed in the Mark I furnace. During this first test, the furnace was operated with 86 lb (39 kg) of INEL soil in the hearth. A 1-1/4-in.-diameter by 16-in.-long starter rod (graphite) extended from the bottom of the furnace hearth to above the surface of the soil (Figure 3.1). This rod provided the conductive path for current flow to the hearth from the center moveable electrode. The arc was struck between the upper moveable center electrode and the

TABLE 3.2. General Overall Material Balances for 13 Mark I Tests

Run Number	Amount of Soil/Ash in Feed (Wt%)	Amount of Additives in Feed (Wt%)	Total Mass Decrease upon Firing (Wt%)	Amount of Oxides in Product (Wt%)	Amount of Metal in Product (Wt%)
1	100%	0%	13%	100%	0%
2	100%	0%	17%	100%	0%
3	100%	0%	24%	100%	0%
4	100%	0%	21%	100%	0%
5	100%	0%	54%	100%	0%
6	76%	24%	14%	69%	31%
7	51%	49%	2%	46%	54%
8	69%	26%	31%	100%	0%
9	73%	27%	28%	100%	0%
10	40%	60%	27%	100%	0%
11	50%	50%	27%	100%	0%
12	98%	2%	15%	100%	0%
13	98%	2%	16%	100%	0%

FIGURE 3.1. Mark I Hearth Loaded With INEL Soil - Run 1

starter rod. Although this approach to initiating the arc was used in the Mark I furnace, the Mark II design will have the ability to operate in either the transferred or the nontransferred mode so that a starter rod will not be necessary to initiate the melt. The melt proceeded by melting the soil from the surface down to the bottom of the hearth. Operation of the arc in this manner is referred to as "unsubmerged arc operation." Runs 2 and 3 were conducted to evaluate submerged arc operation. As was described in Section 2.2.1, the furnace was configured so that the arc was initiated below the surface of the soil contained in the hearth.

The conditions used for runs 2 and 3 were nearly identical, with the exception of run duration. During run 2, the power supplied to the furnace was 10.3 kWh and a total of 16 lb (7.3 kg) of soil were vitrified. In run 3, the total energy supplied was 56 kWh, vitrifying 80 lb (36.3 kg) of soil. In this nonoptimized Mark I furnace, the energy required to melt INEL soil ranged from 1.4 kWh/kg to 1.6 kWh/kg. This compares with energy-to-mass ratios of 1.1 to 1.9 kWh/kg for processing similar INEL soil in the engineering-scale in situ vitrification (ISV) process.

The direct temperature of the melt was not obtained during any of the Mark I experiments; however, during run 1 the use of an optical pyrometer indicated the temperature of the hearth directly above the melt was 896°C. It is estimated that the bulk glass was in the range of 1400°C to 1600°C for most of the Mark I experiments. It was determined that submerged arc operation was readily achieved in the Mark I furnace. Although a quantitative determination of off-gas particulate emissions was not completed during runs 1 through 3, it was observed that the submerged arc operation resulted in a lower buildup of particulate in the off-gas piping. Quantitative off-gas emission determinations for "submerged" versus "unsubmerged" operation are reported in Section 3.5 for runs 12 and 13. As shown in Table 3.2, a mass decrease range (upon firing) of 13% to 24% is observed for these three runs. Additionally, runs 12 and 13 were effectively 100% INEL soil (with small tracer spikes). Table 3.2 shows mass decreases from runs 12 and 13 of 15% and 16%, respectively. An average mass decrease of 17% upon firing can be determined for 100% INEL soil. Although Table 3.2 does not report any discernible metal phase in the melted soil product, most of the INEL soil melts contained a number of small metallic droplets. These droplets were dispersed so that effective separation was not possible. However, the metallic material generated in run 8 (INEL soil with added combustibles) was sufficient to permit ICP analysis of the metal phase.

Table 3.3 compiles elemental analyses of each phase of the Mark I runs (1-3, 12, and 13). These runs can be used to describe 100% INEL soil. Table 3.3 shows that the primary constituents in the products of these tests are SiO_2 (50% to 63% by weight), Al_2O_3 (12% to 15%), CaO (5% to 12%), and FeO (1% to 3%). By referring to the liquidus temperature ternary diagram for the SiO_2 - Al_2O_3 - CaO system, the predicted melting temperature of these three major INEL soil oxides was found to be between 1200°C to 1400°C (Maun and Osborn 1965). However, minor variations in any of these three components can produce a material with a melting temperature as high as 1600°C. Fortunately, impurities in the feed stock can drastically affect the melting temperature. FeO will reduce the melting temperature of the material, along with more conventional "fluxing" materials such as Na_2O , K_2O , and Li_2O . Therefore, based on the oxides shown in the INEL soil products, it appears that 100% INEL soil

TABLE 3.3. Compositional Phase Analysis of Mark I Runs (Runs 1-3, 12, 13)

Element Analyzed by ICP	Elemental Oxide Formula	Run #1 Oxide Phase (Wt% Oxide)	Run #2 Oxide Phase (Wt% Oxide)	Run #3 Oxide Phase (Wt% Oxide)	Run #12 Oxide Phase (Wt% Oxide)	Run #13 Oxide Phase (Wt% Oxide)
Al	Al ₂ O ₃	14.6%	12.2%	14.6%	13.4%	13.4%
Ca	CaO	9.4%	8.4%	11.7%	6.1%	5.5%
Cu	CuO	0.0%	0.0%	0.0%	0.0%	0.0%
Cr	Cr ₂ O ₃	0.0%	0.0%	0.0%	0.0%	0.0%
Fe	FeO	2.8%	2.3%	1.2%	1.6%	1.8%
K	K ₂ O	0.0%	0.0%	0.0%	1.2%	1.2%
Mg	MgO	1.3%	1.2%	1.5%	2.5%	2.4%
Na	Na ₂ O	0.8%	1.1%	0.7%	0.7%	0.8%
Ni	NiO	0.0%	0.0%	0.0%	0.0%	0.0%
Si	SiO ₂	50.1%	61.2%	54.9%	61.1%	62.9%
Sr	SrO	0.0%	0.0%	0.0%	0.0%	0.0%
Zn	ZnO	0.0%	0.0%	0.0%	0.0%	0.0%
B	B ₂ O ₃	0.4%	0.5%	0.2%	0.2%	0.3%
Ba	BaO	0.1%	0.1%	0.1%	0.1%	0.1%
Cd	CdO	0.0%	0.0%	0.0%	0.0%	0.0%
Co	Co ₂ O ₃	0.0%	0.0%	0.0%	0.0%	0.0%
Li	Li ₂ O	0.0%	0.0%	0.0%	0.0%	0.0%
Mn	MnO	0.1%	0.1%	0.0%	0.0%	0.0%
P	P ₂ O ₅	0.0%	0.0%	0.0%	0.0%	0.0%
Pb	PbO	0.0%	0.0%	0.0%	0.0%	0.0%
Ti	TiO ₂	0.7%	0.7%	0.6%	0.8%	0.8%
Zr	ZrO ₂	0.0%	0.0%	0.0%	0.0%	0.0%
Other	--	19.8%	12.2%	14.6%	12.4%	10.7%

can be effectively melted below 1500°C in a continuous operation. Note, however, that plasma temperatures (greater than 1600°C) can ensure melting of waste with a large degree of variability. Thus, by using a plasma operation on the INEL soil, a larger degree of conservatism for waste processibility is obtained.

The SiO₂-Al₂O₃-CaO system, which is the primary system in the INEL soil melts, is also known for its durability. Alumina (Al₂O₃) is typically known as a glass-strengthening additive. Its durability also should be reflected in

heavy metal leach tests. The oxide system formed by the melted INEL soil shows good resistance to leaching of heavy metals. TCLP analysis was completed to verify this.

The glassy product formed from this test, shown in Figure 3.2, appeared to be devitrified, probably because of the cool down time (i.e., 1500°C to 300°C in 24 h).

The glass produced in run 1 was analyzed using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS) to qualitatively determine what crystalline structures may have formed. The glass produced in the Mark I furnace is very reduced (e.g., $\text{Fe}^{++}/\text{Fe total} = 0.95 \pm 0.04$), which may have contributed to various crystalline phases observed in the product.

Previous studies of glass produced from the ISV processing of INEL soil have shown similar crystalline character in the vitrified product (Loehr 1991, Detering 1992, Callow 1991). Due to the reduced redox state of this glass, an area of crystals is observed in the Mark I glass that have not been observed in ISV melt tests of INEL soil. These large rectangular crystals, shown in

FIGURE 3.2. Vitrified INEL Soil

Figure 3.2, contain discernible levels of carbon, indicating that this formation may be a carbide formation or simply an area containing free carbon. Shown in Figure 3.3 is an SEM of this high carbon content crystalline structure.

3.2 SOIL AND METALS

Stainless steel, carbon steel, and aluminum pieces (1- to 2-in. long) were mixed into INEL soil within the furnace crucible. This mixture allowed an assessment of the processability of soil-metal mixtures by the DC arc furnace technique. Run 6 was performed with 25% metals by weight in INEL soil and run 7 was performed with a 50/50 mix of the two. In both runs the metal content was held at a constant ratio of stainless steel, carbon steel, and aluminum of 16%, 79% and 5%, respectively. The current was held at 600 amps for the first 15 min for each run, then increased to 700 amps for the remaining 2 h. Additionally, the arc voltage varied between 50 and 80V in both cases.

FIGURE 3.3. Photomicrograph of High Carbon Area in Vitrified INEL Soil

Table 3.2 shows the gross material balances for these two runs. The observed mass decrease for the two runs was 14% and 2%, respectively. Run 7 was expected to show a lower total mass decrease since it contained less soil, which was shown in the 100% INEL soil tests to decrease in mass by approximately 17%.

Both of the "metal" runs yielded products with discernible metal phases, unlike the raw soil test that yielded small metal droplet dispersions. The metals and slag phases produced in run 7 are shown in Figures 3.4 and 3.5. By dropping a separate metal phase, these tests allowed an estimation of the amount of metals (in the metal droplet dispersions) which nominally separated in the 100% INEL soil runs. When large amounts of metals are added to a molten glass/metal mixture, the extra metal acts as a "scavenger" of most of the metallic species in the system. This phenomenon is observed in the fire assay method used in processing gold ore. In this method lead is mixed with the ore and the whole mixture is melted. The result is a large pool of lead below the oxide layer that contains "scavenged" gold metal. By assuming that non-added

FIGURE 3.4. Metal Phase from High Metals Content Run

FIGURE 3.5. Glass/Slag Phase from High Metals Content Run

metals (reduced after processing or metallic from the beginning) are scavenged in the melting operation, run 6 data shows that 4.3% of the melted INEL soil is in metallic form and for run 7, 7.3% is in metallic form.

The mechanisms for metal reduction are not completely clear from the data. Indeed, some of the metallic elements which "dropped" out could have been metallic to start with. However, the majority of the observed metals are from carbon reduction and electrochemical reduction. Some metallic oxides in the presence of carbon at high temperatures will spontaneously be reduced by the free carbon in the system. This mechanism depends upon the Gibb's free-energies of the oxides, with respect to CO_2 . The only major INEL soil oxides that could effectively reduce in the presence of carbon are FeO and SiO_2 (Richardson 1948, Darken 1953). The oxide of aluminum has a greater absolute free-energy than either of the oxides of iron and silicon. Therefore, little or none of the aluminum added to the runs should be observed in the metal phase, as it will be oxidized by the oxygen atoms from the nearby FeO and SiO_2 to become an oxide itself. The iron and silicon are reduced by this process.

The analysis of the metal phase produced in run 6 (shown in Table 3.4) shows 80.5% iron, 8.4% silicon, 2.4% chromium, and only 0.4% aluminum. If the aluminum had not oxidized the composition in the metal phase would have been approximately 5% by weight. Therefore, this reduction shows obvious oxidation of aluminum within the melt.

TABLE 3.4. Compositional Phase Analysis of Mark I Runs 6 and 7

<u>Element Analyzed by ICP</u>	<u>Elemental Oxide Formula</u>	<u>Run #6 Oxide Phase (Wt% Oxide)</u>	<u>Run #6 Metal Phase (Wt% Elem.)</u>	<u>Run #7 Oxide Phase (Wt% Oxide)</u>
Al	Al ₂ O ₃	20.1%	0.4%	21.7%
Ca	CaO	9.4%	0.0%	8.5%
Cu	CuO	0.0%	0.1%	0.0%
Cr	Cr ₂ O ₃	0.0%	2.4%	0.0%
Fe	FeO	1.4%	80.5%	0.4%
K	K ₂ O	0.0%	0.0%	0.0%
Mg	MgO	2.7%	0.0%	2.4%
Na	Na ₂ O	0.8%	0.4%	0.6%
Ni	NiO	0.0%	0.6%	0.0%
Si	SiO ₂	45.1%	8.4%	39.9%
Sr	SrO	0.0%	0.0%	0.0%
Zn	ZnO	0.0%	0.0%	0.0%
B	B ₂ O ₃	0.3%	0.4%	0.1%
Ba	BaO	0.1%	0.0%	0.1%
Cd	CdO	0.0%	0.0%	0.0%
Co	Co ₂ O ₃	0.0%	0.0%	0.0%
Li	Li ₂ O	0.0%	0.0%	0.0%
Mn	MnO	0.1%	0.5%	0.1%
P	P ₂ O ₅	0.0%	0.0%	0.0%
Pb	PbO	0.0%	0.0%	0.0%
Ti	TiO ₂	0.5%	0.2%	0.5%
Zr	ZrO ₂	0.0%	0.0%	0.0%
Other	--	19.4%	6.0%	25.7%

Although reduction of the observed metals in run 7 was due partly to the oxidation of the added aluminum metal, some reduction was due unquestionably to the oxidation of carbon from the electrode, crucible, and organics within the INEL soil. By performing molar balances around the aluminum oxidation (to Al_2O_3) and carbon oxidation (to CO_2) reactions, an estimate of carbon consumption in the melt can be obtained. These calculations, for run 7, estimate that 45% of the reduction is due to aluminum oxidation and 55% is due to carbon oxidation.

Both aluminum and carbon oxidation contribute to the thermal reduction of glass oxides. However, as mentioned previously, a mechanism for electrochemical reduction of the metals may also exist. This can be attributed to the DC arc used in Mark I. Like the electrolysis of water to H_2 and O_2 gas, this effect can be obtained in a molten oxide bath, producing metal and oxygen. Faraday's Laws can be applied to the run 7 conditions to estimate the total amount of metal which could (theoretically) be precipitated. This run consumed approximately 1370 Amp-hr. By assuming that iron oxide (only) reduces to metallic iron from a +3 valence state, a total of 953 g (2.1 lb) of iron could theoretically be produced from the oxide solution. This estimated amount of iron could account for the metal observed in run 7. However, the kinetics between thermal reduction and electrochemical reduction for the material have not yet been established.

A method for quantitatively evaluating the degree of reduction in a glassy material is to measure the relative amounts of each valence state for a multivalent metal. For example, iron has valence states of +2 and +3, hence the oxides FeO and Fe_2O_3 . Therefore, glasses with iron oxide(s) present can be analyzed for both Fe^{+2} and Fe^{+3} . The ratio of these values can then be determined to describe the amount of reduction and oxidation (redox) in the glass. Manganese is another multivalent species that can be used to describe glass redox.

Table 3.5 shows the glass redox information obtained for the first eight Mark I runs. Instead of giving this ratio as $\text{Fe}^{+2}/\text{Fe}^{+3}$, this data is given as $\text{Fe}^{+2}/\text{Fe}_{\text{Total}}$. From the data shown in the table, it appears that the first eight DC arc runs effectively had redox ratios of one (based on incorporating

TABLE 3.5. Redox Values for Mark I Runs

<u>Run Number</u>	<u>Ferrous Iron/ Total Iron</u>
1	0.99
2	0.98
3	0.99
4	0.99
5	0.97
6	0.96
7	1.02
8	0.99
9	0.99
10	0.99
11	0.99

analytical error). This means that all of the iron oxide in the glass phase exists as FeO for those runs, which is a more reduced state than Fe_2O_3 . These runs were expected to yield these types of redox ratios since they were reduced enough to drop some iron and silicon out of the oxide solution.

3.3 SOIL AND COMBUSTIBLES

For run 8 a large amount of combustible material was added to INEL soil and then fired in the Mark I furnace. For this experiment 66.7% by weight of soil was mixed with office paper (11.4%), concrete chips (4.7%), terrycloth (1.3%), sawdust (4.7%), 1-in. wood blocks (10.8%), and pieces of polyethylene bag (0.4%). Approximately 61 kWh were delivered into the furnace in a 2-h run period. Figure 3.6 shows a cross section of the vitrified product from run 8. There was no indication of any residual carbonaceous material in the glass product. Lack of carbon was not anticipated for pyrolytic operation, as was the case for this run. This indicates that the DC arc process may be suitable for buried wastes containing large quantities of combustible material. Typically, this type of material requires pretreatment such as traditional incineration.

FIGURE 3.6. Glass Produced in Run 8

Because incineration is a possible pretreatment option for combustible materials or mixtures of inorganics and combustibles, tests were completed in the Mark I furnace using incinerator ash as a feed material. Ash was obtained from a hospital incinerator and processed in the Mark I arc furnace. The ash was produced from medical waste, which had been incinerated at $\sim 2000^{\circ}\text{F}$ and water quenched. The ash was dried in a 150°F oven at MIT for 5 to 6 h before being loaded into the crucible. The ash was processed at 30 to 50V and 600 amps (53 kWh) for the first run and 50V and 700 amps (61 kWh) for the second run. A top layer of slag was not observed in the first run but in the second run, with drier ash and higher metal and glass contents, a slag material was observed. Figure 3.7 shows the resultant slag/metal layer formed in run 5. However, as previously mentioned, this run was also performed with a higher power input than that used in the first run, which may have contributed to the larger amount of slag.

The material balance values for both ash tests (runs 4 and 5) are also shown in Table 3.2. These values show a large difference in mass decreases

FIGURE 3.7. Slag Layer from Ash Run

for both of the runs (21% and 54%, respectively). However, run 5 was performed in two different heat-up schemes. Bringing the Mark I furnace up to temperature two different times on one sample can burn off extra carbon from the electrode and crucible. Therefore, the higher mass decrease observed in run 5 may not be representative of the true mass decrease for the ash.

The material was pre-dried before testing at 150°C in a 6-h schedule. Therefore, the observed mass decrease after the plasma runs largely should not have been due to water vaporization. Another contributing factor to the observed mass decrease in run 4 was the production of metals in the melt. Results from the inductively coupled plasma (ICP) analysis in Table 3.6 show that the run 4 metal phase contained primarily iron and silicon metal. Therefore, like the INEL soil test with added combustibles (run 8), the observed total mass decrease from run 7 could be due to the reduction of iron oxide and silicon oxide from the carbon present in the feed stock.

TABLE 3.6. Compositional Phase Analysis of Ash Runs

Element Analyzed by ICP	Elemental Oxide Formula	Run #4 Oxide Phase (Wt% Oxide)	Run #4 Metal Phase (Wt% Elem.)	Run #5 Oxide Phase (Wt% Oxide)	Run #8 Oxide Phase (Wt% Oxide)	Run #8 Metal Phase (Wt% Elem.)
Al	Al ₂ O ₃	35.8%	0.2%	36.5%	13.7%	0.2%
Ca	CaO	22.7%	0.1%	25.1%	6.8%	0.2%
Cu	CuO	0.1%	4.6%	0.0%	0.0%	0.0%
Cr	Cr ₂ O ₃	0.0%	3.2%	0.0%	0.0%	0.3%
Fe	FeO	0.5%	34.1%	0.1%	1.7%	42.1%
K	K ₂ O	0.0%	0.0%	0.0%	0.0%	0.0%
Mg	MgO	0.9%	0.0%	1.1%	2.5%	1.0%
Na	Na ₂ O	0.1%	0.2%	0.0%	0.8%	0.8%
Ni	NiO	0.0%	0.4%	0.0%	0.0%	0.0%
Si	SiO ₂	21.5%	9.1%	20.8%	59.2%	32.2%
Sr	SrO	0.1%	0.0%	0.1%	0.0%	0.0%
Zn	ZnO	0.0%	0.0%	0.0%	0.0%	0.0%
B	B ₂ O ₃	1.4%	0.7%	1.3%	0.3%	0.2%
Ba	BaO	3.2%	0.0%	3.2%	0.1%	0.0%
Cd	CdO	0.0%	0.0%	0.0%	0.0%	0.0%
Co	Co ₂ O ₃	0.0%	0.0%	0.0%	0.0%	0.0%
Li	Li ₂ O	0.0%	0.0%	0.0%	0.0%	0.0%
Mn	MnO	0.0%	0.7%	0.1%	0.1%	0.3%
P	P ₂ O ₅	0.0%	0.0%	0.0%	0.0%	0.0%
Pb	PbO	0.0%	0.0%	0.0%	0.0%	0.0%
Ti	TiO ₂	0.6%	4.6%	0.6%	0.5%	0.9%
Zr	ZrO ₂	0.0%	0.0%	0.0%	0.1%	0.0%
Other	--	13.0%	42.2%	11.1%	14.2%	21.9%

The oxide phase compositions produced in runs 4 and 5 are listed in Table 3.6. The primary constituents, according to this data, are Al₂O₃ (35% to 37%), CaO (22% to 25%), and SiO₂ (20% to 22%). Like the INEL soil results, this shows that the system primarily can be described by a SiO₂-Al₂O₃-CaO system. Therefore, the liquidus temperature ternary diagram for this system can again be used to predict the melting temperature range of the material. For the composition ranges described above for the ash, the melting temperature

range for the primary oxides is between 1400°C and 1600°C. However, the impurities that exist in the material should result in a melting temperature below 1500°C, which was supported by the vitrified products pulled from both MGH ash runs.

3.4 SOIL AND RFP SLUDGES

The RFP 74 series sludges, as described previously (see Section 2.2), were prepared and packaged in the 1/8-scale 55-gal drums. The sludge surrogates are described in Table 3.7.

A specific procedure was used for loading the simulated drums containing the RFP sludges into the furnace. The lids of the cans were punctured with three small (1/8-in.) holes before the cans were loaded into the furnace. Figures 3.8 and 3.9 are photographs of the loading configuration for the 16 RFP 743 sludge containers before and after INEL soil was added. For the

TABLE 3.7. RFP 74 Series Sludge

RFP 741/742	Silica (23.2%), $\text{Al}(\text{OH})_3$ (4.6%), $\text{Ca}(\text{OH})_2$ (23.2%), NaOH (24%), KOH (4%) and MgSO_4 (30%)
	Waste Simulant - 60% wt%
	Soil - 40% wt%
RFP 743	Texaco Regal Oil 240 (66.7%) and Microcell-E (33.3%)
	Waste Simulant - 50% wt%
	Soil - 50% wt%
RFP 745	NaNO_3 (60%), KNO_3 (30%) and Portland Cement (10%)
	Waste Simulant - 27% wt%
	Soil - 73% wt%

FIGURE 3.8. Loading of Simulated RFP 743 Series Sludge Drums in Mark I Furnace Hearth

741/742 sludge run, 18 cans were stacked in 3 layers (7-7-4) and 13 lb of INEL soil were added until the soil level reached the middle of the second row of cans. For the RFP 745 sludge run, two layers of cans (6-4) were used.

The RFP 74 series sludges are wastes that could adversely affect the glass durability if the soil-to-waste ratios are not controlled. For this reason it was of interest to evaluate the limiting conditions to assess both waste composition and the Mark I furnace limitations. Table 3.7 shows the soil-to-waste ratios for each of the three RFP sludge runs. Table 3.8 presents the compositional analysis of the glass product produced in these three runs, in addition to the presently accepted compositional range for the IEB waste form. In all three runs the Mark I furnace effectively processed the soil-sludge mixtures. However, in run 11 the furnace operation was terminated prematurely because the off-gas line became plugged. The Mark I off-gas system was by no means optimized for these tests, and it was not the intent of

FIGURE 3.9. RFP 743 Series Sludge Containers with INEL Soil Added to Hearth

the Mark I program to evaluate its performance. As stated earlier, the primary objective was to determine if the INEL waste simulants could be effectively vitrified in a DC arc furnace. The resultant partially vitrified/melted mass from run 11 is shown in Figure 3.10. Useful data did result from this test. However, it was observed that the arc stability was maintained in the furnace, as indicated by the even radial distribution of fused material, even though a large volume of gas was generated in this run.

Nineteen and seven-tenths (19.7) pounds of RFP 741/742 sludge cans were processed with 13.2 lb of INEL soil. The processing time was 90 min and used a total of 32 kWh (approximately 2 kWh/kg) of DC power. The RFP 743 sludge cans were processed at 40V and 400 to 500 amps for 105 min, yielding an input energy of 56 kWh. The RFP 745 cans were processed at 40 to 50V and 500 to 600 amps for 2 h, yielding an energy input of 52 kWh.

TABLE 3.8. Compositional Phases Analysis of RFP 74 Series Sludge Runs

Element Analyzed by ICP	Elemental Oxide Formula	Run #9 Oxide Phase (Wt% Oxide)	Run #10 Oxide Phase (Wt% Oxide)	Run #11 Oxide Phase (Wt% Oxide)	IEB Tolerance (Range - Wt%)
Al	Al ₂ O ₃	12.3%	8.8%	16.9%	9-20
Ca	CaO	9.0%	11.3%	8.4%	8-20
Cu	CuO	0.0%	0.0%	0.0%	--
Cr	Cr ₂ O ₃	0.0%	0.0%	0.0%	--
Fe	FeO	0.5%	4.4%	0.5%	10-38
K	K ₂ O	3.0%	3.5%	2.9%	0-8
Mg	MgO	2.4%	5.2%	4.0%	0-10
Na	Na ₂ O	3.3%	5.5%	0.8%	0-6
Ni	NiO	0.0%	0.0%	0.0%	--
Si	SiO ₂	57.5%	49.3%	60.3%	38-57
Sr	SrO	0.0%	0.0%	0.0%	--
Zn	ZnO	0.0%	0.0%	0.0%	--
B	B ₂ O ₃	0.3%	0.2%	0.1%	--
Ba	BaO	0.1%	0.0%	0.1%	--
Cd	CdO	0.0%	0.0%	0.0%	--
Co	Co ₂ O ₃	0.0%	0.0%	0.0%	--
Li	Li ₂ O	0.0%	0.0%	0.0%	--
Mn	MnO	0.0%	0.0%	0.0%	--
P	P ₂ O ₅	0.0%	0.0%	0.0%	--
Pb	PbO	0.0%	0.0%	0.0%	--
Ti	TiO ₂	0.7%	0.3%	0.9%	--
Zr	ZrO ₂	0.0%	0.0%	0.0%	0-15
Other	--	10.8%	11.5%	5.1%	--

3.5 INEL SOIL AND VOLATILE METALS

Runs 12 and 13 used INEL soil spiked with volatile metals. The objective of runs 12 and 13 were twofold—first to evaluate the high vapor pressure metal (HVPM) release from the DC arc process on an absolute scale, and second to determine the effect of "submerged" arc operation on HVPM volatility rates.

FIGURE 3.10. Partially Melted Slag and 743 Series Sludge Containers

The two different runs corresponded to the submerged and unsubmerged electrode configurations, respectively. The volatile metals chosen for these experiments were cesium and cerium in their nitrate forms. Material balances on the cesium and cerium were performed by analyzing the accumulated off-gas carry-over and comparing the total amount of element with that mixed into the soil.

3.5.1 Off-Gas Sampling and Analysis

Because the primary objective of runs 12 and 13 was to assess HVPM volatility, a full description of the off-gas sampling and analysis is presented in the following sections.

The off-gas sampling for runs 13 and 14 was accomplished using an isokinetic sampling train designed to collect particulate samples from a particulate laden gas stream. The primary objective of the off-gas sampling effort was to quantify the amount of cesium volatilized into the off-gas as a result of the vitrification process and compare the results of the two runs.

Sampling results confirm that the near surface start configuration of run 13 generated approximately double the quantity of particulate in the off-gas as did run 12 (213 g versus 116 g). Run 12 volatilized 0.88% of elemental cesium into the off-gas, compared with 1.9% for run 13.

In addition to the cesium concentrations, the average amount of water vapor determined to be in the off-gas for run 12 was 16.88%, compared with 11.17% for run 13.

3.5.2 Sampling Methodology

The determination of particulate emissions from a stationary source requires the measurement of multiple source variables. The off-gases emitted from a vitrification process are a mixture of gas components and particulate of varying sizes with varying temperature and moisture levels. The off-gas sampling system used for runs 12 and 13 extracts a representative sample while monitoring source and system variables. The data from this representative sample is used in calculating average particulate concentration in the off-gas as well as average moisture concentration. The off-gas sampler consists of a commercial stack sampler built in accordance with the U.S. Environmental Protection Agency (EPA) standards as outlined in the Federal Register Vol. 42, No. 160 dated August 18, 1977. Off-gas samples are isokinetically extracted through a nozzle and a heated probe into a heated filter where the particulate is removed. The hot gases are then passed through a series of cold (contained in an ice bath) impingers where the condensibles are removed and the gases are cooled before going to the pump, dry gas meter, and flow orifice. This sampling method is described in 40 CFR, Part 60, App. A as Method 5, "Determination of Particulate Emissions From Stationary Sources." A schematic of the sampling system used on the off-gas line is shown in Figure 3.11. The filter media used to capture particulate is composed of borosilicate glass fibers and has a collection efficiency of 99.97% on 0.3-micron D.O.P. particles (standard D.O.P. penetration test). Distilled water was used as a scrubbing solution in the impingers to take advantage of the water soluble nature of cesium.

FIGURE 3.11. Schematic of Off-Gas Sampling System

3.5.3 Off-Gas Sampling Setup

The plasma arc furnace used for the vitrification process operates with no air in-leakage and uses a nitrogen gas purge of approximately 5 cfm (142 L/min). This low flow rate, contained in a 3-in. schedule 40 pipe, results in a very low off-gas velocity that requires a larger nozzle diameter (0.75 in.) for sampling than is typically used. The larger nozzle made it possible to acquire enough sample material for an accurate analysis while collecting the sample at a velocity equal to the off-gas velocity to remain isokinetic. The relation of the off-gas sampler location to the furnace is shown in Figure 3.12.

3.5.4 Sampling Operation

The sampling operation for both runs proceeded very smoothly without any sampling downtime. Run 13 was sampled throughout the entire run, with a total sample time of 108 min. The dry sample volume collected totaled 33.67 ft³. This volume is reported as a dry gas because the dry gas meter is located downstream from the impingers and silica gel, which remove all the water. There was 123 mL of water that collected in the scrubbing impingers, with 13.2 g of water absorbed by the silica gel. The particulate filter collected 6.17 g of dry particulate.

FIGURE 3.12. Off-Gas Sampler Location

Run 13 was sampled throughout the entire run with a total sample time of 106 min. The dry sample collected totaled 34.7 ft³ with 78 mL collected in the scrubbing impingers and 9.2 g of water absorbed onto the silica gel. The particulate collected on the particulate filter weighed 11.24 g.

Sample Recovery. Sample recovery involves collecting all the various samples collected in the sample train, including removing and collecting the particulate deposited in the sample line. To achieve a representative particulate sample, all the particulate entering the sample nozzle must be accounted for. Other activities include collecting and measuring volumes of impinger solutions and removing the particulate filter and loose particulate from the filter housing. The filter and particulate were first dried in an oven and then returned to ambient temperature in a desiccator before weighing

for accumulated solids. The sample line rinse was also dried in an oven and the resulting particulate residue weighed to determine the amount of particulate deposited in the sample line.

Sample Analysis. The particulate on the filter samples was analyzed using flame atomic absorption and inductively coupled plasma spectroscopy (ICPS). The samples were divided into thirds and prepared for analysis using potassium fusion, sodium fusion, and water leach techniques. The fusion preparation enables the total inorganic composition of the sample to be determined while the water leach indicates the presence of water soluble components. The results of these analyses for run 12 are displayed in Tables 3.9 and 3.10. Table 3.11 and 3.12 present the results for run 13.

The off-gas particulate components are reported as oxides because of the oxidizing sample preparation technique used for analytical purposes. This reporting method does not suggest these components were collected as oxides during sampling of the tests. Figure 3.13 shows a photograph of the filter samples that were obtained from runs 12 and 13. Figures 3.14, 3.15, and 3.16

TABLE 3.9. Off-Gas Particulate Composition for Run #12

<u>Oxide</u>	<u>KOH Fusion Oxide, Wt%</u>	<u>Na₂O₂ Fusion Oxide, Wt%</u>	<u>H₂O Leach Oxide, Wt%</u>
Al ₂ O ₃	0.69	1.05	0.00
CaO	0.27	0.74	0.08
CeO ₂	0.00	--	--
Cs ₂ O	3.95	4.68	1.52
FeO	1.58	0.77	0.00
K ₂ O	3.13	3.13	1.43
MgO	0.36	0.65	0.06
Na ₂ O	1.79	1.79	1.81
SiO ₂	69.30	74.08	1.02
ZnO	.012	0.00	0.14
B ₂ O ₃	0.32	0.29	0.04
P ₂ O ₅	0.93	1.86	1.79
Total	82.44	89.05	7.89

TABLE 3.10. Volatilized Material (Mass Percent) for Run #12

<u>Oxide</u>	<u>Soil, Wt%</u>	<u>Off- Gas, Wt%</u>	<u>Soil Constituents Volatilized into the Off-Gas, %</u>
Al ₂ O ₃	12.34	0.87	0.02
CaO	9.54	0.51	0.02
CeO ₂	0.26	0.00	0.00
Cs ₂ O	1.44	4.32	0.88
FeO	5.07	1.18	0.07
K ₂ O	2.88	3.13	0.32
MgO	2.56	0.51	0.06
Na ₂ O	2.89	1.79	0.18
SiO ₂	60.77	71.69	0.35
B ₂ O ₃	0.06	0.31	1.61

TABLE 3.11. Off-Gas Particulate Composition for Run #13

<u>Oxide</u>	<u>KOH Fusion Oxide, Wt%</u>	<u>Na₂O₂ Fusion Oxide, Wt%</u>	<u>H₂O Leach Oxide, Wt%</u>
Al ₂ O ₃	0.81	0.65	0
CaO	0.45	0.49	0.03
CeO ₂	0.00	--	--
Cs ₂ O	5.85	4.31	0.80
FeO	0	0	0
K ₂ O	0	0	1.09
MgO	0.56	0.42	0
Na ₂ O	3.04	3.04	1.05
SiO ₂	84.04	75.04	0.94
ZnO	.050	0.12	0.03
B ₂ O ₃	0.37	0.26	0.02
P ₂ O ₅	2.57	1.14	1.00
Total	98.49	85.75	4.96

TABLE 3.12. Volatilized Material (Mass Percent) for Run #13

<u>Oxide</u>	<u>Soil Wt%</u>	<u>Off- Gas Wt%</u>	<u>Soil Constituents Volatilized into the Off-Gas, %</u>
Al ₂ O ₃	12.34	0.73	0.03
CaO	9.54	0.47	0.03
CeO ₂	0.26	0.00	0.00
Cs ₂ O	1.44	5.08	1.91
FeO	5.07	0.00	0.00
K ₂ O	2.88	0.00	0.00
MgO	2.56	0.49	0.10
Na ₂ O	1.50	3.04	0.55
SiO ₂	60.77	79.54	0.69
B ₂ O ₃	0.06	0.32	2.98

are photomicrographs of off-gas particulate from run 13 showing that the size of the particulate is submicron for the majority of the particles.

3.5.5 Summary of Off-Gas Analysis

The volatility of HVPs from a DC arc furnace is comparable to that for the high-level waste treated in liquid fed ceramics melters (LFCMs) and by ISV. For operation of the furnace in the nonsubmerged mode, the retention of Cs within the glass product was measured to be 98.1 wt%. For submerged arc operation, 99.12 wt% Cs retention was observed. These values compare favorably to 90% to 99.25% for the LFCM technology and 99.63% for ISV (Carter 1988, Peterson 1992, Goles 1983). The total mass fraction lost to the off-gas was measured to be 0.3 wt% and 0.54 wt% for submerged and nonsubmerged arc operation, respectively, based on all inorganic constituents of the feed material. There was not any detectable Ce in either of the off-gas samples.

FIGURE 3.13. Photograph of Off-Gas Filters from Runs 12 and 13

FIGURE 3.14. Photomicrograph of Off-Gas Particulate from Run 13

FIGURE 3.15. Photomicrograph of Off-Gas Particulate from Run 13

FIGURE 3.16. Photomicrograph of Off-Gas Particulate from Run 13

4.0 PERFORMANCE OF THE MARK I DC ARC FURNACE RELATIVE TO DESIGN AND FABRICATION OF MARK II FURNACE

A summary of the performance and operating characteristics of the Mark I furnace follows.

- The Mark I furnace was operated only in the batch loading mode, even though the furnace was designed with three loading ports for on-line loading operation. Since the MIT Plasma Fusion Center Safety Committee recommended against staff being present on the furnace floor level when power was applied, only batch-type loading could be used when the furnace was cold. After the melt, the power was shut off and the furnace was allowed to cool. When the furnace was safe to handle, the ingot was removed.

This batch mode of operation was not conducive to accurately measuring the efficiency of the DC arc furnace since the crucible, furnace lining, and the overall thermal insulating systems had not reached the steady state operating temperature. Even so, the total power input approached 0.5 kWh/lb of material being melted.

The Mark II furnace is a continuous fed furnace system; therefore, this information is not directly applicable to the Mark II design.

- Fourteen melts were made with relatively few problems. On three occasions the melt time was shortened relative to the planned melt campaign. Each of these melt time reductions was caused by a release of furnace gas, or by furnace pressure buildup from the improper removal of particulate that had accumulated in the 3-in. gas duct from previous runs.

The information obtained from the Mark I runs relative to the particulate loading was used in the design and specification of the Mark II off-gas system.

- The entire electrical system performed flawlessly during every melt, regardless of the composition of the material in the crucible.

Arc voltage fluctuations were minimal, arc stability was good, and in all cases rate of power input and total power input was easy to control regardless of whether the furnace was operated in the submerged arc mode, cold topping mode, or the "above melt" hot topping mode.

At the end of each of the melts, with the power still on, the moveable electrode was lowered through the melt until measurements showed that the arc was extinguished by causing the graphite electrodes to touch. This procedure permitted easy removal of the ingot from the crucible by raising the moveable electrode after the ingot had cooled sufficiently.

This information confirmed the electrical design for Mark II.

- Erosion of the negative electrode was significantly less than erosion of the positive electrode by the amount predicted. When "hot topping" tests were performed, it was desirable to control the erosion of the positive electrode so that arc penetration through the cold material in a direction toward the bottom of the crucible would proceed at a desired rate. Arc and electrode erosion were controllable so that the positive electrode could be consumed in the predetermined melt time.

This information again confirmed the design approach for the Mark II electrode system.

Composition of initial materials in the waste may reduce the weight of the 2-in.-dia negative electrode material consumed by as much as 3.6 lb, but in general the weight of the negative electrode consumed is on the order of 1.2 lb per run.

- The Mark I furnace can be operated only in the transferred arc mode, but in this mode DC arc stability, concentricity relative to the walls of the graphite crucible, arc voltage control versus electrode position, and negative electrode erosion were readily controlled over a wide range of current in any of the melts.

DC arc stability is generally better at high current when used with the proper electrode size and configuration. Since the low current characteristics of stability and controllability were demonstrated to be excellent with the Mark I design, this reconfirms that graphite electrode DC arc furnaces can be scaled up by a factor of more than two orders of magnitude on future designs.

- The Mark I design has further demonstrated that carbon and graphite experience very little erosion even in direct contact with molten INEL soil with a rather wide range of surrogate additives and with short DC arcs. The Mark II furnace lining should therefore perform well when subjected to INEL materials similar to those melted in the Mark I furnace.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The results of the Mark I furnace testing program indicate the graphite electrode DC technology is well suited to the treatment of buried wastes. The technology has been demonstrated to be effective for treating a diverse mixture of materials, including metals, combustibles, sludges similar to those found at the INEL site, and mixtures containing high vapor pressure metals.

The unique capability of the graphite electrode to operate in the submerged mode will substantially improve the partitioning of materials processed or treated using this technology. This was effectively demonstrated with operation of the arc in both the submerged and unsubmerged modes. The results of these two runs showed conclusively that the emissions from an arc furnace could be reduced by over a factor of two.

The Mark I tests that were completed all produced products that were in a highly reduced state, primarily because the Mark I furnace operating conditions were not optimized. It is not anticipated that the pilot-scale (Mark II) furnace will produce a highly reduced waste form. At this time it is unclear whether or not the reduced state of the glass would diminish the quality of the waste form. The only concern with a reduced redox state is the capability to contain adequate amounts of metal in the glass.

The Mark I tests using large quantities of combustible material resulted in the nearly complete pyrolysis of the carbonaceous material and the formation of a densified glass product from the resultant inorganic ash and soil mixture.

The capability to treat wastes containing large quantities of metals was demonstrated with extremely favorable results. The metal phase separates cleanly from the glass phase. Both the glass and metal products are highly densified and would serve as a suitable waste form. If it is later determined that the majority of the radioactive contaminants remain in the glass phase, it may be advantageous to operate an arc furnace in a mode (reducing environment) that will promote the separation of metals from the glass. This determination will be made under future program activities and in conjunction with ongoing programs within the BWID program.

The overall operability of the furnace proved to be a success. Information gained from the Mark I program was used to enhance the design of the Mark II furnace. The Mark II furnace is an optimized furnace that has been specifically designed with the goal of treating buried wastes such as those found at the INEL. The future activities planned for the Mark II furnace will build on the information obtained during the Mark I experiments.

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