Clemson Final Report - High Temperature Formulations for SRS Soils

by

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Clemson Final Report - High Temperature Formulations for SRS Soils (U)

SUMMARY

Previous experience with in-situ (Joule-heated) vitrification (ISV) of Savannah River Site (SRS) highly weathered soils has shown that the SRS soil was both very refractory and a poor electrical conductor. These findings bring into question the likelihood of utilizing this type of vitrification treatment for waste sites and basins at SRS. An alternative approach may be in-situ plasma vitrification (ISPV). The ISPV approach also has a safety advantage in that the melting is initiated at the bottom of a bore hole compared to top-down melting for Joule heated ISV.

Vitrification research utilizing DOE Landfill Focus Area funds was recently completed. A series of experiments on flux additions to the SRS soil was evaluated at the Clemson DOE-Industrial Center for Vitrification Research. Three flux additions were evaluated in high-temperature crucible tests and DC graphite arc furnace studies. The three additives selected were colemanite, dolomitic lime, and hydrated lime. Of the three additives, the dolomitic lime material was the most advantageous fluxing additive. A level of about 25 weight percent was required to reduce the melting temperature into the 1500°C range. This material was successfully fed through the primary graphite electrode in the Electro Pyrolysis DC arc melter to the region of the plasma arc over the SRS soil. Analysis of the offgas streams revealed very high particulate content and very low oxygen content. Only trace amounts of hydrogen and carbon monoxide were detected. Dry flux injection is potentially feasible. Glass and soil analyses for samples obtained from these studies will be presented in a later report.

INTRODUCTION

In-situ vitrification of radioactive and hazardous waste sites by Joule heating is a commercially developed technology (Geosafe Corporation) with hundreds of tests and fairly extensive commercial demonstrations. It should be noted that during the testing there have been a small number of glass or hot gas expulsions from the melt.

This process utilizes the passage of large electrical currents through the soil between graphite electrodes to provide the thermal energy required to melt the soil. The melt is initiated on the surface and as the melt progresses deeper into the earth the electrodes are gradually lowered. Initial attempts to use this technology for the Savannah River Site (SRS) were not successful due to the low electrical resistivity and refractory nature of the SRS soils. In order to achieve vitrification of the SRS soils it would be necessary to mix or saturate the SRS soils with a suitable flux or generate extremely high temperatures in the vitrification regions. The SRS soils would be representative of other highly weathered sedimentary coastal regions of the United States.

Utilizing funding from DOE's Office of Science and Technology (OST), investigations were undertaken at two regional Universities to study soil additives and in-situ plasma arc vitrification of SRS soils. This report presents the conclusions of the Clemson University study on the addition of additives to the SRS soil. The results from the Georgia Institute of Technology will be distributed in the near future.

BACKGROUND

Based on previous high-temperature vitrification studies on SRS soils(1-2), the Land-Fill Stabilization and Containment Focus Area (SR-16LF-52), under the DOE Office of Science and Technology, funded an additional study of in-situ vitrification additives at Clemson University. This study was initiated early in FY 96 and completed with the release of the report included as Appendix A. A SCUREF Task Order, No. 187, was prepared and approved to start in January of 1996. This task was to be completed within nine months at a total cost of \$65,500.

The goal of this effort was to conduct crucible-melt studies with potential soil additives and demonstrate the feasibility of high-temperature melting technology with the Electro-Pyrolysis Inc. DC electric arc melter.

CONCLUSIONS

The experimental procedures and detailed conclusions are presented in the attached final report - See Appendix A.

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2. A.L. Kielpinski, J.C. Marra, J. Etheridge, R.Kirkland, R.F. Schumacher, and V. Rogers, "Development of Plasma Vitrification Technology for Contaminated Soil at the Savannah River Site," Tuscon Waste Management 95, OSTI as DE05060091, WSRC-MS-95-0056.

APPENDIX A

**Final Report on High Temperature Vitrification Formulation Demonstration for
SRS Contaminated Soils** by D.L. Erich, T.J. Overcamp, M.Speer, D.M. Cash, S.J.
Griner, Received January 22, 1997.

**FINAL REPORT
ON
HIGH TEMPERATURE VITRIFICATION
FORMULATION DEMONSTRATION FOR SRS
CONTAMINATED SOILS**

SCUREF Task Order #187

Clemson University Project #6338

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**FINAL REPORT
ON
HIGH TEMPERATURE VITRIFICATION FORMULATION
DEMONSTRATION FOR SRS CONTAMINATED SOILS**

EXECUTIVE SUMMARY

This study was undertaken to demonstrate the application of a DC arc melter to *in-situ* vitrification of SRS soils. The melter that was available at the DOE/Industrial Vitrification Laboratory at Clemson University was equipped with opposing solid electrodes. To simulate field conditions, two hollow electrode configurations were evaluated which allowed fluxes to be injected into the melter while the soils were being vitrified. The first 4 runs utilized pre-blended flux (two runs) and attempted flux injection (two runs). These runs were terminated prematurely due to offgas sampling problems and melt freezing. The remaining four runs utilized a different electrode geometry, and the runs were not interrupted to change out the offgas sampling apparatus. These runs were conducted successfully.

Three fluxes were evaluated in this study; colemanite, dolomitic lime and hydrated lime. Colemanite did not sufficiently decrease the viscosity of the melt and was not used successfully in this study. Conversely, both dolomitic and hydrated lime promoted fluidity of the soil and formation of glassy waste products. Because of its relatively coarse particle size, the dolomitic lime was not fed successfully through the feeder that was devised and built at Clemson University. The hydrated lime fed well.

Analysis of the offgas streams revealed very high particulate content and very low O₂ content. The high particulate content is attributed to a combination of melt agitation caused by the electric arc, and by highly reducing conditions at the melt surface caused by use of graphite electrodes. Despite the presence of reducing conditions, the processed soil products formed appeared to be glassy with little or no visible crystalline phases.

No accurate estimate of operating costs could be made during this study because of the limited size and batch mode of operation. Subsequent to these tests, an additional modification was made to the melter which allowed feeding an additional 25 kg of soil into the melter during operation. This doubled the batch size, with no additional power input, and caused the mode of energy input to change from the transferred arc mode to a joule heating mode as the ends of both electrodes became submerged in the melt. The study conducted for DOE/WSRC revealed much useful information about the performance of the DC arc melter. However, subsequent runs conducted by ESE students performing MS degree thesis research indicate that a great deal remains to be learned about more efficiently and effectively using this technology.

INTRODUCTION

The Department of Energy is currently evaluating methods of treatment for disposal of hazardous and mixed wastes. Preferred methods of treatment include those technologies that are capable of consistently producing a durable, leach resistant product, while simultaneously minimizing disposal volumes. The selected technology must be demonstrated over a range of waste characteristics, including compositions, chemistries, moistures, and physical characteristics.

Vitrification has been selected as a technology that is capable of producing a highly durable product, while minimizing disposal volumes through organic destruction, moisture evaporation, and porosity reduction. In this study *in-situ* vitrification of soil using a graphite electrode DC arc melter was simulated. 25 kg batches of soil were charged into the melter's graphite crucible and the response of these batches to treatment by the melter was determined. To promote glass formation it was decided to operate the melter in the presence of air, and to introduce fluxing aides into the soil. Blending of the fluxes with the soil, while ensuring a uniform distribution of the flux, would not be representative of field operating conditions. The desired approach for making flux additions was injection through a hollow electrode while the melter was in operation. It was felt that this approach would come closest to simulating operation of a concentric electrode, non-transferred arc melter which, purportedly, would permit flux injection through the electrode. No such electrodes were available so Clemson University designed two geometries for evaluation. These electrodes were used to inject fluxes and air into the soil and the vitrification response of the soil was assessed.

EXPERIMENTAL PROCEDURE

Description of the DC Arc Melter

The Mark I-W DC Arc melter was designed by Electro-Pyrolysis, Inc., (EPI) of Wayne, PA. It was purchased by the Westinghouse Savannah River Company (WSRC) and installed at the DOE/Industrial Vitrification Laboratory at Clemson University. The Mark I-W water-cooled melter is the successor to the Mark I melter operated at the Massachusetts Institute of Technology. Promising results from the Mark I Melter prompted the fabrication and further testing with the Mark I-W.

The melter currently has the capability to process 1 ft³ of feed in batch mode only. The Mark I-W DC arc melter, shown in Figure 1, is comprised of the following systems, which are described in more detail below.

- Furnace Assembly
- Electrode Assembly
- Offgas System
- Water Cooling System

- Nitrogen Purge System
- Electrical Power Supply

Figure 1. EPI DC Arc Melter, as received

Furnace Assembly

The Mark I-W furnace features a removable, stainless steel, lower chamber and a stainless steel, fixed upper roof. The lower chamber can be raised or lowered to mate with the roof by a screw-driven mechanism. When in the lowered position, the chamber can be rolled away from the fixed roof assembly. When the components are bolted together the combined height is 43½ inches with an OD of 37½ inches. Both the lower chamber and the roof have a water-cooled shell.

A cutaway view of the chamber section is shown in Figure 2. The lower chamber contains two nested graphite crucibles. The area between the outer crucible and the water-cooled shell is lined with fiberboard insulation and refractory bricks. Nitrogen is supplied through a dispersion tube at the bottom of the chamber. The outer graphite crucible is connected to the positive side of the power supply by graphite rods at four locations. The inner crucible rests in direct contact with the outer crucible and acts as the positive electrode for the melt. A graphite disk with a hole in the center is placed on the bottom of the inner crucible. This disk allows starting electrodes of various sizes to be placed inside the inner

crucible. The graphite components and nitrogen purge promote a highly reducing atmosphere in the melter during operation.

Figure 2. Cutaway view of melting chamber.

Electrode Assembly

The electrode superstructure houses the movable upper electrode and is mounted on top of the furnace roof. The movable upper electrode is mounted to the electrode carriage, which is raised and lowered by a screw-driven mechanism. The height of the electrode is measured by a position indicator. The superstructure is subject to a nitrogen purge during operation to keep oxygen out of this superstructure. Negative voltage from the power supply is applied to the upper graphite electrode.

To create the arc, the movable upper electrode is lowered to contact the lower starter electrode. As current is passed through the electrodes, the upper electrode is raised and a DC plasma arc is initiated. The arc is sustained until the material is melted. Once the run is complete, the power is turned off and the melter is cooled. When a melt has cooled sufficiently, the inner crucible is lifted out and inverted to allow the contents to be removed.

Offgas System

The offgas system consists of the following major pieces of equipment:

- Quench Chamber
- Induced Draft Blower
- Gas Flow Meter

Figure 1 shows the location of the major components of the offgas system as it was received. The nitrogen and gases evolved from processing the wastes exit the melt chamber through a 4" ID port through the chamber roof section. From there, the gases enter a cyclone where larger particulate matter is to be removed. Offgas then enters the quench chamber where it is cooled prior to entering a water-jet venturi scrubber. This scrubber uses a jet of water to induce a flow of gas through the venturi and provide water droplets for the collection of particles.

The scrubber is followed by a separation tank which separates the gases and the scrub water. The water supply to the scrubber flows into a polypropylene holding tank. Water is pumped from this tank, filtered, and returned to the scrubber.

The gases are then drawn through an explosion proof induced draft blower. The blower provides the additional suction to draw the gases through the equipment. Following the blower, the gases pass through a mass flow meter, and are exhausted to the atmosphere.

The offgas system was modified to accommodate sampling during the runs. These modifications are discussed later.

Water Cooling System

Many components of the Mark I-W furnace are water cooled:

- Lower Chamber Shell
- Upper Chamber Shell
- Lower Chamber Electrode Connections
- Power Supply Transformer
- Recirculation Tank

A total of 20 gpm flow is used to keep the system cool.

Nitrogen Purge System

Nitrogen is typically used as a purge gas during the melt and cool-down periods. The reducing atmosphere promoted by the inert gas purge prolongs the life of the graphite electrodes and crucible as well as decreasing the volume of offgas that would be evolved under an oxidizing atmosphere. In the as-received system, nitrogen was introduced into the melter area through a dispersion tube at the bottom of the chamber and through the upper electrode housing.

Electrical Power Supply

The power supply was manufactured by the Halmar Robicon Group. In this supply, 480V, 3 phase, 60 Hz power is chopped and then transformed to 1000 Hz using power transistors. This high frequency power is then rectified and filtered to provide DC current.

The power at 100% output is 133.3V and 750A. The power supply is current controlled, and current is set with a manual potentiometer.

Melter Operation

After charging and closing the melter, it was purged with air at a flowrate of ~6 scfm through one of the ports in the melter lid. Subsequently, the electrodes were brought into contact and the melter preheated by maintaining the voltage and current at ~10-15 volts and ~200 amps (~2-3 kW) for about 20 minutes. After preheating, the electrodes were separated and the power increased to ~14 kW (35 V and 400 A). It was attempted to maintain these operating conditions for the duration of the run.

Upon completion of the run, the melter was allowed to cool overnight. The crucible was removed from the melter the next day and the contents of the crucible were dumped from it.

Equipment Modifications

Flux Feeder Design and Operation

The flux feeding system consisted of a feed bottle used to fluidize the flux, a flexible stainless steel hose used to transport the fluidized flux, an electrical isolator through which the flexible hose entered the melter and a hollow electrode, described later, which transported the fluidized flux to the melt. The feed bottle was composed of a one gallon Nalgene Bottle with an air nozzle mounted in the bottom and a connection in the top to which the flexible hose was connected as shown in Figure 3.

The electrical isolator is shown in Figure 4. The flexible hose connected to a 0.65 inch hole in the side of the hollow electrode and was secured to the electrode with stainless steel wire. The portion of the flexible hose inside the headspace was wrapped with kaolinite wool fire blanket to

Figure 3. Feed Bottle

Figure 4. Electrical Isolator

provide protection from heat and molten splatter. A compressed air line with a 0-5 scfm flowmeter in-line was used to blow air into the feed bottle nozzle. Air flow rates of 1/2, 1, 1-1/2, 2, 3 and 4 scfm were tried. It was found that a flow of 1-1/2 scfm was sufficient to fluidize the flux and that higher flow rates were unnecessary. Starting with a bottle completely filled with flux, it took one to two minutes to feed approximately two-thirds of the flux. The last third of the bottle would not feed with the bottle upright since the flux level approached the top of the nozzle. The bottle was blown down to this level and then refilled. After the last refill, the flux remaining in the bottom of the bottle was fed by turning the bottle upside down several times and shaking. All of the flux was fed in by half of the total run time on runs 6, 7 and 8.

Hollow Electrode Design

Demonstration of *in-situ* vitrification is not accurately modeled by preblending fluxes and soil. In these studies it was desired to inject the flux at the arc location through a hollow electrode. Since this melter had not been previously operated with a hollow electrode, and none were available for this use, two concepts were developed by Clemson University and tested during this project.

The hollow electrodes used for feeding flux were UCAR grade CS extruded graphite machined to three inches in diameter. The hollow electrodes used in runs 2 and 3 (the second and third experimental runs in this project) had a one inch hole bored down the center through which air and flux was blown.

See Figure 5 for details of this electrode design. This design was modified for runs 4 through 8 to allow air and flux to exit

from the sides of the electrode rather than from the bottom. The center hole was plugged and a pair of one inch holes were drilled in the sides of the electrode as shown in Figure 6. The holes were drilled at an angle to guide the air and flux toward the melt and to prevent flux from accumulating inside the electrode. The electrodes were also shortened by 1-1/2 inches. The flexible hose installed in the 0.65 inch hole limits the upward travel of the electrode. Reducing the distance from the 0.65 inch hole to the bottom of the electrode allowed more electrode travel, which gives greater flexibility in controlling the arc.

Off-gas System Changes

The off-gas system was modified by removing the cyclone and installing sample ports for EPA method 5 and gas chromatograph sampling. The cyclone was ineffective due to the low flow rates in the off-gas system. Details of the off-gas modifications are shown in Figure 7.

Figure 5. Hollow Electrode Design

Figure 6. Modified Electrode Design.

Figure 7. Offgas system modifications made to the EPI melter for this study.

Purge Gas System Modifications

A compressed air line with a 0-6 scfm flowmeter in-line was connected to a threaded hole in the 4" blank flange adjacent to the electrical isolator to allow the addition of air into the headspace. See Figure 4.

Video Camera

A water-cooled Combustion Tec lens assembly two foot in length passes through the chamber roof and records the arc vitrification process by means of a color video camera equipped with filters to decrease the intensity of the light.

Description of the Soils

Two Savannah River Site soil types were used in this project; a sandy soil and a clay soil. The compositions investigated in this study were the sandy soil and a 50:50 mix of the clay and sandy soils. The compositions of the two soils types are shown in Table I.

Table I Normalized SRS Clay and Sandy Soil Compositions

Values are in Weight % (ADS #67186&67187)

Oxide	Clay Soil	Sandy Soil
Al ₂ O ₃	4.310	0.733
B ₂ O ₃	0.014	0.014
BaO	0.006	0.006
CaO	0.094	0.021
CdO	0.006	0.006
Cr ₂ O ₃	0.016	0.015
Fe ₂ O ₃	1.730	0.580
LaO	0.004	0.004
MgO	0.014	0.002
MnO	0.004	0.004
Na ₂ O	0.342	0.337
NiO	0.034	0.034
P ₂ O ₅	0.077	0.060
PbO	0.035	0.034
SiO ₂	93.017	97.977
SrO	0.003	0.003
TiO ₂	0.236	0.147
ZnO	0.004	0.004
ZrO ₂	0.056	0.020
Total	100.000	100.000

Crucible Studies

Crucible Study Run Plan

The crucible study investigated the effects of 3 different flux types on the two SRS soils (clay and sandy). In this study the clay soil contained 93% SiO₂ and the sandy soil contained 98% SiO₂, with the remaining component being primarily Al₂O₃. Based on

these soil compositions, the WSRC Task Order Representative selected the CaO-Al₂O₃-SiO₂ system for investigation, with the Ca to be added as the flux.

The three fluxes considered were (1) hydrated lime [Ca(OH)₂], (2) dolomitic, or pelletized, lime (a Ca-Mg compound) and (3) colemanite. Colemanite is a naturally occurring mineral comprised mainly of B₂O₃ and CaO [(CaO)₂(B₂O₃)₃•5H₂O)]. In addition to its CaO content, colemanite was of interest because of boron's ability to capture neutrons liberated during radioactive decay.

The crucibles used for this study were B&B Refractors #6 assay crucibles. These are an alumina-silicate composition which have very good thermal shock resistance and a temperature rating of 1700°C for their intended use. Experimentation has shown a life expectancy of between four and six hours at temperatures between 1400 and 1600°C when used in glass studies of the CaO-Al₂O₃-SiO₂ ternary system.

The crucible study consisted of 23 trials, 16 selected initially and an additional 7 added to clarify some issues that arose during the originally planned trials. These trials included the two soils and flux additions ranging from 5% to 35%. Table II identifies the actual variations evaluated.

LOD's and LOI's were determined for each of the soils and fluxes to account for losses of water and CO₂, and transformations to the stable oxide phase. Oxide-basis calculations were then made and the appropriate amounts of each constituent were mixed to prepare the crucible study batches. LOD/LOI results, calculated batch chemistries and amounts weighed are shown in Table III.

The prepared batches were placed in crucibles which were then placed in the Deltech bottom loading furnace at a temperature of 1550°C. This temperature was chosen based on the combination of furnace capability, crucible life expectancy, and temperatures on the respective phase diagrams in the regions of interest. After stabilizing at 1550°C, the crucible remained in the furnace for 3.5 hours. At the end of the 3.5 hour hold, the melt was visually examined for fluidity. Then the crucible was cooled at a rate of 2°C /minute. While *in-situ* cooling of the soil might be expected to occur at a slower rate, the 2°C /minute rate was chosen because it more closely simulated the cooling rate of the EPI melter and was expected to be slow enough to allow devitrification of unstable glasses.

Table II. Crucible Study Experimental Matrix.

<i>Clay Soil</i>			
Weight % Flux	Colemanite	Hydrated Lime	Dolomitic Lime
5	✓	✓	
10	✓	✓	
15	✓	✓	
20	✓	✓	
30	✓		
<i>Sandy Soil</i>			
Weight % Flux	Colemanite	Hydrated Lime	Dolomitic Lime
5	✓	✓	
10	✓	✓	
15	✓	✓	
20	✓	✓	
25		✓	
30		✓	✓
35	✓	✓	✓

When the Deltech reached a temperature of 1100°C during cooling, the crucible was quickly transferred to a Lindberg Blue-M furnace at the same temperature. The cool down cycle continued until a temperature of 100°C was reached. This interrupted cycle was used because a single furnace capable of performing the entire cycle was not available. The Deltech's elements are prone to embrittlement if cooled below 850°C, and the Blue-M has an upper temperature limit of 1200°C. The transfer also allowed the Deltech to be reheated and stabilized for the next run, while the cool-down cycle was being completed in the Blue-M. The total crucible study cycle, including the hold at 1550°C and the cool down to 100°C, was approximately 15.6 hours, (3.5 hours at 1550°C + 12.1 hours cooling).

After the crucible cooled, it was broken open and a visual observation was made to determine whether a glass or non-glassy phase had been made. Candidate compositions for processing in the arc melter were chosen based on the appearance of the phase formed.

Table III. LOD/LOI results, calculated batch chemistries and amounts weighed

LOD LOI	Fractional Weight Remaining				
	0.97792774	1		0.88905325	0.9490554
	0.63786659	0.75683646		0.97537438	0.99373742
	COLEMANITE	HYDRATED LIME	DOLOMITIC LIME	W.S.R.C CLAY	W.S.R.C SANDY
Crucible ID	Weight of each component added to yield 150 gm (Oxide Basis)				
HS-C-C05	12.03			164.34	
HS-C-C10	24.05			155.69	
HS-C-C15	36.07			147.04	
HS-C-C20	48.10			138.39	
HS-S-C05	12.03				151.10
HS-S-C10	24.05				143.16
HS-S-C15	36.07				135.18
HS-S-C20	48.09				127.24
HS-C-HL05		9.91		164.32	
HS-C-HL10		19.82		155.67	
HS-C-HL15		29.73		147.04	
HS-C-HL20		39.65		138.41	
HS-S-HL05		9.90			151.10
HS-S-HL10		19.82			143.13
HS-S-HL15		29.72			135.20
HS-S-HL20		29.65			127.25
Test Series Interrupted, New Values Determined					
LOD LOI	Fractional Weight Remaining				
	1	1	0.996229	1	1
	0.740747087	0.756431674	0.561042	0.891916303	0.967989757
	COLEMANITE	HYDRATED LIME	DOLOMITIC LIME	W.S.R.C CLAY	W.S.R.C SANDY
Crucible ID	Weight of each component added to yield 150 gm (Oxide Basis)				
HS-C-C30	60.76			117.73	
HS-S-C35	70.86				100.73
HS-S-HL25		49.48			116.21
HS-S-HL30		59.50			108.49
HS-S-HL35		69.42			100.72
HS-S-DL30			80.55		108.48
HS-S-DL35			93.94		100.79

Arc Melter Runs

Arc melter runs were conducted according to the test matrix shown in Table IV. In all cases, the targeted product mass was 20 kg. The total amount of feed charged into the crucible varied depending on LOD and LOI values. All of the batches contained 0.5% each of cerium and copper as surrogate radionuclide materials. Prior to charging the crucible, the lower electrode was shielded using a hollow tube. This prevented dirt from falling onto the lower electrode, but allowed passage of the upper electrode. Soil was then charged into the crucible. An example of this set-up is shown in Figure 8. The bottom electrode is shown in the center of the figure, protected by a tube made from grafoil. The outer, circularly arranged black platelets are grafoil sheets positioned to protect the upper chamfered portion of the crucible. Not visible in this figure is the grafoil liner between the soil and the crucible wall.

Figure 8. Typical Crucible Set-up

To assist soil melting, a small blend containing flux and soil was prepared and placed around the base of the bottom electrode. This was done to promote formation of a molten glass pool early in the run, thereby minimizing the chance of the arc damaging the graphite crucible.

Runs were conducted by flowing air at 5 scfm into the headspace of the melter for 10 minutes prior to applying power to the electrodes. After the 10 minute purge, a low level of power was applied to allow heat-up of the electrodes. During this time, the analytical equipment used to record the progress of the run was given a final check. Finally, after an additional 10 minutes the arc was struck and the run was begun.

Table IV. EPI Melter Run Matrix

Run #	ID	Soil	Debris	Flux	Flux Loading	Feed Method	Electrode Geometry
1	014	50:50	No	Colemanite	25%	Blended	Solid
2	015	→	→	→	→	Injected	Hollow - Bottom Opening
3	016					Blended	”
4	017			Dolomitic Lime		Injected	Hollow - Side Openings
5	018			”		Blended	→
6	020	Hydrated Lime	Injected				
7	021	Sandy	→	→	→	→	
8	022	50:50					

Data Acquisition

Gas Analyses

Sample Gas Conditioning

Figure 9 shows the flow diagram for conditioning the sample gases. In this system, a continuous gas sample is passed through a mini-cyclone and two filters to remove particulate matter. The primary filter is configured in a parallel arrangement with an identical filter. This allows the primary filter to be changed during sampling without disrupting continuous sampling and measurement. The secondary filter is a back-up to the primary filter. Shake-down experiments have not resulted in the collection of particulate matter on the secondary filter.

The offgases are drawn through the particulate matter controls by a vacuum pump. The vacuum pump then passes the gas sample into a Genie membrane filter to remove moisture. Moisture is further removed by a Perma-Pure co-axial drying tube. The gases to be sampled are passed through the inner shell of the Perma-Pure drier while warm, dry nitrogen is passed countercurrent to the gas sample through the outer shell. The inner and outer shells are separated by a naphion membrane which allows moisture in the gas sample to be collected by the warm, dry nitrogen. As a last precaution the gas sample is passed through a 0.5 micron filter before being introduced into the analytical instruments.

Gas Chromatograph Analysis

A steady, representative sample of the offgases was introduced into a MTI™ M200 gas chromatograph. Data acquisition is obtained using MTI™'s EZ Chrome 200 v. 3.5.8 software. The gas chromatograph is capable of continuous on-line sampling, configured to cycle every 2½ minutes. The characteristics of the gas chromatograph are shown in Table V.

The gas chromatograph is capable of measuring all of these gases in any range, as long as the gas chromatograph is calibrated within the expected range of measurement. Previous experience with the gas chromatograph applied to the offgas from the DC arc melter indicates only H₂, O₂, N₂, CO, CO₂, and CH₄ are detectable with this particular instrument.

EPA Method 5 Sampling

Off-gases were sampled starting at the beginning of each run for particulate matter and moisture content using a sampling scheme based on EPA Reference Method 5 (40 CFR 60). An off-gas sample was drawn through a 1/2" stainless steel sample probe at approximately isokinetic conditions, then through a heated probe assembly, into a heated

glass fiber filter and finally through a series of ice cooled impingers. The first impinger was an empty, modified Greenburg-Smith (straight center tube and no impinger plate).

Figure 9. Flow Diagram for Sample Gas Conditioning

Table V Characteristics of gas chromatograph.

Column A	Column B
Molecular Sieve 5A (MS5A)	HeySep R (HSR)
H ₂ - Hydrogen	O ₂ /N ₂ - Air Composite
O ₂ - Oxygen	CH ₄ - Methane
N ₂ - Nitrogen	CO ₂ - Carbon Dioxide
CH ₄ - Methane	C ₂ H ₆ - Ethane
CO - Carbon Monoxide	C ₃ H ₆ - Propane
Carrier Gas - Argon	Carrier Gas - Helium

The second impinger was a modified Greenburg-Smith containing 100 ml of distilled water. The third impinger was a standard Greenburg-Smith (tapered tip and impinger plate) containing 100 ml of distilled water. The final impinger was a modified Greenburg-Smith containing 200-300 g of silica gel.

RESULTS and DISCUSSION

Crucible Studies

Crucibles containing the soil and flux combination of interest were placed in the kiln at 1550°C, allowed to remain at temperature for 3.5 hours and then cooled at 2°C/min to 1100°C. At this time the crucible was transferred to a lower temperature kiln, where the same cooling rate was continued down to 100°C. The following coding designation was used to differentiate between the various samples.

HS-x-yyzz;

- where HS indicates the humid soils project,
- x indicates soil type, (S = sandy or C= clay)
- yy is flux type, (C=colemanite, HL=hydrated lime, or DL=dolomitic lime), and
- zz is flux level (wt % on an oxide basis)

So, for example, the code HS-S-HL05 indicates Humid Soils-Sandy soil-Hydrated Lime at 5%.

Assuming that the contribution of other impurities can be ignored, the Savannah River soils and hydrated lime mixtures can be described by the CaO-Al₂O₃-SiO₂ phase diagram, shown in Figure 10. If colemanite (~50:50 CaO:B₂O₃) is the flux being considered, it is convenient to ignore the minor impact of the Al₂O₃ and use the CaO-B₂O₃-SiO₂ phase diagram to represent the soil-flux blends (Figure 11). Using this simplified approach, the soils processed using up to 20% hydrated lime as the flux would not be expected to show any melting at 1550°C. Conversely, all of the compositions processed with colemanite would be expected to melt at 1550°C.

The results obtained with flux additions up to 20% agreed reasonably well with the results predicted by the phase diagrams. The sandy soil, with its low Al₂O₃ content, showed no melting when hydrated lime was the flux. The only reaction which did occur was sintering of the soil in the crucibles.

The clay soil-hydrated lime mixtures showed evidence of melting with flux additions of 15% or more. This melting response may have been influenced by the presence of ~2% iron in the soil. Even though melting occurred, some of the reaction product was non-vitreous and not sufficiently fluid to be poured. Consequently, it was decided to investigate higher flux loadings using the higher melting point sandy soil.

Figure 10. CaO-Al₂O₃-SiO₂ phase diagram showing compositional changes associated with hydrated lime additions.

Additional studies at 25, 30 and 35% hydrated lime in sandy soil resulted in formation of a pourable fluid which formed a glass on cooling. At 25 and 30% hydrated lime the glass was a milky white color. At 35% hydrated lime, the glass took on a light blue tint as well.

In addition to hydrated lime, two crucibles of sandy soil were prepared with 30 and 35% dolomitic lime. In addition to CaO, dolomitic lime contains ~28% MgO. The dolomitic lime was evaluated because of its fine size, thereby making injection into the melt through the electrode easier than with the pelletized hydrated lime.

Both loadings of dolomitic lime created a fluid glass. However, a pellet of the 30% dolomitic lime glass was not poured because the crucible was destroyed and the glass leaked through a hole eaten through the crucible bottom.

The glass formed with 35% dolomitic lime was a milky white color tinted with blue.

The crucible study results, summarized in Table VI, revealed that a flux loading of <25% would not result in melting of soil at 1550°C. Since the DC arc creates temperatures considerably greater than 1550 °C, a flux loading of 25% was believed to be adequate for soil processing.

Figure 11. CaO- B₂O₃-SiO₂ phase diagram showing compositional changes associated with colemanite additions.

Table VI. Crucible study results for the initial 16 scheduled trials:

(1550°C/3.5 hours)

Batch	Results
<i>Clay soil - Colemanite flux</i>	
HS-C-C05	Sintered soil
HS-C-C10	Some opaque pale green glass with large unreacted layer on top-trace of brown glass
HS-C-C15	More opaque pale green glass with unreacted layer on top-trace of brown glass
HS-C-C20	Large amount of opaque green glass with smaller unreacted layer on top-trace of brown glass
HS-C-C30	Not pourable. Formed same opaque pale green glass as earlier colemanite runs
<i>Sandy soil - Colemanite flux</i>	
HS-S-C05	Sintered sand
HS-S-C10	Sintered sand
HS-S-C15	Sintered sand- some distinct opaque green glass phase
HS-S-C20	Foamy green tinted white glass-ceramic- some distinct opaque green glass phase
HS-S-C35	Not pourable. Formed same opaque pale green glass as earlier colemanite runs
<i>Clay soil - Hydrated lime flux</i>	
HS-C-HL05	Sintered soil
HS-C-HL10	Sintered soil
HS-C-HL15	Sintered soil/ceramic
HS-C-HL20	Pale green glass-ceramic
<i>Sandy soil - Hydrated lime flux</i>	
HS-S-HL05 to HL20	Sintered sand
HS-S-HL25	Fluid, poured pellet, crucible contents and pellet both formed milky white glass upon cooling
HS-S-HL30	Fluid, poured pellet, crucible contents and pellet both formed milky white glass upon cooling
HS-S-HL35	Fluid, poured pellet, crucible contents and pellet both formed milky white glass with a light blue tint upon cooling
<i>Sandy soil - Dolomitic lime flux</i>	
HS-S-DL30	Crucible was removed from kiln due to failure. A fluid glass had formed, but ate a hole through the crucible bottom resulting in loss of melt
HS-S-DL35	Fluid, poured pellet, crucible contents and pellet both formed heavily blue tinted milky white glass

Arc Melter Runs

Arc melter run results are summarized in Table VII. Additional details regarding compositions and electrode consumption are contained in Appendices A and B, respectively. The runs are discussed below.

Run 1 (ID# 014)

The melter was charged with a preblended batch consisting of equal parts of sandy and clay-type soils (37 wt % each), 25% colemanite as the flux, and 0.5% each of CuO and Ce_2O_3 . The total weight of the batch was 24.26 kg, calculated to produce 20 kg of glass.

After charging and closing the melter, it was purged with air at a flowrate of ~6 scfm through one of the ports in the melter lid. The arc was established and the melter preheated by maintaining the voltage and current at ~10-15 volts and ~200 amps (~2-3 kW) for about 20 minutes. Subsequently, the electrodes were separated and the power increased to ~14 kW (35 V and 400 A). These operating conditions were maintained for approximately an additional 80 minutes (100 minutes cumulative run time) for a total power input of 29 kWh as measured by a power meter on the melter.

As the power was increased, there was an immediate increase in CO_2 emissions, coupled with a corresponding decrease in O_2 present in the offgas, see Figure 12. Continuous monitoring of the offgas with a gas chromatograph showed that the O_2 level decreased from an initial value of ~21% to <1% ninety-three minutes into the run. At the same time, CO_2 increased from ~0.3-0.4% up to slightly greater than 16%. During this time the N_2 and H_2 contents varied from 74.4 to 78.6% and 0 to 1.1%, respectively.

After ~93 minutes of operation, the particulate analysis sample train became clogged and it was decided to install a second train for another analysis. To prevent escape of the offgas into the lab during the sample train change out, power to the electrodes was reduced to ~2-3 kW. Opening the offgas system allowed air ingress into the system, with the result that O_2 level increased and CO_2 level decreased at the gas chromatograph. The increase in O_2 and decrease in CO_2 does not necessarily reflect changes in reactions taking place in the melter since the opening in the offgas system is beyond the melter. A graphical presentation of the changes observed in gas composition is shown in Figure 12. Tabulated data from all the runs are contained in Appendix C.

Table VII. Arc Melter Run Summary

Run/ Trial	Feed	Mass of Feed [kg]	Mass of Product [kg]	Expected Mass of Product [kg]	Power Input [kWh]	Description
1/014	Sandy Soil, Clay Soil, and Colemanite	24.26 kg	Trial not completed	20.00	29	Melt froze during probe swapping. Electrode broke at threads. Arced to head space.
2/015	Sandy Soil, Clay Soil, and Colemanite	23.39 kg	Trial not completed	20.02	9	Used hollow electrode to introduce flux. Interfered with arc stability causing melt to freeze during the trial. Mauve glass formed in the arc zone.
3/016	Sandy Soil, Clay Soil, and Colemanite	23.37 kg	Trial not completed	20.00	34	Melt froze during probe swapping. Formed a bowl due to viscosity of the melt.
4/017	Sandy Soil, Clay Soil, and Dolomitic Limestone	25.31 kg	Trial not completed	20.00	22	Electrode feed system clogged preventing fluxes from being added. Melt froze while attempting to introduce flux through plenum.
5/018	Sandy Soil, Clay Soil, and Dolomitic Limestone	25.30 kg	17.68	19.99	70	Used modified hollow electrode configuration to blow air through the electrode. Produced a gray product.
6/020	Sandy Soil, Clay Soil, and Hydrated Lime	23.22 kg	18.65	20.00	70	Used modified hollow electrode to blow air and hydrated lime into molten pool. Produced a homogeneous, light gray product. Unusual build-up of dust on crucible bevel.
7/021	Sandy Soil and Hydrated Lime	22.67 kg	15.16	20.00	64	Used modified hollow electrode to blow air and hydrated lime into molten pool. Produced a homogeneous, medium gray product. Unusual build-up of dust on crucible bevel.
8/022	Sandy Soil, Hydrated Lime, and Debris	23.212	14.92 glass 4.14 unmelted batch 19.06 total	20.00	70	Used modified hollow electrode to blow air and hydrated lime into molten pool. Produced a homogeneous blue/lavender product. Some unmelted feed near the edges. Unusual build-up of dust on crucible bevel.

Figure 12 Offgas composition changes observed during Run #1

The second sample train was installed after approximately 10 minutes and the electrodes were separated to reestablish the previous operating conditions of ~14 kW. Although unknown at the time, the melt that had been created at the bottom of the melter solidified around the electrodes during the time that the power level had been reduced. When an attempt was made to separate the electrodes, the upper electrode broke at the point where it threaded into the extension. When the arc was reestablished, it occurred between the top of the broken electrode and the bottom of the extension, about midway along the thickness of the refractory in the lid of the melter. This situation was realized when molten refractory was seen dripping from the top of the melter while the melter operation was being observed through the video camera. The run was terminated immediately.

After disassembling the melter, it was discovered that establishing the arc at the electrode breakage point resulted in destruction of some of the refractory in the melter roof. This refractory melted and ran down the electrode, giving it the appearance of a candle with heavy wax build-up along its length. This and the bowl-shaped sintered charge are shown in Figure 13(a). Figure 13(b) shows that fusion of the soil was limited to a fairly narrow region between the top and bottom electrodes, forming a small amount of glass. The glass was medium gray in color with swirls of lighter gray. Outside the arc zone, the remaining soil was sintered, creating a bowl-shaped mass, but showed no evidence of melting.

Run 2 (ID #015)

After repairing the damaged melter roof, a batch containing a 50:50 mixture of sandy soil and clay soil, plus 0.5% each of copper and cerium, was prepared. 25% colemanite flux was to be injected into the melt through a hollow electrode. Start up of the run proceeded similarly to run #1. After the arc was stabilized, injection of the colemanite flux was begun. It was observed that the arc was very unstable while the flux and air were being injected through the hollow electrode. Several times the arc extinguished but could be reignited. During one attempt at reigniting the arc, the electrode stuck in the glass at the bottom of the crucible and broke. The run was terminated after 9 kWh vs a target of 80 kWh. Figure 14 shows the sintered batch after removal from the crucible. Once again, the electrode broke at the base of the threaded stub. A small quantity of mauve colored glass was formed during this run. Permanent gas analysis results are shown in Figure 15 and Appendix C.

(a) Sintered mass created when charge cooled during sample train change-out. Also shown is melter refractory that ran down electrode.

(b) Example of small fusion zone created in the sintered mass between the electrodes. Also note damage at top of electrode caused by arcing.

Figure 13. Product Formed During Run

Figure 14. Product From Run 2.

Figure 15. Offgas composition changes observed during Run #2.

Run 3 (ID #016)

This run also consisted of a 50:50 blend of sandy and clay soil, but with the colemanite flux blended into the soil rather than being injected as in Run 2. After stabilizing the arc, only air was injected into the melter through the hollow electrode. The purpose of this test was to determine if the previously observed arc instability was caused by the injected flux, or by the injection of air alone. Arc instability was reduced greatly during this test, confirming that arc instability occurred when flux flowed between the two electrodes.

During the run the off gas particulate sampling train again became clogged. Once again an attempt was made to change it out, but within a greatly reduced time frame. Even though the change out required only about 1 minute, the electrode froze in the melt. The run was terminated after 34 kWh. A small quantity of gray glass was produced during the run. Permanent gas analysis results are shown in Figure 17 (see next page) and Appendix C.

Post-run examination of the upper electrode revealed that one side eroded much faster than the other. This is attributed to a slight misalignment of the upper and lower electrodes. Figure 16, although not clearly showing the side-to-side erosion, does provide an indication of the damage realized by the electrode due to injecting air through it. The base of the electrode is rounded and the central hole is enlarged. During this run 758 gms (18.1%) of the top electrode was destroyed. (2.9%/kWh)

Figure 16. Example of Electrode Damage Occurring During Air Injection.

Figure 17. Offgas composition changes observed during Run #3.

Run 4 (ID #017)

The modified electrode, shown in Figure 6 and discussed previously, was used during this run for flux injection. Dolomitic lime was selected in favor of colemanite because of the high viscosity of the colemanite flux melts. The batch was a 50:50 mixture of sandy and clay soil, with 0.5% Cu and Ce radionuclide surrogate materials. The pelletized lime would not feed into the melter and eventually clogged the feed system. The run was terminated after 22 kWh. A small quantity of gray glass was produced during the run. Permanent gas analysis results are shown in Figure 19 (see next page) and Appendix C.

Run 5 (ID #018)

This run also consisted of a 50:50 blend of sandy and clay soil with Cu and Ce radionuclide surrogate additions, but with the dolomitic lime flux blended into the soil rather than being injected as in Run 4. After stabilizing the arc, only air was injected into the melter through the modified hollow electrode. The purpose of this test was to determine the effect of using dolomitic lime as the flux.

This run was successfully terminated after 70 kWh with 17.68 kg of gray glass produced compared to a predicted 19.99 kg. Permanent gas analysis results are shown in Figure 20 and Appendix C.

Severe erosion of the electrode air exit holes was observed at the completion of this run. As shown in Figure 18, the left side of the electrode shows nearly complete erosion between the front and rear air exit holes. This electrode experienced 766 gms weight loss (21.2% total loss or 0.3%/kWh.)

Figure 18. Damage Experienced by the Modified Electrode Caused by Air Injected into the Melt during Run 5.

Figure 19. Offgas composition changes observed during Run #4.

Figure 20. Offgas composition changes observed during Run #5.

Figure 20. Offgas composition changes observed during Run #5.(cont.)

This run demonstrated that soil could be processed to a glassy form in the EPI melter by preblending the flux into the soil and injecting air through the electrode. However, the form of the flux was not suitable for the feeding mechanism that was fabricated for this project and the ability of flux additions through the electrode had not been demonstrated. The last three runs were conducted with finer size, hydrated lime.

Run 6 (ID #020)

The batch was a 50:50 mixture of sandy soil and clay soil, with 0.5% Cu and Ce radionuclide surrogate additions. 25% hydrated lime flux was injected into the melt through a modified hollow electrode. The purpose of this run was to attempt injection of the finer particle size lime flux into the melt. This run progressed smoothly until terminated by the operator at 70 kWh. Permanent gas analysis results are shown in Figure 22 (see next page) and Appendix C.

A homogeneous, light gray, glassy product weighing 18.65 kg was formed and is shown in Figure 21. (The predicted recovery weight was 20 kg.) One unusual feature of this run was the copious amount of white particulate matter that was present on the bevel of the crucible after the run, shown in Figure 23. This build-up is attributed to the fineness of the flux; it is believed that some of the flux was carried away from the surface of the melt and was deposited on the crucible bevel.

Figure 21. Homogeneous Glassy Product Produced During Run 6.

Figure 22. Offgas Composition Changes Observed During Run #6.

Figure 22. Offgas Composition Changes Observed During Run #6. (cont.)

Figure 23. Flux Build-up on Rim of Crucible.

Run 7 (ID #021)

This batch was composed solely of sandy soil with 25% hydrated lime flux to be injected into the melt through a modified hollow electrode. The purpose of this run was to verify that the higher melting point sandy soil could also be successfully processed using the approach used for the sandy and clay soil mix. This run also progressed smoothly and was terminated by the operator after 64 kWh. A homogeneous, medium gray, glassy product weighing 15.16 kg was formed. (The predicted recovery weight was 20 kg.) Permanent gas analysis results are shown in Figure 24 and Appendix C. This run also showed a build-up of particulate matter on the crucible bevel.

Run 8 (ID #022)

This batch was also sandy soil, but simulated pieces of debris were placed on the top of the batch, as shown in Figure 25. These consisted of an 884 gm, 0.25" thick stainless steel disk, a 152 gm piece of threaded rod, and a 1,430 gm piece of fiberglass reinforced concrete. 25% hydrated lime flux (based on charge weight exclusive of debris) was injected into the soil during the run using a modified electrode. This run progressed smoothly, producing 14.92 kg of a blue/lavender glass (vs 20 kg calculated) after 70 kWh. The metallic debris melted during the run and settled at the bottom of the crucible where it reacted with the bottom plate. (The bottom plate is a sacrificial graphite disk used to protect the crucible.) 4.14 kg of unmelted batch material remained near the edges of the melt, resulting in 19.06 kg of material being recovered after the run.

Figure 24. Offgas Composition Changes Observed During Run #7.

Figure 24. Offgas Composition Changes Observed During Run #7. (cont.)

Figure 25. Placement of debris for Run 8. Note that access to the bottom electrode is provided by a rectangular box.

Permanent gas analysis results are shown in Figure 26 and Appendix C. This run also showed a build-up of particulate matter on the crucible bevel.

Particulate analysis

Although the intended sample time was one hour, the actual sample time was less due to the filter becoming clogged with particulate matter. The following results were obtained:

Table VIII Particulate analysis result summarized for all runs

Run/ Trial	Sample Duration (minutes)	Particulate Concentration (gm/m³)	Moisture Content (% by standard volume)
1/014	31.0	28.4	11.4
2/015	54.2	15.5	10.1
3/016	25.0	34.6	12.8
4/017			
5/018	21.9	9.0	7.9
6/020	26.1	22.9	23.4
7/021	19.7	28.5	15.0
8/022	30.0	34.5	21.5

Figure 26. Offgas composition changes observed during Run #8.

Figure 26. Offgas composition changes observed during Run #8. (cont.)

Electrode Consumption

Complete details of electrode weight loss are contained in Appendix . In summary, approximately equal upper electrode loss, as measured by weight % loss per kWh, was observed for both the hollow (central hole) and modified (side holes) electrodes. This result is somewhat surprising given the increased surface area for air passage in the modified electrode, and the aggressive wear observed around the air exit holes during run 5 as shown in Figure 18. A review of the electrode wear data reveals that the hollow electrode suffers severe length loss compared to the modified electrode. While the hollow electrode lost an average of 0.6 inches/kWh, the modified electrode lost only 0.014"/kWh on average.

CONCLUSIONS

- Soil can be processed into a glassy, macroscopically homogeneous products in the graphite electrode, DC arc melter if air is injected into the melter during the run.
- Flux injection through a hollow (central hole) electrode results in frequent extinguishing of the arc.
- Flux injection through a modified (side holes) electrode does not extinguish the arc.
- Dolomitic lime was found to be the best flux for these trials based on its ability to be fed through the electrode without clogging, and its ability to enhance processing of the soil.
- Colemanite is not an acceptable flux for runs done under the conditions used in this study because it does not lower the viscosity of the melt or promote melting sufficiently to allow processing.
- lime used as a flux was adequate to promote processing of the soil for the conditions used in this study.
- When using lime as a flux, 70kWh energy input was sufficient for essentially complete processing.
- In terms of weight % loss per kWh of power input to the melter, electrode geometry (central hole vs side holes) did not exert an influence on electrode wear.
- To attain essentially complete processing of the soils studied in this project typically required 4.2 kWh/kg of glass produced.
- Processing atmosphere within the vessel was very reducing despite injection of air during the run.

- The processing atmosphere within the melter was very reducing despite air injection through the electrode. This resulted in offgas particulate matter emissions several orders of magnitude higher than typically observed within a joule-heated melter.

RECOMMENDATIONS

The graphite electrode, DC arc melter is capable of very high operating temperatures and, as a result, can consume a wide variety of objects that would not be destroyed in a traditional joule-heated melter. The reducing atmosphere that exists in the melter allows pyrolysis of high organic and debris-containing wastes without incineration. A subsequent transition to joule heating and an oxidizing atmosphere allows control over a wide range of melting temperatures and provides wide latitude for formation of stable glasses. Extinguishing the arc also quiets the melt pool, and is expected to diminish the release of particulate matter and potentially volatile constituents. Based on these observations, the following actions are recommended.

- Evaluate alternate electrodes, such as refractory metal or water non-consumable electrodes, to allow greater control over the reducing conditions existing in the melter.
- Further evaluate the transition from transferred arc to joule heating mode as a means of destroying debris and high organic materials followed by creation of a stable glass.
- Improve the offgas system to accommodate the potential creation of hazardous and flammable organic compounds.
- Explore the glass composition range that would be allowed by the wide temperature capability of the melter.
- Use the melter to do treatability studies on actual mixed waste streams.

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RECORDS

This report is based on data contained in the following record books.

<u>Record Book</u>	<u>Pages</u>
AY	76-78, 80, 83-87, 89-90, 96
CG	1-3, 7-15, 17-37, 41-54.
AX	19, 20, 29-39
BW	77-93
CI	1-16
CE	39, 40

Appendix A

Feed Compositions

Trial	Soil	Other	Flux	Total
014	8.26 kg SRS Clay Soil 7.72 kg SRS Sandy Soil	0.173 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.253 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$	8.02 kg Colemanite	24.26 kg
015	8.32 kg SRS Clay Soil 7.62 kg SRS Sandy Soil	0.172 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.262 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$	7.02 kg Colemanite	23.394 kg
016	8.32 kg SRS Clay Soil 7.60 kg SRS Sandy Soil	0.172 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.262 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$	7.02 kg Colemanite	23.374 kg
017	8.32 kg SRS Clay Soil 7.60 kg SRS Sandy Soil	0.170 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.262 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$	8.96 kg Dolomitic Limestone	25.312 kg
018	8.30 kg SRS Clay Soil 7.60 kg SRS Sandy Soil	0.172 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.267 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$	8.96 kg Dolomitic Limestone	25.299 kg
020	8.28 kg SRS Clay Soil 7.76 kg SRS Sandy Soil	0.177 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.261 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$	6.74 kg $\text{Ca}(\text{OH})_2$	23.218 kg
021	15.50 kg SRS Sandy Soil	0.174 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.256 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$	6.74 kg $\text{Ca}(\text{OH})_2$	22.670 kg
022	8.30 kg SRS Clay Soil 7.74 kg SRS Sandy Soil	0.174 kg $\text{Ce}_2(\text{CO}_3)_3$ 0.258 kg $\text{Cu}(\text{C}_2\text{H}_3\text{O}_2)_2$ 0.884 kg steel disk 0.152 kg threaded rod 1.430 kg fiberglass reinforced concrete	6.74 kg $\text{Ca}(\text{OH})_2$	23.212 kg

Appendix B

Electrode Consumption

Trial	Upper Electrode Before	Upper Electrode After	Upper Electrode Loss	Lower Electrode Before	Lower Electrode After	Lower Electrode Loss
014 Diameter Length Mass 29 kWh	Standard 3" 21.0" 4.267 kg	3" 19.5" 3.804kg 0.052"/ kWh	 1.50" 0.463 kg	2" 3.5" 0.351 kg	2" NA NA	Run halted.
015 Diameter Length Mass 9 kWh	Hollow 3" 22.0" NA	3" 22.0" 4.403 kg	 0" NA	2" 3.5" 0.347 kg	2" NA NA	Two upper electrodes used. Both snapped. Run Halted
016 Diameter Length Mass 34 kWh	Hollow 3" 22.0" 4.178 kg	3" 19.375" 3.420kg 0.07"/ kWh	 2.625" 0.758 kg	2" 2.375" 0.247 kg	2" 0" 0 kg	Lower Electrode entirely consumed. 2.375" 0.247 kg
017 Diameter Length Mass 22 kWh	Modified 3" 18.875" 3.540kg	3" NA NA	 NA NA	2" 3.0" 0.317 kg	2" NA NA	Feed System Clogged. Run Halted. NA NA
018 Diameter Length Mass 70 kWh	Modified 3" 20.375" 3.619 kg	3" 19.75" 2.853 kg 0.009"/ kWh	 0.625" 0.766 kg	2" 2.0" 0.229 kg	2" 0" 0.072 kg	2.0" 0.157 kg

Trial	Upper Electrode Before	Upper Electrode After	Loss	Lower Electrode Before	Lower Electrode After	Loss
020 Diameter Length Mass 70 kWh	Modified 3" 16.875" 3.039 kg	3" 16.125" 2.339 kg 0.011"/ kWh	 0.75" 0.7 kg	2" 2.438" 0.554 kg	2" 0.75" 0.090 kg	 1.688" 0.464 kg
021 Diameter Length Mass 64 kWh	Modified 3" 20.563" 3.742 kg	3" 19.75" 3.036 kg 0.013"/ kWh	 0.813" 0.706kg	2" 3.313" 0.335 kg	2" 1.0" 0.135 kg	 2.313" 0.20 kg
022 Diameter Length Mass 70 kWh	Modified 3" 20.563" 3.727 kg	3" 19.0" 2.757 kg 0.022"/ kWh	 1.563" 0.970kg	2" 2.875" 0.296kg	2" 0" 0kg	 2.875" 0.296 kg

Appendix C

Permanent Gas Analysis Results (Runs 1-8)

Permanent Gas
Analyses
Run #1 4/9/96

Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO
1	2.5	2.5	0.094	20.946	78.324	0.350	No
2	2.5	5.0	0.010	20.574	78.644	0.330	No
3	2.5	7.5	0.003	20.540	78.418	0.290	No
4	2.5	10.0	0.002	20.625	78.494	0.285	No
5	2.5	12.5	0.001	20.743	78.456	0.276	No
6	2.5	15.0	0.001	20.803	78.281	0.260	No
7	2.5	17.5	0.001	20.815	78.236	0.213	No
8	2.5	20.0	0.000	20.821	78.138	0.272	No
9	2.5	22.5	0.000	20.837	78.113	0.177	No
10	2.5	25.0	0.210	17.275	77.860	2.891	No
11	2.5	27.5	0.262	15.786	77.257	4.390	Yes
12	2.5	30.0	1.133	18.194	75.374	2.209	Yes
13	2.5	32.5	0.566	15.848	76.219	4.416	Yes
14	2.5	35.0	0.318	15.736	76.428	4.930	Yes
15	2.5	37.5	0.447	16.424	75.547	4.549	Yes
16	2.5	40.0	0.279	15.684	75.193	5.836	Yes
17	2.833	42.8	0.279	15.158	74.779	6.515	Yes
18	2.5	45.3	0.313	13.945	74.636	7.639	Yes
19	2.5	47.8	0.225	11.875	74.593	9.556	Yes
20	3.333	51.2	0.137	10.813	74.406	11.228	No
21	3.0	54.2	0.159	7.970	75.038	12.616	No
22	3.0	57.2	0.044	6.187	75.935	13.708	No
23	3.0	60.2	0.085	5.200	76.380	13.898	No
24	3.0	63.2	0.034	4.608	76.422	14.286	No
25	3.0	66.2	0.014	4.835	76.590	14.361	No
26	3.0	69.2	0.012	4.341	76.564	14.456	No
27	3.0	72.2	0.037	2.932	77.098	14.923	No
28	3.0	75.2	0.039	2.480	77.035	15.479	No
29	3.0	78.2	0.059	2.439	77.198	15.141	No
30	3.0	81.2	0.201	1.273	76.919	16.170	No
31	3.0	84.2	0.254	1.160	76.541	16.378	No
32	3.0	87.2	0.102	1.996	76.516	16.230	No
33	3.0	90.2	0.502	1.231	76.314	16.134	No
34	3.0	93.2	0.861	0.798	75.363	16.211	Yes
35	3.0	96.2	0.511	4.155	75.525	14.096	No

Permanent Gas
Analyses
Run #1 4/9/96

Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO
36	3.0	99.2	0.159	12.907	76.402	7.366	No
37	3.0	102.2	0.086	16.555	76.902	4.182	No
38	3.0	105.2	0.083	17.616	76.990	3.088	No
39	3.0	108.2	0.089	18.100	77.083	2.487	No
40	3.0	111.2	0.097	17.922	77.137	2.578	No
41	3.0	114.2	0.124	18.019	77.094	2.515	No
42	3.0	117.2	0.172	17.628	77.102	2.812	No
43	3.0	120.2	0.278	17.597	76.879	2.785	No
44	3.0	123.2	0.312	18.071	76.643	2.114	No
45	3.0	126.2	0.352	18.202	76.530	2.012	No
46	3.0	129.2	0.454	18.079	76.460	2.176	No

Run #2 Raw Data	4/19/96						
Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	Total % Vol.
1	3	3.0	0.121	19.868	79.562	0.172	99.723
2	3	6.0	0.018	12.628	87.629	0.052	100.327
3	3	9.0	0.006	11.606	88.557	0.031	100.2
4	3	12.0	0.003	10.896	89.488	0.027	100.414
5	3	15.0	0.003	13.335	86.535	0.031	99.904
6	3	18.0	0.001	13.784	86.091	0.031	99.907
7	3	21.0	0.328	12.505	86.134	0.575	99.542
8	3	24.0	0.273	12.972	85.353	0.65	99.248
9	3	27.0	0.603	13.195	84.293	0.887	98.978
10	3	30.0	1.03	12.662	84.361	1.359	99.412
11	3	33.0	0.566	12.511	84.306	1.746	99.129
12	3	36.0	0.335	10.955	84.906	3.303	99.499
13	3	39.0	0.231	8.55	85.562	5.423	99.766
14	3	42.0	0.158	10.872	83.242	5.547	99.819
15	3	45.0	0.204	7.705	85.637	6.243	99.789
16	3	48.0	0.195	6.423	84.739	8.626	99.983
17	3	51.0	0.092	6.736	83.886	9.388	100.102
18	3	54.0	0.047	7.133	84.942	7.69	99.812
19	3	57.0	0.035	9.338	83.618	6.723	99.714
20	3	60.0	0.035	7.689	85.062	7.152	99.938
21	3	63.0	0.027	7.248	84.236	7.393	98.904
22	3	66.0	0.099	8.677	82.695	7.448	98.919
23	3	69.0	0.119	10.794	83.339	4.308	98.56
24	3	72.0	0.082	12.465	84.826	1.406	98.779
25	3	75.0	0.063	13.02	84.984	0.786	98.853

Run #3 Raw Data	4/23/96						
Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	Total % Vol.
1	3	3.0	0.006	17.101	80.075	0.057	97.239
2	3	6.0	0.004	16.755	80.233	0.057	97.049
3	3	9.0	0.003	15.632	81.163	0.037	96.835
4	3	12.0	0.002	14.421	82.47	0.033	96.926
5	3	15.0	0.001	13.583	83.243	0.03	96.857
6	3	18.0	0.001	13.744	82.963	0.03	96.738
7	3	21.0	0.141	11.811	83.266	1.142	96.36
8	3	24.0	0.299	10.792	82.209	2.617	95.917
9	3	27.0	0.43	10.631	81.471	3.17	95.702
10	3	30.0	0.575	9.363	81.18	4.241	95.359
11	3	33.0	0.339	8.901	81.655	4.995	95.89
12	3	36.0	0.165	8.628	81.541	5.889	96.223
13	3	39.0	0.067	6.451	81.736	8.144	96.398
14	3	42.0	0.03	3.278	80.896	12.543	96.747
15	3	45.0	0.079	2.775	80.183	13.799	96.836
16	3	48.0	0.294	1.023	79.759	15.153	96.229
17	3	51.0	0.299	0.642	79.251	16.294	96.486
18	3	54.0	1.833	0.425	77.373	14.484	94.115
19	3	57.0	2.598	0.208	76.586	12.816	92.208
20	3	60.0	2.857	0.222	75.809	12.765	91.653
21	3	63.0	2.546	0.383	77.705	12.612	93.246
22	3	66.0	2.88	0.525	77.527	11.736	92.668
23	3	69.0	1.665	0.361	79.347	13.101	94.474
24	3	72.0	2.072	0.254	78.134	13.765	94.225
25	3	75.0	2.014	0.163	77.748	13.848	93.773
26	3	78.0	1.389	0.185	78.158	14.89	94.622
27	3	81.0	1.254	0.291	78.92	14.282	94.747
28	3	84.0	0.524	1.129	80.132	14.369	96.154
29	3	87.0	0.431	2.667	80.447	13.193	96.738
30	3	90.0	0.324	1.662	80.466	13.957	96.409
31	3	93.0	0.086	2.609	80.623	14.078	97.396
32	3	96.0	0.053	2.839	80.651	13.849	97.392
33	3	99.0	0.026	3.631	80.849	13.009	97.515
34	3	102.0	0.885	1.388	78.207	13.81	94.29
35	3	105.0	0.714	0.564	77.966	15.049	94.293
36	3	108.0	0.374	2.308	80.116	13.309	96.107
37	3	111.0	0.437	1.91	81.03	12.782	96.159

Run #3	4/23/96						
Raw Data							
Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	Total % Vol.
38	3	114.0	0.209	3.226	80.714	12.381	96.53
39	3	117.0	0.061	7.999	81.65	7.263	96.973
40	3	120.0	0.043	10.299	82.183	3.999	96.524
41	3	123.0	0.042	11.749	81.839	2.708	96.338
42	3	126.0	0.046	12.269	81.98	1.984	96.279
43	3	129.0	0.049	11.783	83.02	1.533	96.385
44	3	132.0	0.043	13.082	81.918	1.187	96.23
45	3	135.0	0.038	16.192	78.71	0.926	95.866
46	3	138.0	0.037	18.432	77.39	0.885	96.744

Run #4	4/23/96							
Raw Data								
Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
39	2.5	97.5	0.058	13.915	82.506	1.991	0.118	98.588
40	2.5	100.0	0.048	14.428	82.579	1.442	0.04	98.537
41	2.5	102.5	0.046	14.255	82.992	1.169	0.032	98.494
42	2.5	105.0	0.039	14.392	83.037	0.94	0.024	98.432

Run #5 Raw Data	4/30/96							
Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
1	2.5	2.5	0.151	20.764	78.058	0.197	0.000	99.17
2	2.5	5.0	0.012	20.901	78.095	0.043	0.000	99.051
3	2.5	7.5	0.000	20.963	78.228	0.034	0.000	99.225
4	2.5	10.0	0.000	19.172	79.719	0.106	0.000	98.997
5	2.5	12.5	0.000	17.235	81.719	0.078	0.000	99.032
6	2.5	15.0	0.000	16.988	81.784	0.056	0.000	98.828
7	2.5	17.5	0.000	16.843	81.940	0.052	0.000	98.835
8	2.5	20.0	0.000	16.620	81.601	0.050	0.000	98.271
9	2.5	22.5	0.003	16.073	82.106	0.054	0.000	98.236
10	2.5	25.0	0.000	15.902	82.322	0.046	0.000	98.27
11	2.5	27.5	0.168	14.334	80.394	2.536	0.572	98.004
12	2.5	30.0	0.201	14.516	80.409	1.817	0.708	97.651
13	2.5	32.5	0.444	12.453	77.486	5.474	1.991	97.848
14	2.5	35.0	0.344	11.779	76.158	7.617	2.079	97.977
15	2.5	37.5	0.148	11.716	77.863	7.395	0.797	97.919
16	2.5	40.0	0.078	10.644	77.831	8.958	0.408	97.919
17	2.5	42.5	0.100	8.937	74.048	14.096	0.987	98.168
18	2.5	45.0	0.177	7.277	70.477	18.743	1.485	98.159
19	2.5	47.5	0.091	6.352	71.123	19.234	0.769	97.569
20	2.5	50.0	0.037	7.298	73.595	16.911	0.233	98.074
21	2.5	52.5	0.049	6.645	72.839	17.387	0.395	97.315
22	2.5	55.0	0.036	6.052	72.894	18.543	0.326	97.851
23	2.5	57.5	0.012	6.784	75.128	16.212	0.110	98.246
24	2.5	60.0	0.009	6.893	75.751	15.281	0.074	98.008
25	2.5	62.5	0.005	7.983	76.390	13.552	0.049	97.979
26	2.5	65.0	0.003	7.720	75.260	14.794	0.036	97.813
27	2.5	67.5	0.004	7.570	74.603	15.681	0.057	97.915
28	2.5	70.0	0.003	7.625	71.834	18.396	0.073	97.931
29	2.5	72.5	0.002	7.619	72.495	17.714	0.061	97.891
30	2.5	75.0	0.002	8.105	73.594	16.110	0.057	97.868
31	2.5	77.5	0.001	8.159	73.275	16.218	0.045	97.698
32	2.5	80.0	0.002	8.091	73.729	16.213	0.050	98.085
33	2.5	82.5	0.000	19.539	76.412	1.221	0.000	97.172
34	2.5	85.0	0.000	20.419	76.558	0.116	0.000	97.093
35	2.5	87.5	0.000	13.338	75.495	8.133	0.028	96.994
36	2.5	90.0	0.000	9.252	74.647	12.987	0.039	96.925
37	2.5	92.5	0.000	7.724	74.018	15.472	0.038	97.252

Run #5	4/30/96							
Raw								
Data								
Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
38	2.5	95.0	0.000	6.743	71.635	19.045	0.038	97.461
39	2.5	97.5	0.000	6.959	72.477	18.139	0.028	97.603
40	2.5	100.0	0.000	5.592	71.546	19.518	0.027	96.683
41	2.5	102.5	0.000	5.197	71.183	20.285	0.028	96.693
42	2.5	105.0	0.000	4.918	70.370	21.455	0.000	96.743
43	2.5	107.5	0.000	5.300	70.951	20.343	0.000	96.594
44	2.5	110.0	0.000	6.483	72.619	17.951	0.019	97.072
45	2.5	112.5	0.000	6.655	74.541	15.891	0.021	97.108
46	2.5	115.0	0.000	6.790	75.425	14.685	0.017	96.917
47	2.5	117.5	0.000	6.267	75.831	14.747	0.000	96.845
48	2.5	120.0	0.000	5.849	75.541	15.333	0.000	96.723
49	2.5	122.5	0.000	17.845	76.391	2.946	0.000	97.182
50	2.5	125.0	0.000	20.329	76.460	0.161	0.000	96.95
51	2.5	127.5	0.000	20.364	76.356	0.088	0.000	96.808
52	2.5	130.0	0.000	17.077	75.977	2.694	0.000	95.748
53	2.5	132.5	0.000	7.110	76.426	12.568	0.000	96.104
54	2.5	135.0	0.000	5.759	76.835	13.456	0.000	96.05

Trial 020 5/9/96

Raw Data Run #6

Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
1.000	2.500	2.500	0.027	17.687	81.569	0.045	0.000	99.328
2.000	2.500	5.000	0.006	17.513	81.408	0.035	0.000	98.962
3.000	2.500	7.500	0.002	17.078	81.612	0.036	0.000	98.728
4.000	2.500	10.000	0.001	16.949	81.828	0.033	0.000	98.811
5.000	2.500	12.500	0.000	16.946	81.959	0.034	0.000	98.939
6.000	2.500	15.000	0.070	16.384	82.276	0.124	0.030	98.884
7.000	2.500	17.500	0.092	15.617	81.984	0.588	0.162	98.443
8.000	2.500	20.000	1.314	9.462	81.001	4.995	1.983	98.755
9.000	2.500	22.500	0.384	12.445	81.732	3.479	0.338	98.378
10.000	2.500	25.000	0.236	7.890	81.937	7.984	0.456	98.503
11.000	2.500	27.500	0.180	8.736	81.558	7.611	0.205	98.290
12.000	2.500	30.000	0.115	7.214	81.430	9.459	0.183	98.401
13.000	2.500	32.500	0.071	6.419	81.938	9.964	0.142	98.534
14.000	2.500	35.000	0.097	6.051	81.410	10.741	0.215	98.514
15.000	2.500	37.500	0.113	6.905	81.046	9.815	0.241	98.120
16.000	2.500	40.000	0.392	7.095	83.022	6.891	0.358	97.758
17.000	2.500	42.500	0.251	6.523	82.080	8.808	0.434	98.096
18.000	2.500	45.000	0.055	5.797	81.846	10.735	0.097	98.530
19.000	2.500	47.500	0.250	5.283	81.174	11.049	0.353	98.109
20.000	2.500	50.000	0.064	5.167	80.727	12.200	0.178	98.336
21.000	2.500	52.500	0.004	17.849	78.434	2.185	0.000	98.472
22.000	2.500	55.000	0.001	20.719	77.639	0.165	0.000	98.524
23.000	2.500	57.500	0.000	20.812	77.523	0.089	0.000	98.424
24.000	2.500	60.000	0.394	14.057	80.415	2.977	0.149	97.992
25.000	2.500	62.500	0.824	11.186	82.260	2.308	0.809	97.387
26.000	2.500	65.000	0.128	10.769	83.330	3.497	0.122	97.846
27.000	2.500	67.500	0.133	10.023	83.078	4.893	0.036	98.163
28.000	2.500	70.000	0.612	10.917	81.147	4.250	0.609	97.535
29.000	2.500	72.500	1.068	11.575	82.044	1.450	0.993	97.130
30.000	2.500	75.000	0.371	12.019	81.451	2.967	0.431	97.239
31.000	2.500	77.500	0.049	11.284	81.847	4.722	0.021	97.923
32.000	2.500	80.000	0.009	9.551	82.206	6.625	0.000	98.391
33.000	2.500	82.500	0.003	8.847	81.716	7.858	0.000	98.424
34.000	2.500	85.000	0.002	8.091	81.604	8.912	0.000	98.609
35.000	2.500	87.500	0.001	6.703	80.334	11.168	0.000	98.206
36.000	2.500	90.000	0.001	7.084	79.648	11.427	0.000	98.160
37.000	2.500	92.500	0.000	6.439	80.224	11.594	0.000	98.257
38.000	2.500	95.000	0.000	5.497	79.976	12.469	0.000	97.942

Trial 020 5/9/96

Raw Data **Run #6**

Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
39.000	2.500	97.500	0.000	5.112	79.118	13.423	0.000	97.653
40.000	2.500	100.000	0.000	16.439	77.759	3.925	0.000	98.123
41.000	2.500	102.500	0.000	20.366	77.001	0.304	0.000	97.671
42.000	2.500	105.000	0.000	14.710	77.783	4.892	0.000	97.385
43.000	2.500	107.500	0.000	6.030	79.569	12.070	0.000	97.669
44.000	2.500	110.000	0.000	4.954	79.646	12.848	0.000	97.448
45.000	2.500	112.500	0.000	4.717	79.939	13.173	0.000	97.829
46.000	2.500	115.000	0.000	3.632	80.006	13.703	0.000	97.341
47.000	2.500	117.500	0.000	3.765	79.724	13.634	0.000	97.123
48.000	2.500	120.000	0.000	3.634	79.907	13.616	0.000	97.157
49.000	2.500	122.500	0.000	3.052	79.703	14.025	0.000	96.780
50.000	2.500	125.000	0.000	2.949	79.641	14.240	0.000	96.830
51.000	2.500	127.500	0.000	3.057	81.152	14.364	0.000	98.573
52.000	2.500	130.000	0.000	19.930	77.917	0.748	0.000	98.595
53.000	2.500	132.500	0.000	20.789	77.659	0.107	0.000	98.555
54.000	2.500	135.000	0.000	20.765	77.413	0.081	0.000	98.259
55.000	2.500	137.500	0.000	15.521	77.848	3.812	0.000	97.181

Trial 021 5/10/96

Raw Data Run #7

Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
1	2.67	2.67	0.000	21.181	79.327	0.049	0.000	100.557
2	2.67	5.34	0.000	21.164	79.257	0.054	0.000	100.475
3	2.67	8.01	0.000	21.139	79.153	0.048	0.000	100.340
4	2.67	10.68	0.000	17.041	83.842	0.031	0.000	100.914
5	2.67	13.35	0.000	17.324	83.418	0.037	0.000	100.779
6	2.67	16.02	0.000	17.337	83.374	0.036	0.000	100.747
7	2.67	18.69	0.000	16.864	83.824	0.031	0.000	100.719
8	2.67	21.36	0.000	17.056	83.691	0.035	0.000	100.782
9	2.67	24.03	0.619	7.381	83.556	8.733	1.738	102.027
10	2.67	26.70	0.255	11.495	83.629	5.242	0.664	101.285
11	2.67	29.37	0.602	10.053	83.921	6.275	0.764	101.615
12	2.67	32.04	0.150	11.571	83.900	5.950	0.147	101.718
13	2.67	34.71	0.096	11.511	83.669	6.385	0.054	101.715
14	2.67	37.38	0.100	11.998	83.278	6.243	0.056	101.675
15	2.67	40.05	0.040	11.378	83.465	6.766	0.000	101.649
16	2.67	42.72	0.166	13.316	84.024	3.239	0.201	100.946
17	2.67	45.39	0.030	12.421	82.436	6.298	0.037	101.222
18	2.67	48.06	0.027	10.918	83.672	6.759	0.041	101.417
19	2.67	50.73	0.027	10.771	83.701	6.910	0.000	101.409
20	2.67	53.40	0.009	9.532	83.222	8.829	0.039	101.631
21	2.67	56.07	0.003	18.546	79.182	1.646	0.000	99.377
22	2.67	58.74	0.050	3.626	83.663	15.718	0.167	103.224
23	2.67	61.41	0.006	18.398	78.714	2.289	0.000	99.407
24	2.67	64.08	0.002	19.518	78.434	1.211	0.000	99.165
25	2.67	66.75	0.042	19.644	78.238	1.045	0.057	99.026
26	2.67	69.42	0.016	19.819	78.495	0.717	0.000	99.047
27	2.67	72.09	0.000	19.967	78.268	0.724	0.000	98.959
28	2.67	74.76	0.020	2.614	85.888	14.343	0.162	103.027
29	2.67	77.43	0.017	3.810	84.522	14.377	0.190	102.916
30	2.67	80.10	3.421	3.622	79.312	13.689	2.998	103.042
31	2.67	82.77	0.001	6.019	84.620	11.594	0.000	102.234
32	2.67	85.44	0.000	7.204	83.047	11.852	0.000	102.103
33	2.67	88.11	0.000	8.742	83.420	9.502	0.000	101.664
34	2.67	90.78	4.207	6.020	77.777	11.779	2.394	102.177
35	2.67	93.45	0.003	10.233	83.193	7.866	0.000	101.295
36	2.67	96.12	0.000	5.568	84.217	12.701	0.000	102.486
37	2.67	98.79	0.000	4.659	84.321	13.675	0.000	102.655
38	2.67	101.46	0.741	3.894	82.449	14.143	0.663	101.890

Trial 021 5/10/96

Raw Data **Run #7**

Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
39	2.67	104.13	0.000	9.728	82.628	8.553	0.000	100.909
40	2.67	106.80	0.008	7.008	83.689	10.358	0.000	101.063
41	2.67	109.47	0.000	11.759	82.822	5.774	0.000	100.355
42	2.67	112.14	0.000	11.238	82.816	6.312	0.000	100.366
43	2.67	114.81	0.000	9.954	83.040	7.925	0.000	100.919
44	2.67	117.48	0.000	9.065	82.035	10.021	0.000	101.121
45	2.67	120.15	0.000	7.216	82.181	12.137	0.000	101.534
46	2.67	122.82	0.000	7.360	81.270	12.843	0.000	101.473
47	2.67	125.49	0.000	4.501	81.710	15.992	0.000	102.203
48	2.67	128.16	0.000	5.169	82.196	14.639	0.000	102.004
49	2.67	130.83	0.000	6.461	81.649	13.529	0.000	101.639
50	2.67	133.50	0.000	7.046	82.101	12.408	0.000	101.555
51	2.67	136.17	0.001	10.259	79.958	9.938	0.000	100.156
52	2.67	138.84	0.001	20.246	77.590	0.452	0.000	98.289
53	2.67	141.51	0.001	20.912	78.506	0.084	0.000	99.503
54	2.67	144.18	0.001	20.942	78.629	0.067	0.000	99.639
55	2.67	146.85	0.001	20.941	78.570	0.069	0.000	99.581

Trial 022 5/13/96

Raw Data Run #8

Sample	Cycle Time (min)	Run Time (min)	H ₂ % Vol.	O ₂ % Vol.	N ₂ % Vol.	CO ₂ % Vol.	CO % Vol.	Total % Vol.
39	2.67	104.13	0.000	21.322	79.654	0.039	0.000	101.015
40	2.67	106.80	0.000	21.328	79.586	0.044	0.000	100.958
41	2.67	109.47	0.000	19.349	79.422	1.337	0.000	100.108
42	2.67	112.14	0.000	21.011	78.576	0.069	0.000	99.656
43	2.67	114.81	0.000	21.058	78.701	0.047	0.000	99.806
44	2.67	117.48	0.000	21.128	78.865	0.045	0.000	100.038
45	2.67	120.15	0.000	21.349	79.646	0.035	0.000	101.030
46	2.67	122.82	0.000	11.258	83.417	8.320	0.000	102.995
47	2.67	125.49	0.000	11.379	83.310	8.234	0.000	102.923
48	2.67	128.16	0.000	11.969	83.012	8.000	0.000	102.981
49	2.67	130.83	0.000	13.190	82.468	7.162	0.000	102.820
50	2.67	133.50	0.000	13.647	82.372	6.665	0.000	102.684
51	2.67	136.17	0.000	13.727	82.036	6.808	0.000	102.571
52	2.67	138.84	0.000	13.117	82.115	7.492	0.000	102.724
53	2.67	141.51	0.000	14.417	81.598	6.457	0.000	102.472
54	2.67	144.18	0.000	16.265	80.794	4.978	0.000	102.037
55	2.67	146.85	0.000	20.219	79.843	1.188	0.000	101.250
56	2.67	149.52	0.000	13.733	81.264	7.791	0.000	102.788
57	2.67	152.19	0.000	21.363	79.714	0.072	0.000	101.149
58	2.67	154.86	0.000	21.400	79.857	0.048	0.000	101.305
59	2.67	157.53	0.000	21.382	79.971	0.040	0.000	101.393