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## **Crucible Melts and Bench-Scale ISV Tests on Simulated Wastes in INEL Soils**

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**May 1990**

**Prepared for the U.S. Department of Energy  
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest Laboratory  
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BATTELLE MEMORIAL INSTITUTE  
*for the*  
UNITED STATES DEPARTMENT OF ENERGY  
*under Contract DE-AC06-76RLO 1830*

Printed in the United States of America

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PNL-7344  
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CRUCIBLE MELTS AND BENCH-SCALE  
ISV TESTS ON SIMULATED WASTES  
IN INEL SOILS

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## SUMMARY

This report summarizes the results of eight crucible melt tests and three bench-scale in situ vitrification (ISV) tests that were performed on simulated metals/soil mixtures containing actual site soils from the Idaho National Engineering Laboratory (INEL). The crucible melt and bench-scale ISV tests are a part of efforts by the Pacific Northwest Laboratory (PNL) to assist the INEL in conducting a treatability study on ISV for application to the mixed waste buried at the INEL subsurface disposal area (SDA). The crucible melt tests were performed to evaluate the effect of various chemical additives and metal oxidation techniques on soil melting temperatures, melt viscosities, metals versus electrode oxidation potentials, and metals incorporation in the glass. The bench-scale ISV tests were performed to supplement the existing ISV data base with information on certain hazardous materials that have not been adequately evaluated in previous ISV tests. These materials included five EP toxicity metals (arsenic, barium, cadmium, chromium, and silver), various volatile organic materials fixed in a cementitious matrix [including carbon tetrachloride ( $CCl_4$ ), trichloroethylene (TCE), and tetrachloroethylene (PCE)], and asbestos. In addition, the bench-scale tests were used to evaluate the effect of the proposed chemical additive (from crucible melt testing) on ISV processing performance and product quality.

Results from the crucible melt tests indicate that sodium tetraborate ( $Na_2B_4O_7$ ) was the preferred chemical additive for reducing melt viscosity. No void volumes were present in a crucible melt of the INEL metals/soil mixture with 11.6 wt%  $Na_2B_4O_7$ , indicating that the viscosity reduction was sufficient to allow off gases within the melt to escape to the melt surface without excessive void formation. None of the chemical additives tested in the crucible melts actually resulted in enhanced metal incorporation in the glass matrix, however.

Process performance results from bench-scale ISV testing indicate that the presence of metals in the melt can lead to potential processing concerns such as electrode shorting, decreased maximum power inputs (due to equipment limitations), increased downward melting rates (at the expense of horizontal

melt growth), void formations in the vitrified block, and increased energy-to-mass ratios for the soils. Most of these concerns are only concerns on soils with a large enough amount of metal to produce a full or partial short at the bottom of the vitrified melt during processing. As a result, it is expected that these concerns may be eliminated with the implementation of electrode feeding, since the electrode locations in relation to these shorts are controlled by the test operator. Penetrometer measurements of the molten glass surface during the first bench-scale test showed that the melt surface was soft enough to permit electrode insertions.

The bench-scale tests showed that the addition of sodium tetraborate did not eliminate the large void areas in the vitrified block. Instead of being caused by the highly viscous INEL soil, it appears that the voids are primarily due to the presence of a high metals content in the soil. Sodium tetraborate also affects ISV processing adversely by limiting the maximum power input, which effectively increases the energy-to-mass ratio and limits achievable depth. As a result, the addition of sodium tetraborate is not recommended for ISV processing of INEL soils.

Analytical results from the second bench-scale ISV test indicate that portions of the cadmium and arsenic were removed from the melt during ISV processing. All of the other EP Toxicity metals added to the second bench-scale test (barium, chromium, and silver) were completely incorporated in the vitrified block. Cadmium and traces of arsenic were also found in the crusty soils within 1 in. from the bottom edge of the second bench-scale melt. Because of the relatively low boiling points of cadmium (765°C) and arsenic (613°C), it is thought that these materials may volatilize before they are encapsulated by the vitrified melt. However, it is expected that the cadmium and arsenic would have eventually been incorporated by either the vitrified melt or the ISV off-gas system, if ISV processing had continued to a greater depth.

Analytical results from the third bench-scale ISV test detected the presence of trace amounts of PCE and (to a lesser extent) TCE in the surrounding soils, less than 5 cm (2 in.) away from the vitrified block. However, the concentration of PCE was less than 1 ppm and the concentration of TCE was less

than 0.1 ppm. This is 10,000 to 100,000 times less than the PCE and TCE that were added to the original cemented sludge/grease mixture. The trace amounts of PCE and TCE in the surrounding soils are not considered to be problems from a hazardous waste standpoint.

The second and third ISV bench-scale tests produced high-quality vitrified masses that passed the EP Toxicity and TCLP tests. In addition, microscopic tests revealed that the asbestos added to the second bench-scale ISV test was completely incorporated in the vitrified mass.



## CONTENTS

SUMMARY . . . . .	iii
1.0 INTRODUCTION . . . . .	1.1
2.0 CONCLUSIONS . . . . .	2.1
2.1 CRUCIBLE MELT TESTING . . . . .	2.1
2.2 BENCH-SCALE ISV TESTING . . . . .	2.1
3.0 RECOMMENDATIONS . . . . .	3.1
4.0 PROCESS DESCRIPTION AND STATUS OF DEVELOPMENT . . . . .	4.1
5.0 INEL CRUCIBLE TESTS . . . . .	5.1
5.1 CHEMICAL METHODS AND EXPERIMENTAL APPROACH . . . . .	5.1
5.1.1 Moist Aeration . . . . .	5.2
5.1.2 Sodium Nitrate Addition . . . . .	5.3
5.1.3 Viscosity Control of the Melt . . . . .	5.4
5.2 RESULTS AND DISCUSSION . . . . .	5.4
6.0 INEL BENCH-SCALE ISV TESTS . . . . .	6.1
6.1 SOIL CHARACTERIZATIONS . . . . .	6.1
6.2 ISV BENCH-SCALE TEST SETUP . . . . .	6.1
6.3 BENCH-SCALE TEST 1 . . . . .	6.4
6.3.1 Test Configuration . . . . .	6.4
6.3.2 Performance Evaluation . . . . .	6.6
6.3.3 Product Quality . . . . .	6.9
6.4 BENCH-SCALE TEST 2 . . . . .	6.10
6.4.1 Test Configuration . . . . .	6.10
6.4.2 Performance Evaluation . . . . .	6.14
6.4.3 Product Quality . . . . .	6.18

6.5 BENCH-SCALE TEST 3 . . . . .	6.24
6.5.1 Test Configuration . . . . .	6.24
6.5.2 Performance Evaluation . . . . .	6.27
6.5.3. Product Quality . . . . .	6.30
6.6 COMBINED BENCH-SCALE TEST EVALUATION . . . . .	6.32
7.0 REFERENCES . . . . .	7.1

## FIGURES

4.1 The Process of In Situ Vitrification . . . . .	4.1
5.1 Cross Section of the Crucible Test 1 Melt . . . . .	5.6
5.2 Cross Section of the Crucible Test 2 Melt . . . . .	5.6
5.3 Cross Section of the Crucible Test 3 Melt . . . . .	5.7
5.4 Cross Section of the Crucible Test 4 Melt . . . . .	5.7
5.5 Cross Section of the Crucible Test 5 Melt . . . . .	5.8
5.6 Cross Section of the Crucible Test 6 Melt . . . . .	5.8
5.7 Cross Section of the Crucible Test 7 Melt . . . . .	5.9
5.8 Cross Section of the Crucible Test 8 Melt . . . . .	5.9
6.1 Engineering-Scale ISV Processing Container . . . . .	6.3
6.2 Thermocouple and Test Soil Locations for BS-INEL-1 . . . . .	6.5
6.3 Vitrified Product Resulting from BS-INEL-1 . . . . .	6.6
6.4 Electrical Data for BS-INEL-1 . . . . .	6.8
6.5 Thermocouple and Test Soil Locations for BS-INEL-2 . . . . .	6.11
6.6 Off-Gas Sample Train Configuration for BS-INEL-2 . . . . .	6.13
6.7 Direct Metal Short on the Bottom of the BS-INEL-2 Vitrified Block . . . . .	6.15
6.8 Electrical Data for BS-INEL-2 . . . . .	6.16
6.9 Vitrified Product Resulting from BS-INEL-2 . . . . .	6.17
6.10 Cross Section of the BS-INEL-2 Vitrified Block . . . . .	6.17
6.11 Thermocouple and Test Soil Locations for BS-INEL-3 . . . . .	6.25
6.12 Electrical Data for BS-INEL-3 . . . . .	6.28
6.13 Vitrified Block Resulting from BS-INEL-3 . . . . .	6.29

6.14 Electrical Resistance Versus Energy Input for the Bench-Scale ISV Tests . . . . .	6.34
6.15 Melt Depth Versus Energy Input for Each Bench-Scale ISV Test . .	6.35

TABLES

4.1 Testing Units for Developing In Situ Vitrification Technology . . . . .	4.2
5.1 Test Matrix for the INEL Crucible Tests . . . . .	5.2
5.2 Corrosion Product Mix Composition . . . . .	5.3
5.3 INEL Crucible Test Results--Melt Viscosity . . . . .	5.5
5.4 INEL Crucible Test Results--Split Crucible Observations . . . . .	5.10
5.5 Normalized Composition of Glasses Produced in Crucible Tests 1, 2, and 3 . . . . .	5.12
5.6 Normalized Composition of Glasses Produced in Crucible Tests 4 Through 8 . . . . .	5.13
6.1 INEL Soil Sample Analyses . . . . .	6.2
6.2 Pretest and Posttest Soil and Glass Composition from BS-INEL-1 . . . . .	6.9
6.3 Hazardous Chemical Additions to BS-INEL-2 . . . . .	6.12
6.4 Pretest and Posttest Soil and Glass Compositions from BS-INEL-2 . . . . .	6.19
6.5 Off-Gas Sample Train Compositions and Loadings for BS-INEL-2 . . . . .	6.20
6.6 Posttest Surrounding Soil Compositions from BS-INEL-2 . . . . .	6.22
6.7 TCLP and EP Toxicity Concentrations for the BS-INEL-2 Glass and Metal . . . . .	6.23
6.8 Volatile Organic and Cement Additions to BS-INEL-3 . . . . .	6.26
6.9 Pretest and Posttest Soil and Glass Compositions for BS-INEL-3 . . . . .	6.30
6.10 Glass and Surrounding Soil Analysis for BS-INEL-3 . . . . .	6.31
6.11 TCLP and EP Toxicity Concentrations for the BS-INEL-3 Glass . . . . .	6.32
6.12 System Performance During Bench-Scale ISV Testing . . . . .	6.33



## 1.0 INTRODUCTION

In situ vitrification (ISV) is a thermal treatment process developed at the Pacific Northwest Laboratory (PNL) that converts contaminated soils (radioactive or hazardous) into a chemically inert and stable product composed of glass and crystalline phases. The high-temperature process destroys or removes organic contaminants while solidifying inorganic or radioactive contaminants in the glass or crystalline product, all in one processing step.

Currently, PNL is assisting the Idaho National Engineering Laboratory (INEL) in conducting a treatability study the application of ISV to the mixed transuranic (TRU) waste buried at the INEL subsurface disposal area (SDA). Based on preliminary information of the INEL SDA waste characteristics, depth, and contaminants, the ISV process appears well suited for SDA remediation. However, the application of ISV at INEL requires that the ISV technology be expanded to improve methods for processing soils with high metal contents. In addition, further ISV testing is needed to supplement the existing ISV data base with information on certain hazardous materials that have not been adequately evaluated in previous ISV tests.

As a result, eight crucible melts and three bench-scale ISV tests were performed on simulated metals/soil mixtures containing INEL site soils. This work is a continuation of ISV treatability tests on INEL soils that were started in FY 1988 (Oma, Reimus, and Timmerman 1989). The crucible tests were conducted to evaluate the effect of various chemical additives and metal oxidation techniques on soil melting temperatures, melt viscosities, metals versus electrode oxidation potentials, and metals incorporation in the glass. The bench-scale ISV tests were performed to supplement the existing ISV data base with information on certain hazardous materials that have not been adequately evaluated in previous ISV tests. These materials included five EP Toxicity metals (arsenic, barium, cadmium, chromium, and silver), various volatile organic materials fixed in a cementitious matrix [including carbon tetrachloride (CCl<sub>4</sub>), trichloroethylene (TCE), and tetrachloroethylene (PCE)], and asbestos. In addition, the bench-scale tests were used to evaluate the

effect of the proposed chemical additive (from crucible melt testing) on ISV processing performance and product quality.

Conclusions and recommendations from the crucible melt and bench-scale ISV tests are presented in Sections 2.0 and 3.0 of this report. A description of the ISV process is presented in Section 4.0. Crucible melt testing of the INEL metals/soil mixtures is discussed in Section 5.0; bench-scale ISV testing of the INEL soils and waste simulants is discussed in Section 6.0.

## 2.0 CONCLUSIONS

Eight crucible melts and three bench-scale ISV tests were performed on simulated metals/soil mixtures containing actual INEL site soils. An analysis of each crucible melt along with evaluations of the ISV processing behavior and the quality of vitrified product resulting from each bench-scale ISV test produced the following conclusions.

### 2.1 CRUCIBLE MELT TESTING

- None of the chemical additives that were tested enhanced metals incorporation in the glass. Although sodium nitrate is routinely added to glass to promote oxidizing melt conditions, it selectively oxidizes graphite instead of metal, and is not recommended for ISV processing with graphite electrodes.
- The greatest reduction in melt viscosity was achieved by adding sodium tetraborate ( $Na_2B_4O_7$ ) to the melt. No voids were observed in the crucible melt containing 11.6 wt%  $Na_2B_4O_7$ , indicating that the viscosity was sufficient to allow gases within the melt to escape to the melt surface.

### 2.2 BENCH-SCALE ISV TESTING

- Process performance results from bench-scale ISV tests indicated that the presence of metals in the melt can lead to processing concerns such as electrode shorts, decreased maximum power inputs (due to equipment limitations), increased downward melting rates (at the expense of horizontal melt growth), void formations in the glass, and increased energy-to-mass ratios for the soils. Most of these concerns require a large enough amount of metal (11 to 20 wt%) to produce a full or partial short at the bottom of the vitrified melt during processing.
- The addition of sodium tetraborate to INEL soils did not eliminate void formation in the vitrified block. Sodium tetraborate additions also appear to affect ISV processing adversely, by decreasing the maximum power inputs during ISV processing (which increases energy-to-mass ratios). It appears that the presence of voids in the INEL vitrified blocks is primarily due to the high metals content of INEL soils, not the relatively high viscosity of unmodified INEL soils.
- Penetrometer measurements of the molten glass surface during the first bench-scale test showed that the melt surface should be soft enough for electrode feeding to occur.

- Preliminary results indicate that portions of the cadmium and arsenic added to the second bench-scale test were removed from the melt during ISV processing. All of the other EP Toxicity metal additives (barium, chromium, and silver) were completely incorporated in the vitrified block. Cadmium and traces of arsenic were also found in the crusty soils within 1 in. from the bottom edge of the melt. Because of the relatively low boiling points of cadmium (765°C) and arsenic (613°C), it is thought that these materials may volatilize before they are encapsulated by the vitrified melt. However, it is expected that the cadmium and arsenic would have eventually been caught by either the vitrified melt or the ISV off-gas system if ISV processing had continued to a greater depth.
- Trace organic concentrations (PCE at <1 ppm and TCE at <0.1 ppm) were found in the surrounding soil less than 5 cm from the vitrified block. This is a 10,000- to 100,000-fold reduction over the concentration in the original cemented sludge/grease mixture. As a result, the presence of trace organics in the surrounding soils of the third bench-scale ISV test would not affect the nonhazardous nature of uncontaminated INEL soil.
- Due to the highly reducing nature of the ISV process, traces of arsenic and silver were found incorporated in the metallic slab at the bottom of the BS-INEL-2 melt. The quality of the vitrified block was not compromised by these reductions, since the leachability of the metal was negligible.
- The asbestos that was added to the second bench-scale test was completely incorporated into the vitrified mass.
- The second and third ISV bench-scale tests produced a high-quality vitrified mass that passed TCLP tests and EP Toxicity tests.

### 3.0 RECOMMENDATIONS

The following recommendations are made as a result of this study:

- Because of the strong potential for full or partial shorting during ISV processing of the INEL site soils, it is recommended that electrode feeding concepts be developed. With electrode feeding, the electrode locations in relation to these potential shorts are controlled by the test operator. As a result, the potential problems associated with ISV processing of soils with high metals concentrations (i.e., electrode shorting, power limitations, void formations, downward melt growth) may be controlled through proper test operations.
- Since sodium tetraborate addition does not eliminate voids in the vitrified block, it is recommended that future ISV treatability tests on INEL soils be performed without adding sodium tetraborate.
- Further studies are needed on the behavior of cadmium, arsenic, and other semivolatile heavy metals.



#### 4.0 PROCESS DESCRIPTION AND STATUS OF DEVELOPMENT

In situ vitrification has been developed as a remedial action process for soils contaminated with hazardous chemical wastes or radionuclides. Figure 4.1 illustrates how the ISV process operates. A square array of four molybdenum and graphite electrodes is inserted into the ground to the desired treatment depth. Because soil is not electrically conductive when its moisture is driven off, a conductive mixture of flaked graphite and glass frit is placed among the electrodes to act as a starter path. An electrical potential is applied to the electrodes to establish an electrical current in the starter path. The resultant power heats the starter path and surrounding soil to 1600°C to 2000°C, well above the initial soil melting temperatures of 1100°C to 1450°C. The graphite starter path is eventually consumed by oxidation, and the current is transferred to the molten soil, which is processed at temperatures between 1450°C and 2000°C. As the molten or vitrified zone grows, it incorporates radionuclides and nonvolatile hazardous elements such as heavy metals while destroying organic components by pyrolysis. The pyrolyzed byproducts migrate to the surface of the vitrified zone, where they combust in the presence of oxygen. A hood placed over the area being vitrified directs the gaseous effluents to an off-gas treatment system.

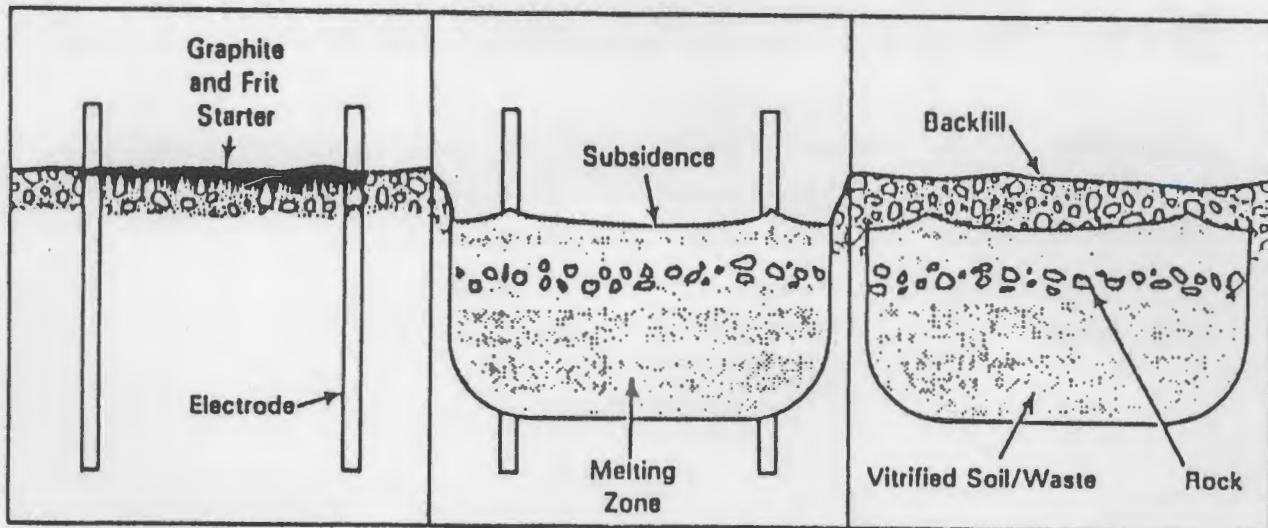


FIGURE 4.1. The Process of In Situ Vitrification

The process could also accommodate a staging operation alternative. In this processing alternative, outlying materials are placed on the surface of an area to be vitrified to increase the effective vitrification depth of each setting. Since ISV is a batch operation, this alternative will increase the amount of vitrified material in each batch setting, thereby improving the efficiency of the operation.

Since PNL began developing the ISV technology, numerous experimental tests under a variety of conditions have been conducted (Buelt et al. 1987; Timmerman and Oma 1984; Timmerman et al. 1983; Buelt and Carter 1986a and 1986b; Carter, Bates, and Maupin 1987). Table 4.1 describes the different scales of testing units used in developing ISV technology. The successful results of 74 bench-, engineering-, pilot-, and large-scale tests have proven the feasibility of the process. Also, economic studies have indicated that tremendous economies of scale are attainable with the ISV process (Oma et al. 1983).

In situ vitrification technology has been refined to the point that it is now ready for technology transfer and commercialization for specific hazardous waste types. In 1988, Battelle Memorial Institute established the Geosafe Corporation to commercialize the ISV technology for remediation of non-DOE waste sites that contain chemically and radioactively hazardous waste.

TABLE 4.1. Testing Units for Developing In Situ Vitrification Technology

<u>Equipment Size</u>	<u>Electrode Separation, m</u>	<u>Block Size</u>	<u>Tests Completed As of 1/26/90</u>
Bench-Scale	0.11	1 to 10 kg	19
Engineering-Scale	0.23 to 0.36	0.05 to 1.0 t	33
Pilot-Scale	0.9 to 1.5	10 to 50 t	17
Large-Scale	3.5 to 5.5	400 to 800 t	5

## 5.0 INEL CRUCIBLE TESTS

The objective of INEL crucible testing was to perform preliminary tests on various chemical methods that may prevent electrode shorting. The results of these tests provided preliminary indications of the feasibility of each method. The testing also served as a preliminary screening method for evaluating the effect of various chemical additions on melt viscosity control.

### 5.1 CHEMICAL METHODS AND EXPERIMENTAL APPROACH

All tests were conducted in preweighed, fused silica/alumina crucibles at 1450°C. If sufficient melting had not occurred after 30 min at 1450°C, the temperature was raised in 50°C increments (to a maximum of 1550°C) until sufficient melting occurred. Once the melting temperature was established, the crucible was covered and held at melt temperature for 6 h. The crucible was then removed and quickly placed into an annealing oven preheated to 600°C. A standard annealing time of 2 h was used. When the crucible was sufficiently cool, it was removed, weighed, and sectioned. Test measurements included visual observation of the melt, estimation of melt viscosity, inductively coupled plasma (ICP) spectroscopy of the glass matrices, and thin sectioning and microscopic examination of the test melts.

The crucibles were prepared by mixing 75 wt% INEL soil and 25 wt% metal shavings (unless noted otherwise). The 25 wt% metal consisted of a mixture of 70 wt% stainless steel and 30 wt% carbon steel shavings that had been precleaned with water and ethanol to remove any cutting fluids. The resultant metals/soil mixtures were then mixed with various chemical additives thought to promote lower melt temperatures and viscosities. The soil, metals, and chemicals were well mixed before being added to the crucible. Once addition was complete, a piece of graphite electrode (approximately 8 g) was placed in each crucible to simulate the presence of graphite electrodes in the melt.

Table 5.1 gives the test matrix for the INEL crucible tests. The first crucible melt (Crucible Test 1) contained the 25 wt% metals without any chemical additions. This crucible melt was used as a control for evaluating

TABLE 5.1. Test Matrix for the INEL Crucible Tests

<u>Test Number</u>	<u>Metals/Chemicals Addition</u>
1	25 wt% metal, no chemical additives
2	12.5 wt% metal, 12.5 wt% corrosion products, no chemical additives
3	25 wt% corrosion products, no chemical additives
4	25 wt% metal, 10 wt% $\text{NaNO}_3$ addition
5	25 wt% metal, 6.5 wt% $\text{Na}_2\text{CO}_3$ addition
6	25 wt% metal, 11.5 wt% $\text{CaCO}_3$ addition
7	25 wt% metal, 11.6 wt% $\text{Na}_2\text{B}_4\text{O}_7$ addition
8	25 wt% metal, 5.2 wt% NaOH addition

the other crucible melts. Crucible Tests 2 and 3 were performed to evaluate the effect of a chemical method called moist aeration, which attempts to oxidize the metals before vitrification proceeds. Crucible Test 4 was performed to evaluate the effect of  $\text{NaNO}_3$  addition on melt oxidation conditions. Finally, Crucible Tests 5, 6, 7, and 8 were performed to evaluate the effect of various chemical additives on melt viscosities. A description of these chemical methods, along with the experimental approaches that were used to evaluate each method, is presented below.

#### 5.1.1 Moist Aeration

Moist aeration is a previtrification oxidation technique that consists of treating the site before ISV processing with methods designed to oxidize the metals present. Metals in the oxide form are more readily soluble in the vitrified mass than unoxidized metals. The oxidation is accomplished by subjecting the site to aeration under moist conditions. The presence of chlorine ion and/or an acid pH during the aeration greatly enhances oxidation. This treatment takes a long time; approximately 20 years is required to corrode most mild steel drums under average burial conditions. However, the INEL burial sites may not require oxidation pretreatment because long periods of time have passed since burial, and any metals buried on site may have oxidized or corroded during this time.

The experimental approach assumes that 50% of the metal present at the site is oxidized. A crucible melt (Crucible Test 2) was performed with INEL soil containing 12.5 wt% metal and an amount of dry chemicals to represent corrosion products corresponding to 12.5 wt% corroded metal. Another crucible melt containing 25 wt% simulated corrosion products (Crucible Test 3) was also run for comparison. Both of these crucible melts were analyzed to determine the amount of metal element incorporated in the melt.

Table 5.2 gives the desired oxide composition of the corrosion product mix. This mix simulates the resulting corrosion product of a mixture of 70 wt% stainless steel and 30 wt% carbon steel. However, due to an error in the test instructions, the actual corrosion product mixture was high in nickel and low in chromium. This should result in higher levels of nickel and lower levels of iron and chromium than expected in the Crucible Test 2 and Crucible Test 3 melts.

### 5.1.2 Sodium Nitrate Addition

Sodium and potassium nitrate are routinely added to glass pools in the glass industry to promote oxidizing melt conditions. The nitrate acts as an oxidizer by releasing  $O_2$  as it decomposes at high temperatures ( $>400^\circ C$ ). The addition of  $NaNO_3$  to the site would also affect the melting properties of the soil by decreasing melt temperature and increasing the electrical conductivity of the melt. Addition of  $CaO$  with  $Na$  tempers the increase in electrical conductivity while achieving a comparable decrease in melt temperature.

Sodium/potassium nitrate should be added as mixtures with the soil to prevent the formation of a conductive layer of alkali at the melt surface. A surface layer may not readily mix with the soil beneath it and will thereby

TABLE 5.2. Corrosion Product Mix Composition

<u>Oxide</u>	<u>Desired Weight Percent</u>	<u>Actual Weight Percent</u>
$Fe_2O_3$	80.66	13.70
$Cr_2O_3$	13.70	5.64
$NiO$	5.64	80.66

slow or stop downward processing. The soil mixture can be layered on top of the site to be processed and will not form a conductive layer upon melting.

It should be noted that the addition of sodium/potassium nitrate may be limited by hazardous waste regulations. Since nitrates have a D class toxicity, approximately 10 wt%  $\text{NaNO}_3$  can be added to the soil without creating a hazardous waste condition. The addition of 10 wt%  $\text{NaNO}_3$  would provide enough  $\text{O}_2$  to oxidize approximately 3.6 wt% of the iron metal in the soil.

Sodium nitrate was added to INEL soil and metal to the 10 wt% level. The nitrate was well mixed with the soil.

#### 5.1.3 Viscosity Control of the Melt

Crucible tests were also performed to discern the effects of sodium compounds on melt viscosity. Three different sodium compounds were evaluated in the INEL metals/soil mixture. These compounds included  $\text{Na}_2\text{CO}_3$  (Crucible Test 5),  $\text{Na}_2\text{B}_4\text{O}_7$  (Crucible Test 7), and  $\text{NaOH}$  (Crucible Test 8). The amount of each addition was based on supplying a molar amount of sodium equivalent to that added by the 10 wt%  $\text{NaNO}_3$  addition (Crucible Test 4). Table 5.1 gives the relative concentrations of each chemical addition. The results were then compared to those obtained with the  $\text{NaNO}_3$  crucible melt (Crucible Test 4).

In addition to the sodium compounds listed above, the effect of calcium carbonate addition on melt viscosity was also examined (Crucible Test 6). The amount of  $\text{CaCO}_3$  that was added supplied a molar amount of calcium that was equivalent to the molar amount of sodium that was added in Crucible Tests 4, 5, 7, and 8. The crucible melt results were compared to those results obtained by the crucible melts with sodium compound additions.

## 5.2 RESULTS AND DISCUSSION

The observed melt viscosity of each crucible test is listed in Table 5.3, which also includes approximate sintering and fusion temperatures of INEL soil. The results indicate that  $\text{Na}_2\text{B}_4\text{O}_7$  is the most effective viscosity reducer of all of the chemical additives tested. All of the other sodium and calcium compounds were similar in their viscosity reduction actions. The reason that  $\text{Na}_2\text{B}_4\text{O}_7$  is so effective is that, in addition to its sodium

TABLE 5.3. INEL Crucible Test Results--Melt Viscosity

<u>Test Number</u>	<u>Description</u>	<u>Melt Viscosities</u>	<u>Comments</u>
--	Pure INEL soil	--	Sintering temperature: 1300°C Melt temperature: 1450°C
1	25 wt% metal	1000 P at 1550°C	Thick surface layer
2	12.5 wt% metal, 12.5 wt% corrosion product	300-500 P at 1500°C	--
3	25 wt% corrosion product	500 P at 1500°C	--
4	25 wt% metal, 10 wt% NaNO <sub>3</sub>	300-500 P at 1500°C	--
5	25 wt% metal, 6.5 wt% Na <sub>2</sub> CO <sub>3</sub>	1000 P at 1500°C	
6	25 wt% metal, 11.5 wt% CaCO <sub>3</sub>	300-500 P at 1500°C	
7	25 wt% metal, 11.6 wt% Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub>	200-400 P at 1450°C	--
8	25 wt% metal, 5.2 wt% NaOH	300-500 P at 1500°C	--

content, this compound contains boron, which also acts as an effective melt flux. Boron increases the relative durability of the glass by actually being incorporated in the glass matrix; sodium is not incorporated in the glass matrix and can be easily leached out, decreasing glass durability.

Cross sections of each of the crucible melts are shown in Figures 5.1 through 5.8. In addition, Table 5.4 lists the visual and optical observations of the split crucibles. All of the crucibles contained a number of small metal particles that were suspended within the melt and did not pool at the melt bottom. These metal particles were observed surrounding portions of the graphite in the melts, demonstrating that the graphite electrodes act as a melt reductant in the ISV vitrified block. The small size of these particles



FIGURE 5.1. Cross Section of the Crucible Test 1 Melt



FIGURE 5.2. Cross Section of the Crucible Test 2 Melt



FIGURE 5.3. Cross Section of the Crucible Test 3 Melt



FIGURE 5.4. Cross Section of the Crucible Test 4 Melt



**FIGURE 5.5.** Cross Section of the Crucible Test 5 Melt



**FIGURE 5.6.** Cross Section of the Crucible Test 6 Melt



FIGURE 5.7. Cross Section of the Crucible Test 7 Melt



FIGURE 5.8. Cross Section of the Crucible Test 8 Melt

**TABLE 5.4. INEL Crucible Test Results--Split Crucible Observations**

<u>Test Number</u>	<u>Description</u>	<u>Split Crucible Observations</u>
--	Pure INEL soil	Yellowish-brown glass with clumps
1	25 wt% metal	Black glass with green phase, some large cavities, large chunk of metal at crucible bottom, many small metal particles suspended in melt
2	12.5 wt% metal, 12.5 wt% corrosion product	Black glass, many small metal particles covering bottom half of crucible, larger particles toward bottom
3	25 wt% corrosion product	Black-brown glass, small, fine metal product particles throughout melt, bubbles at top of melt, metal surrounding graphite
4	25 wt% metal, 10 wt% $\text{NaNO}_3$	Black glass, graphite severely corroded, large chunk of metal at bottom, small voids in melt and bubbles at melt surface, some metal particles suspended in melt
5	25 wt% metal, 6.5 wt% $\text{Na}_2\text{CO}_3$	Black glass, large metal chunk at bottom, some fine metal particles present, many voids
6	25 wt% metal, 11.5 wt% $\text{CaCO}_3$	Black glass, large metal chunk at bottom, some fine metal particles present, one small void
7	25 wt% metal, 11.6 wt% $\text{Na}_2\text{B}_4\text{O}_7$	Black glass, large metal chunk at bottom, some small metal particles present, no voids
8	25 wt% metal, 5.2 wt% $\text{NaOH}$	Black glass, large metal chunk at bottom, some small metal particles present, metal surrounding graphite

prohibited their removal from the surrounding glass; therefore, a mass balance of the unincorporated metal in each crucible melt could not be made. Instead, ICP analysis was performed on the glass in each crucible melt to evaluate metals dissolution.

Voids were observed in all but one melt; that containing  $\text{Na}_2\text{B}_4\text{O}_7$  (see Figure 5.7). Apparently, the addition of  $\text{Na}_2\text{B}_4\text{O}_7$  reduced the melt viscosity

sufficiently to allow gases within the melt to escape to the melt surface. This finding is important as voids forming within the melt can hinder the progress of an ISV melt through some loss of electrical conductivity across the void.

The addition of sodium nitrate to the INEL metals/soil mixture appeared to serve as an effective oxidant of the metals/soil mixture (see Figure 5.4). However, selective oxidation of the graphite occurred instead of the desired metal oxidation. As a result,  $\text{NaNO}_3$  was eliminated as a potential chemical additive.

The glass from each of the crucibles was then analyzed using ICP to determine how the presence of differing amounts of fluxing agents and metal oxidation affected glass composition. The results of these analyses are shown in Tables 5.5 and 5.6. Each crucible melt was analyzed twice using ICP fusion. This was necessary to determine the representativeness of the sample and the homogeneity of the glass in the melt. In addition, the ICP results were normalized to 100% total oxides prior to being reported. This was due to the presence of graphite material in each of the crucible melts, which occasionally resulted in a low oxide composition for a particular sample.

Table 5.5 shows the normalized glass compositions for the crucible melts containing different amounts of oxidized metal. Included in this table is the normalized glass composition of pretest INEL soil, without the 25 wt% metal/corrosion products and 8 g of graphite that were added to each crucible melt.

In general, the analyses for Crucible Tests 1 and 3 were relatively uniform (with the exception of  $\text{Cr}_2\text{O}_3$  composition in Crucible Test 3); however, there was a significant difference in the concentrations of  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{NiO}$ , and  $\text{K}_2\text{O}$  for the two Crucible 2 analyses. The different compositions indicate that the Crucible 2 melt was relatively heterogeneous. This is suspected to be due to the presence of small metal particles in the melt and the relatively reducing nature of the melt itself. There is no apparent explanation as to why the melt heterogeneity was not seen in ICP samples from Crucible Tests 1 and 2.

TABLE 5.5. Normalized Composition of Glasses Produced in Crucible Tests 1, 2, and 3

Oxide	Pre-Test Soil	Crucible 1 (No Corrosion Products)		Crucible 2 (50% Corrosion Products)		Crucible 3 (100% Corrosion Products)	
		Test #1	Test #2	Test #1	Test #2	Test #1	Test #2
SiO <sub>2</sub>	71.2	62.9	62.7	60.0	49.0	55.4	54.6
Al <sub>2</sub> O <sub>3</sub>	13.5	12.1	11.7	11.2	9.97	10.3	10.1
Fe <sub>2</sub> O <sub>3</sub>	4.63	9.77	9.80	17.8	14.9	8.46	8.74
CaO	3.58	3.50	3.62	2.93	2.82	2.90	3.18
Na <sub>2</sub> O	1.55	1.27	1.29	1.24	1.11	1.19	1.13
Cr <sub>2</sub> O <sub>3</sub>	--	4.54	4.80	1.23	5.57	0.578	1.46
NiO	0.123	0.154	0.172	0.536	12.6	17.1	16.2
K <sub>2</sub> O	2.47	2.21	2.46	2.35	1.50	1.77	2.16
MgO	1.87	1.75	1.73	1.50	1.31	1.38	1.48
TiO <sub>2</sub>	0.763	0.705	0.685	0.630	0.523	0.575	0.568
MnO <sub>2</sub>	0.112	0.856	0.827	0.471	0.414	0.102	0.100
BaO	0.101	0.087	0.086	0.082	0.068	0.076	0.075
ZrO <sub>2</sub>	0.067	0.056	0.069	0.058	0.034	0.073	0.077
V <sub>2</sub> O <sub>3</sub>	--	0.029	0.039	0.016	0.034	0.000	0.050
SrO	0.022	0.025	0.025	0.023	0.019	0.023	0.023
La <sub>2</sub> O <sub>3</sub>	--	0.023	--	0.020	--	0.017	--
CuO	--	--	<u>0.012</u>	<u>0.014</u>	<u>0.017</u>	--	<u>0.011</u>
Total (Norm.)	100	100	100	100	100	100	100

All of the crucible melts exhibited higher concentrations of Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, and NiO than that observed in the pretest soil melt. This is due to the oxidation and incorporation of a portion of the added stainless steel in the glass melt during ISV processing. However, the primary difference between the three crucible melts from Table 5.5 are the concentrations of Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, and NiO. Crucible Test 1 exhibited the highest concentration of Cr<sub>2</sub>O<sub>3</sub>

TABLE 5.6. Normalized Composition of Glasses Produced in Crucible Tests 4 Through 8

in the three melts, Crucible Test 2 exhibited the highest concentration of  $Fe_2O_3$ , and Crucible Test 3 exhibited the highest concentration of NiO.

The different compositions are primarily attributed to the differing amounts and forms (metal or oxide) of iron, chromium, and nickel that were present in each crucible (see Table 5.2). The high concentrations of NiO in Crucible Tests 2 and 3 were due to the increased amount of nickel that was added as an oxide to each crucible; the low concentration of  $Cr_2O_3$  in Crucible Test 3 was due to the decreased amount of chromium that was added to the melt.

The increased concentration of  $Fe_2O_3$  in Crucible Test 2 was due to the large amount of iron (30%) that was present in an oxide form prior to melting. Although the amount of total iron present in Crucible Test 2 was less than that in Crucible Test 1, the oxide form of the iron was completely incorporated into the glass during melting. This same effect was also seen in Crucible Test 3, which was 100% corrosion products; however, the reduced amount of iron in the crucible limited the concentration of iron in the glass.

Table 5.6 shows the normalized compositions for crucible melts containing different fluxing additives. Glass compositions for the pretest soil melt and the crucible melt with no fluxing agents (Crucible Test 1) were also added to this table for comparison purposes.

In general, all of the melts exhibited similar glass compositions; the only differences were in the concentrations of  $Na_2O$  and  $CaO$  in Crucible Test 6,  $B_2O_3$  in Crucible Test 7, and concentrations of  $Fe_2O_3$ ,  $Cr_2O_3$  and NiO in Crucible Test 1. The increased concentrations of  $CaO$  and  $B_2O_3$  in Crucible Tests 6 and 7 and the decreased concentration of  $Na_2O$  in Crucible Test 6 were primarily due to the different fluxing chemicals that were added to these tests. However, the increased concentrations of  $Fe_2O_3$ ,  $Cr_2O_3$ , and NiO in Crucible Test 1 implies that the addition of fluxing agents to the INEL soil will not increase the amount of metal that is incorporated into the glass; rather, it appears that the amount of metal incorporation is decreased by the addition of fluxing agents.

In summary, it appears that the greatest viscosity reduction was achieved by adding  $Na_2B_4O_7$ . No voids were present in the Crucible 7 melt, indicating

that the glass viscosity reduction was sufficient to allow gases within the melt to escape to the melt surface. As a result, this compound is recommended for use as a viscosity reductant in ISV tests. However, there are concerns that the addition of  $\text{Na}_2\text{B}_4\text{O}_7$  may lower the electrical conductivity of the INEL soil enough to limit maximum power input. This will be evaluated during bench-scale ISV testing of the INEL soils. The addition of  $\text{NaNO}_3$  to the INEL metals/soil mixture did not significantly improve metal incorporation due to its selective oxidation of graphite instead of metal. As a result, it is not recommended for use during ISV processing.



## 6.0 INEL BENCH-SCALE ISV TESTS

Following the INEL crucible tests, three bench-scale ISV tests were performed on INEL soils. Each bench-scale test involved different amounts and types of materials that were mixed with the INEL soils. The bench-scale tests were performed to provide a preliminary evaluation of the effect of ISV processing on representative simulations of the metal-heavy INEL waste soils, and whether chemical additives were required to lower the melting temperatures of the INEL soils. In addition, various hazardous materials were added to two of the bench-scale tests to determine how these materials were affected by ISV processing.

### 6.1 SOIL CHARACTERIZATIONS

Samples of the uncontaminated soil from the INEL burial site were analyzed prior to each bench-scale test to determine the moisture contents, tap densities, and cationic oxide composition in the soils (using ICP analysis). The data are presented in Table 6.1. The ICP analyses confirm that the soil has enough silica (66%) and alumina (12%) to produce an acceptably durable glass product. However, the low concentrations of alkali material in the waste (4.0%) may increase the soil's melting temperature to a level that is too high for ISV processing. As a result, various levels of sodium tetraborate were added to each of the bench-scale tests to determine its effect on ISV processing of the INEL soils. Results from the INEL crucible tests have shown that sodium tetraborate is an effective chemical additive in reducing the viscosity of molten INEL soil.

### 6.2 ISV BENCH-SCALE TEST SETUP

The ISV bench-scale tests were performed in 55-gal drums that were placed in the ISV engineering-scale processing container. The engineering-scale container (illustrated in Figure 6.1) measures 1.8 m (6 ft) in diameter by 2.4 m (8 ft) tall, and is maintained at a slight negative pressure, with back-pressure high-efficiency particulate air- (HEPA) filtered relief on its lid to prevent particulate releases during testing. Any off gases generated during

TABLE 6.1. INEL Soil Sample Analyses

Oxide	Composition (a) (wt%)		
	BS-INEL-1	BS-INEL-2	BS-INEL-3
SiO <sub>2</sub>	67.1	65.0	69.4
Al <sub>2</sub> O <sub>3</sub>	12.3	12.2	13.3
Fe <sub>2</sub> O <sub>3</sub>	4.23	4.14	4.41
CaO	3.18	3.35	2.95
K <sub>2</sub> O	2.5	2.6	3.11
MgO	1.76	1.70	1.75
Na <sub>2</sub> O	1.51	1.44	1.64
TiO <sub>2</sub>	0.72	0.67	0.72
B <sub>2</sub> O <sub>3</sub>	<0.03	0.16	<0.03
MnO <sub>2</sub>	0.10	0.10	0.11
BaO	0.11	0.09	0.09
ZrO <sub>2</sub>	0.05	0.04	0.07
NiO	0.04	<0.03	<0.03
SrO	0.03	0.03	0.03
V <sub>2</sub> O <sub>3</sub>	<u>0.02</u>	<u>&lt;0.02</u>	<u>&lt;0.02</u>
Total Oxides	93.7	91.5	97.6
% Moisture	NM	11.5	7.1
Density, g/cc	NM	NM	1.25

(a) Average of two analyses of soil samples.  
 NM = Not measured.

ISV processing were swept out of the processing container by the ISV engineering-scale off-gas system. Each bench-scale ISV test was operated with an off-gas flow of 600 Lpm (21 scfm). The bench-scale tests were performed using a 10-kW, single-phase transformer with five voltage taps (400, 320, 240, 16, and 80 V) and silicon-controlled rectifier control.

Each bench-scale test was performed using two molybdenum electrodes, 1.3 cm (0.5 in.) in diameter. The electrodes were surrounded by a 3.8-cm-(1.5-in.-) diameter graphite collar in accordance with the ISV reference design for fixed electrodes. Melting was initiated by laying a

6.3

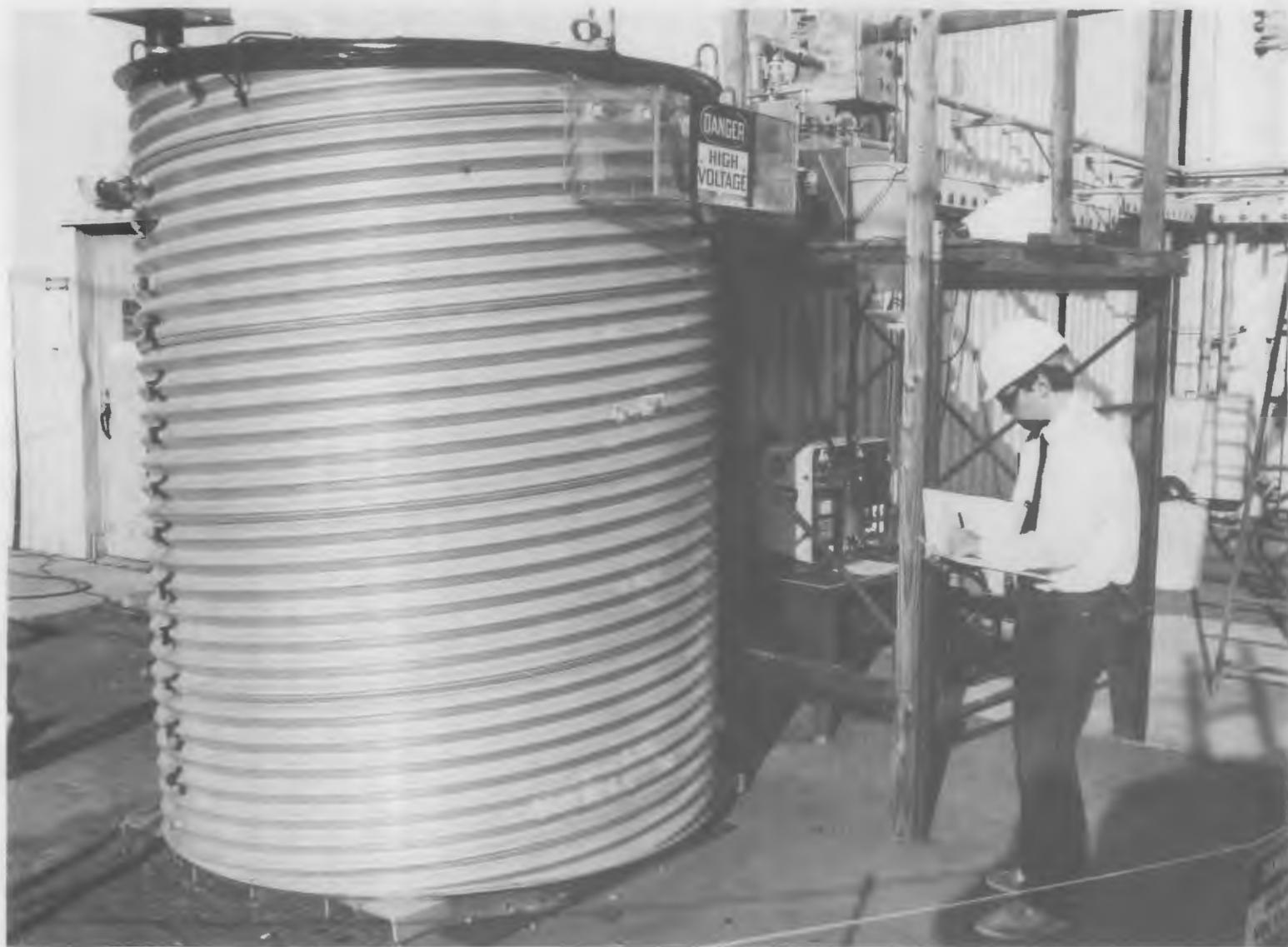


FIGURE 6.1. Engineering-Scale ISV Processing Container

2.5-cm- (1-in.-) deep, 2.5-cm- (1-in.-) thick conductive path of graphite/frit mixture between the two electrodes. During processing, the melt area was covered with 5 cm (2 in.) of blanket insulation to minimize surface heat loss and promote melt subsidence.

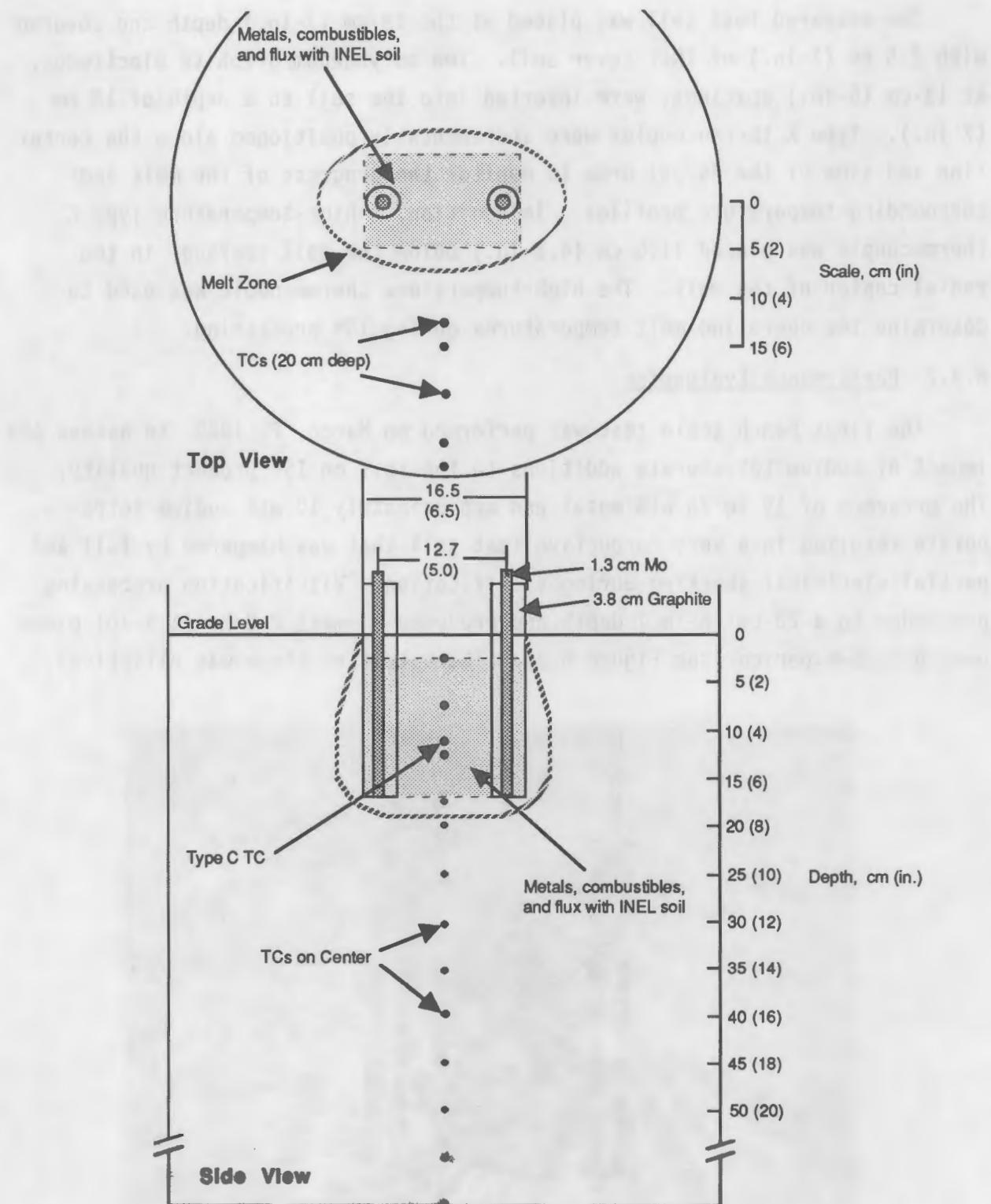
Descriptions of the test configurations and specific equipment requirements for each bench-scale ISV test are presented in the following sections.

### 6.3 BENCH-SCALE TEST 1 (BS-INEL-1)

#### 6.3.1 Test Configuration

Bench-scale test 1, identified as BS-INEL-1, was initially performed to evaluate the effects of sodium tetraborate addition on ISV processing of INEL soils that contained 11.6 wt% metals (50 wt% carbon steel, 50 wt% stainless steel) and 1 wt% paper/wood. Included in this test was an evaluation of the force necessary to penetrate the surface of the glass melt at various times during melting. This information was needed to aid in design of the electrode feeding systems that were to be used in future engineering-scale and intermediate-scale INEL tests.

Figure 6.2 shows the test configuration of BS-INEL-1. In the first bench-scale test, 672 g of metal (50 wt% stainless steel, 50 wt% carbon steel), 58 g of paper/wood, and 551 g of sodium tetraborate decahydrate were evenly distributed in a  $2,560 \text{ cm}^3$  (156 in.<sup>3</sup>) volume of prepared test soil that was placed between the two bench-scale electrodes. The amount of metal, paper/wood, and sodium tetraborate added to the prepared test soil volume was based on an assumption that the total weight of glass in the vitrified block would be 5.9 kg (13 lb) at the end of the test. The concentration of metals, paper/wood, and sodium tetraborate in the vitrified block would then be equivalent to the desired concentrations of 11.6 wt%, 1 wt%, and 5 wt%, respectively. However, the actual bench-scale test only produced a 2.3-kg (5-lb) block that encapsulated or consumed 63 to 83% of the metal, paper/wood, and sodium tetraborate. As a result, the actual concentrations of metal, paper/wood, and sodium tetraborate in the melt were increased 65% to 115%.



**FIGURE 6.2.** Thermocouple and Test Soil Locations for BS-INEL-1

The prepared test soil was placed at the 18-cm (7-in.) depth and covered with 2.5 cm (1 in.) of INEL cover soil. Two molybdenum/graphite electrodes, at 13-cm (5-in.) spacings, were inserted into the soil to a depth of 18 cm (7 in.). Type K thermocouples were incrementally positioned along the center line and side of the 55-gal drum to monitor the progress of the melt and surrounding temperature profiles. In addition, a high-temperature Type C thermocouple was placed 11.5 cm (4.5 in.) below the soil surface, in the radial center of the melt. The high-temperature thermocouple was used to determine the operating melt temperatures during ISV processing.

### 6.3.2 Performance Evaluation

The first bench-scale test was performed on March, 7, 1989, to assess the impact of sodium tetraborate additions to the soil on ISV product quality. The presence of 19 to 25 wt% metal and approximately 10 wt% sodium tetraborate resulted in a very conductive test soil that was hampered by full and partial electrical shorting during vitrification. Vitrification processing proceeded to a 20-cm (8-in.) depth and produced a small 2.0-kg (4.5-lb) block over a 3.15-h period (see Figure 6.3). The vitrified block was elliptical,



FIGURE 6.3. Vitrified Product Resulting from BS-INEL-1

with a maximum diameter of 10 cm (4 in.), and contained a significant void volume. Because of the small melt diameter, the electrodes were not an integral part of the vitrified block (see Figure 6.3).

Electrical data for BS-INEL-1 are illustrated in Figure 6.4. Only 4.6 kWh of electricity was input into the melt during the 3.15 h that the power was on. The resultant average power level of 1.46 kW is significantly below the desired average power level of 4.6 kW because the high metal concentrations in the test soil caused the melt to proceed at a power input level between 2 kW and 3 kW after the graphite path had burned off to prevent excessive arcing. The resultant energy-to-mass ratio for the vitrified block (1.9 kWh/kg of soil) was significantly higher than typical energy-to-mass ratios, which ranged from 0.8 to 1.1 kWh/kg.

During the latter portion of the test, the power level for BS-INEL-1 averaged 1.8 kW. Assuming a maximum elliptical melt cross-sectional geometry with an area of 51 cm<sup>2</sup> (7.9 in.<sup>2</sup>), the actual power density for this test was 350 kW/m<sup>2</sup> (33 kW/ft<sup>2</sup>). Although somewhat higher than the large-scale ISV system density of 286 kW/m<sup>2</sup> (26.6 kW/ft<sup>2</sup>), the power density was still within acceptable limits to ensure that the tests were operated under representative conditions.

Data on the operating melt temperature were limited because the high-temperature Type C thermocouple failed 2.8 to 3.4 h after testing. However, melt temperatures of 1040°C to 1780°C were recorded by the Type C thermocouple for the 0.6 h before it completely oxidized. The significant oscillations in temperature during this time may have been caused by the large voids that were present in the molten glass. This was verified during post-test analysis of the block. Glass temperatures above 1550°C are needed to produce a high-quality vitrified block.

Penetrometer tests of the molten glass surface were taken at three different times during testing, using a 1-in.-diameter quartz rod. The penetrometer readings were necessary to determine whether the molten-glass surface, with sodium tetraborate addition, was soft enough to allow electrode penetration into the glass (an important requirement for the proposed electrode feeding design). The penetrometer pressure measured on the surface of

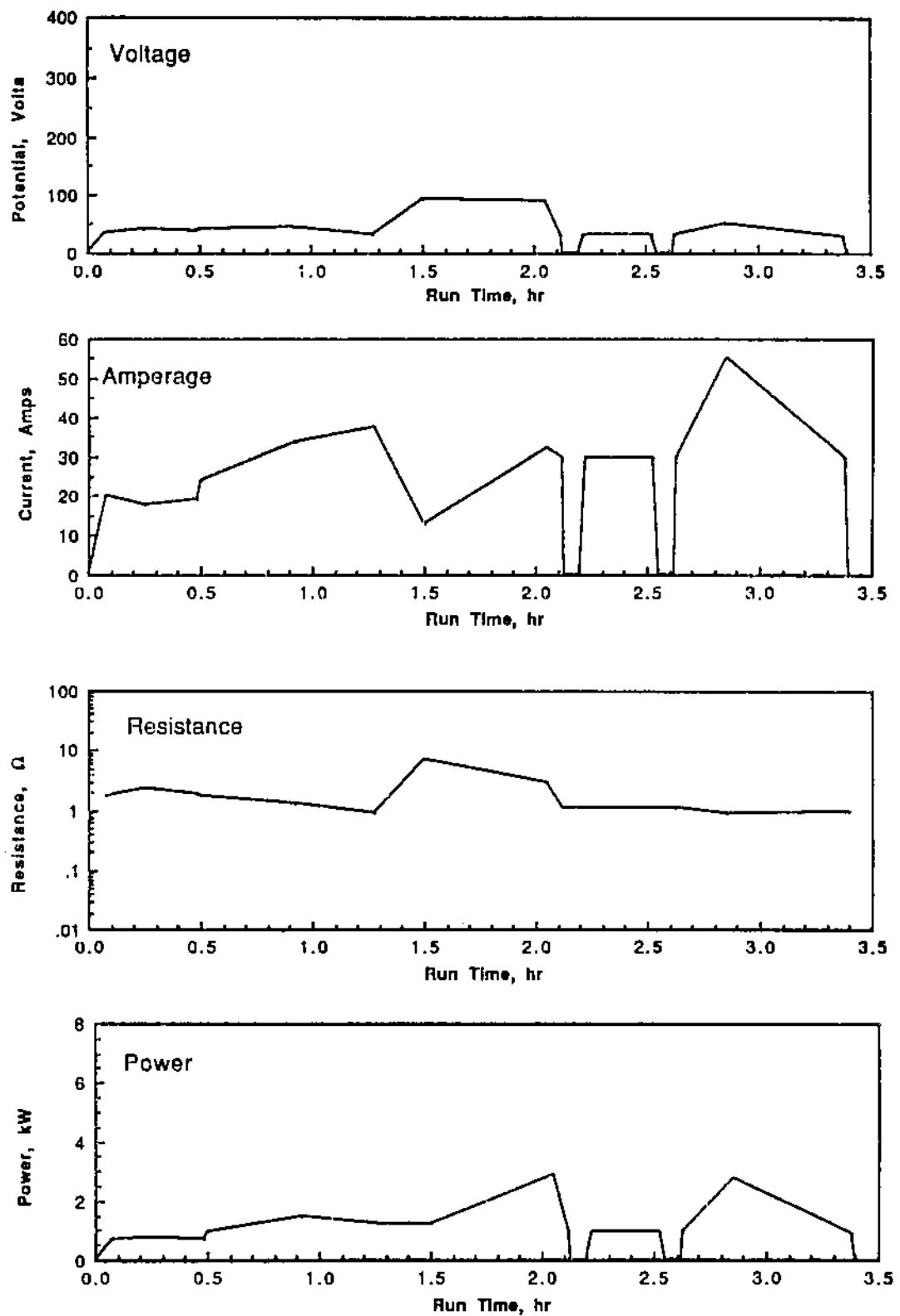


FIGURE 6.4. Electrical Data for BS-INEL-1

the BS-INEL-1 melt ranged from 0.1 to 1.8 kg/cm<sup>2</sup> (1.4 to 26 psig). The current electrode feeding design for the intermediate-scale ISV system has a maximum applied surface pressure of 2.5 kg/cm<sup>2</sup> (35 psig).

### 6.3.3 Product Quality

The composition of glass from the first bench-scale test is shown in Table 6.2 along with the original soil composition for the first bench-scale test, before the addition of metal and sodium tetraborate. The results show significant increases in sodium, boron, manganese, chromium, and lithium concentration, and a significant decrease in iron concentration between the pretest soil and the posttest glass. The increased sodium and boron concentrations in the BS-INEL-1 glass are due to the addition of sodium tetraborate

**TABLE 6.2. Pretest and Posttest Soil and Glass Composition from BS-INEL-1**

Oxide	Composition, wt%	
	Pretest Soil	Vitrified Glass
SiO <sub>2</sub>	67.1	66.6
Al <sub>2</sub> O <sub>3</sub>	12.3	12.4
Na <sub>2</sub> O	1.51	9.77
Fe <sub>2</sub> O <sub>3</sub>	4.23	2.16
CaO	3.18	3.20
K <sub>2</sub> O	2.5	2.35
MgO	1.76	1.95
B <sub>2</sub> O <sub>3</sub>	<0.01	0.87
TiO <sub>2</sub>	0.72	0.72
MnO <sub>2</sub>	0.10	0.40
Cr <sub>2</sub> O <sub>3</sub>	<0.02	0.23
BaO	0.11	0.10
ZrO <sub>2</sub>	0.05	0.06
Li <sub>2</sub> O	<0.01	0.06
NiO	0.04	0.04
SrO	0.03	0.03
V <sub>2</sub> O <sub>3</sub>	<u>0.02</u>	<u>0.01</u>
Total Oxides	93.7	100

to the BS-INEL-1 soil. The increased chromium concentration suggests that some of the stainless steel may have been oxidized and incorporated into the melt; however, this theory is refuted by the accompanying decrease in iron concentration. As a result, the effect of ISV processing on metals incorporation in the BS-INEL-1 melt is inconclusive.

#### 6.4 BENCH-SCALE TEST 2 (BS-INEL-2)

##### 6.4.1 Test Configuration

The second bench-scale ISV test (identified as BS-INEL-2) was needed to evaluate the ISV product quality of vitrified INEL soil containing metals, paper/wood, sodium tetraborate, asbestos, and various heavy metals. The product quality evaluation included a determination of the leachability of heavy metals from the glass and an evaluation of the extent of asbestos incorporation in the glass. In addition, the surrounding posttest soils were tested for the presence of heavy metals, and an estimate was made of the amount of heavy metals that were removed by the ISV off-gas system.

Figure 6.5 shows the test configuration for BS-INEL-2. The desired concentrations of metal, paper/wood, and sodium tetraborate in the vitrified block were the same as that originally called for with BS-INEL-1 (11.6 wt%, 1 wt%, and 5 wt%, respectively). To produce a more representative test configuration, however, the entire soil layer to be vitrified (see Figure 6.5) was prepared with a uniform concentration of 11.6 wt% metal, 1 wt% paper/wood, and 5 wt% sodium tetraborate. This change was designed to limit downward melt growth to a rate that is more representative of the actual site remediation rate. In contrast with the metals, paper/wood, and sodium tetraborate additions, the asbestos and heavy metals in BS-INEL-2 were placed in a 13 cm (5 in.) x 5 cm (2 in.) x 15 cm (6 in.) inner test volume between the two electrodes (see Figure 6.5). Use of the inner test volume ensures that these potentially hazardous materials are able to be completely encapsulated during ISV processing. The desired concentrations of asbestos (0.25 wt%) and heavy metals (0.2 wt% each) in BS-INEL-2 are based on an assumed glass block weight of 9.9 kg (22 lb). The amounts of asbestos and heavy metals added to the inner test volume of BS-INEL-2 are shown in Table 6.3.

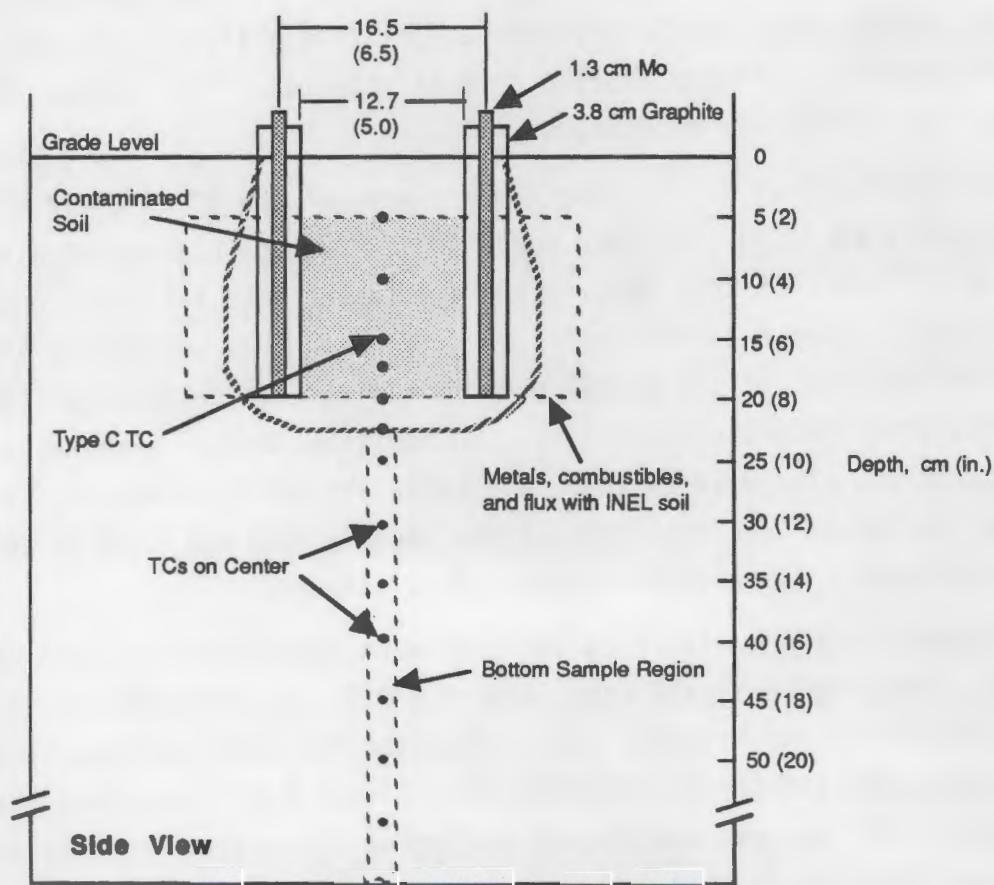
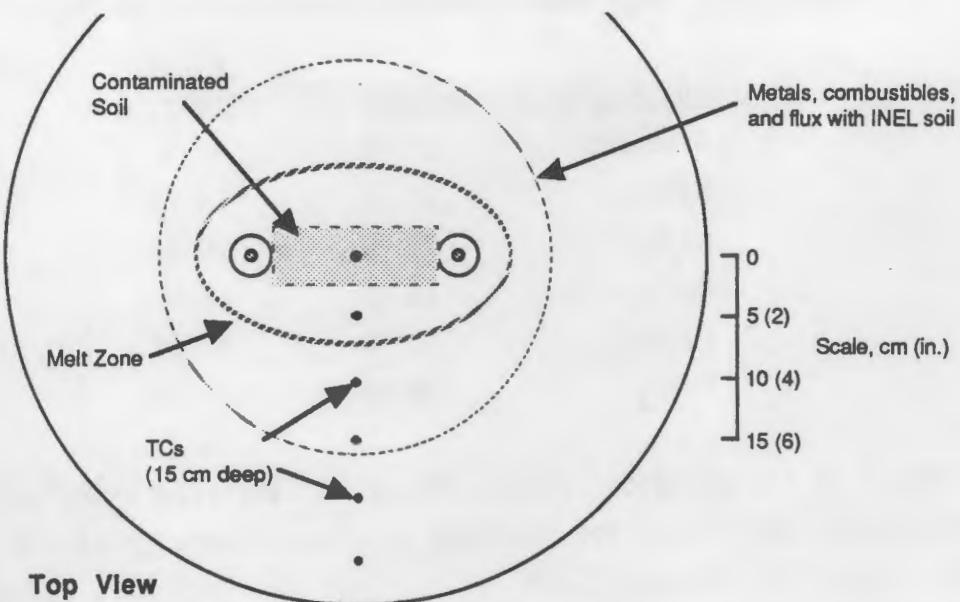


FIGURE 6.5. Thermocouple and Test Soil Locations for BS-INEL-2

TABLE 6.3. Hazardous Chemical Additions to BS-INEL-2

<u>Hazardous Material</u>	<u>Weight, g</u>	<u>Metal Weight, g</u>
Asbestos	24.75	--
$\text{AgNO}_3$	31.11	19.75
$\text{As}_2\text{O}_3$	26.22	19.86
$\text{BaCO}_3$	28.45	19.80
$\text{Cd}(\text{NO}_3)_2$	41.73	19.84
$\text{Cr}_2\text{O}_3$	28.92	19.78

A sample of the asbestos from BS-INEL-2 was analyzed using polarized light microscopy and dispersion staining in accordance with 40 CFR Ch. I, Part 763, Subpart F, App. A (1-1-87 ed.). This was necessary to determine the type of asbestos used in the test. The results of this analysis showed that the asbestos was a white granular and fibrous material containing 15 to 25% amosite, a highly friable form of asbestos. The balance of the asbestos (75 to 85%) was nonfibrous.

The prepared test soil volumes were placed at the 20-cm (8-in.) depth and covered with 5 cm (2 in.) of INEL cover soil. Two molybdenum/graphite electrodes at 16.5-cm (6.5-in.) spacings were inserted into the soil to a depth of 20 cm (8 in.). Type K thermocouples were incrementally positioned along the center line and side of the 55-gal drum to monitor the progress of the melt and surrounding temperature profiles. In addition, a high-temperature Type C thermocouple was placed 14 cm (5.5 in.) below the soil surface, in the radial center of the melt. The high-temperature thermocouple was used to determine the operating melt temperatures during ISV processing.

Off-gases from the vitrification zone were isokinetically sampled during the test. The sampling train consisted of a U.S. Environmental Protection Agency (EPA) Reference Method 5 sampling scheme to sample off-gas particulate and semivolatiles (40 CFR 60, Appendix A). Figure 6.6 illustrates the sample train setup. Off-gas particulate was collected by drawing a representative sample from the 5-cm (2-in.) off-gas process line through a heated probe assembly leading into a glass fiber filter. The filter was housed in an oven

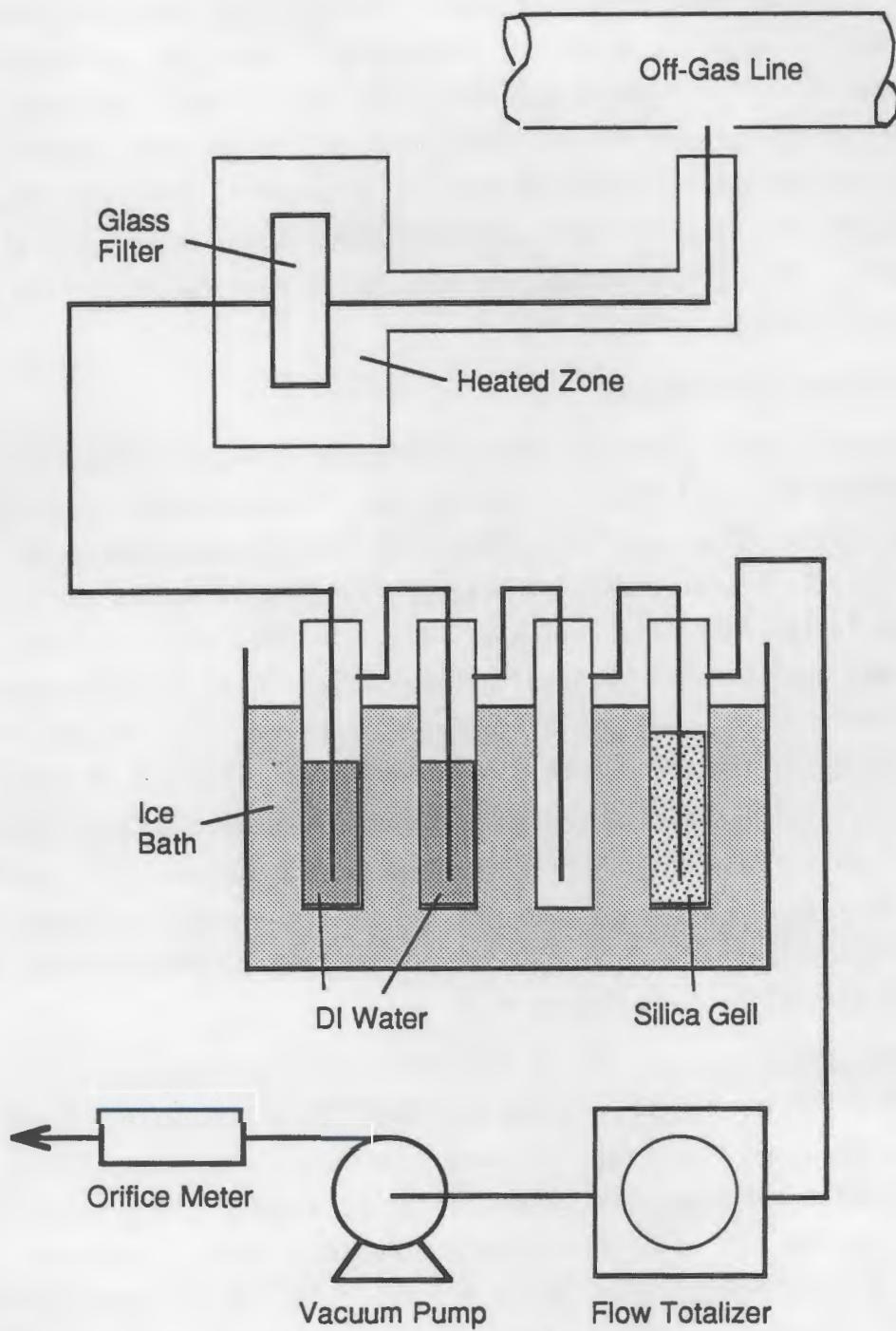


FIGURE 6.6. Off-Gas Sample Train Configuration for BS-INEL-2

compartment maintained above 120°C (250°F). After exiting the filter/oven assembly, the gases were drawn through a series of two Greenburg-Smith impingers containing deionized water for condensate collection. A third empty impinger was placed in line to collect carry-over liquid/condensate, and a final impinger containing 300 g of 6-16 mesh silica gel was used to trap water vapor. A control unit was used to monitor and control sampling rates, record sample volume, and track off-gas sampling temperatures during the vitrification process. The off-gas stream was sampled at an isokinetic flow rate of 19.8 Lpm (0.70 scfm) during BS-INEL-2.

#### 6.4.2 Performance Evaluation

The second bench-scale ISV test on INEL soils was performed on March 24, 1989. The test was performed to evaluate the ISV processing performance and product quality of INEL metals/soil mixtures containing asbestos and various heavy metals. To better control the concentrations of metals and sodium tetraborate in the INEL test soil simulants, the metals/soil mixture for BS-INEL-2 was applied over an area that was larger than the projected maximum diameter of the melt, based on ISV computer modeling. This reduced the metals and soil concentrations in the melt to the desired values of 11.6 wt% and 5 wt%, respectively. The lowered metals and sodium tetraborate concentrations resulted in an improved melt performance, with full or potential shorting occurring only in the last 0.8 h of testing. A full short developed in the vitrified block after 2.35 h of testing, which led to termination of the run after 2.8 h of testing (see Figure 6.7).

Electrical data for BS-INEL-2 are illustrated in Figure 6.8. The data profiles were similar to those found in other ISV melts, with a relatively conductive, low-power start-up, followed by a significant increase in melt resistance and power input that gradually stabilized as the graphite path burned off and the soil started to melt. The total amount of energy input into the block was 6.8 kWh over the 2.8-h run time, for an average power input of 2.44 kW. However, 6.0 kWh of the total energy was input into the vitrified block in the 1.45 h between the initiating of soil melting (0.9 h after startup) and the occurrence of a full short between the two electrodes (2.35 h



**FIGURE 6.7.** Direct Metal Short on the Bottom of the BS-INEL-2 Vitrified Block

after startup), an average power input of 4.0 kW over this period. The power input over this period was relatively close to the desired power average of 4.6 kW.

Vitrification processing proceeded to a depth of over 23 cm (9 in.) and produced a 4.5-kg (10.0-lb) block over the 2.8-h period. The energy-to-mass ratio of the resultant vitrified block was 1.2 kWh/kg of soil--slightly higher than typical ISV melt ratios of 0.8 to 1.1 kWh/kg. A photograph of the resultant glass block is shown in Figure 6.9. The vitrified block was elliptical, with maximum dimensions of 24 cm (9.5 in.) high and 23 cm (9 in.) by 14.6 cm (5.75 in.) wide. A significant void volume was also observed in the top half of the vitrified block after it was cross sectioned (see Figure 6.10).

During the latter portion of the test, the power level averaged 4.46 kW. Assuming an elliptical melt cross-sectional geometry and a surface cross-sectional area of  $162 \text{ cm}^2$  (25 in. $^2$ ), the actual power density for this test

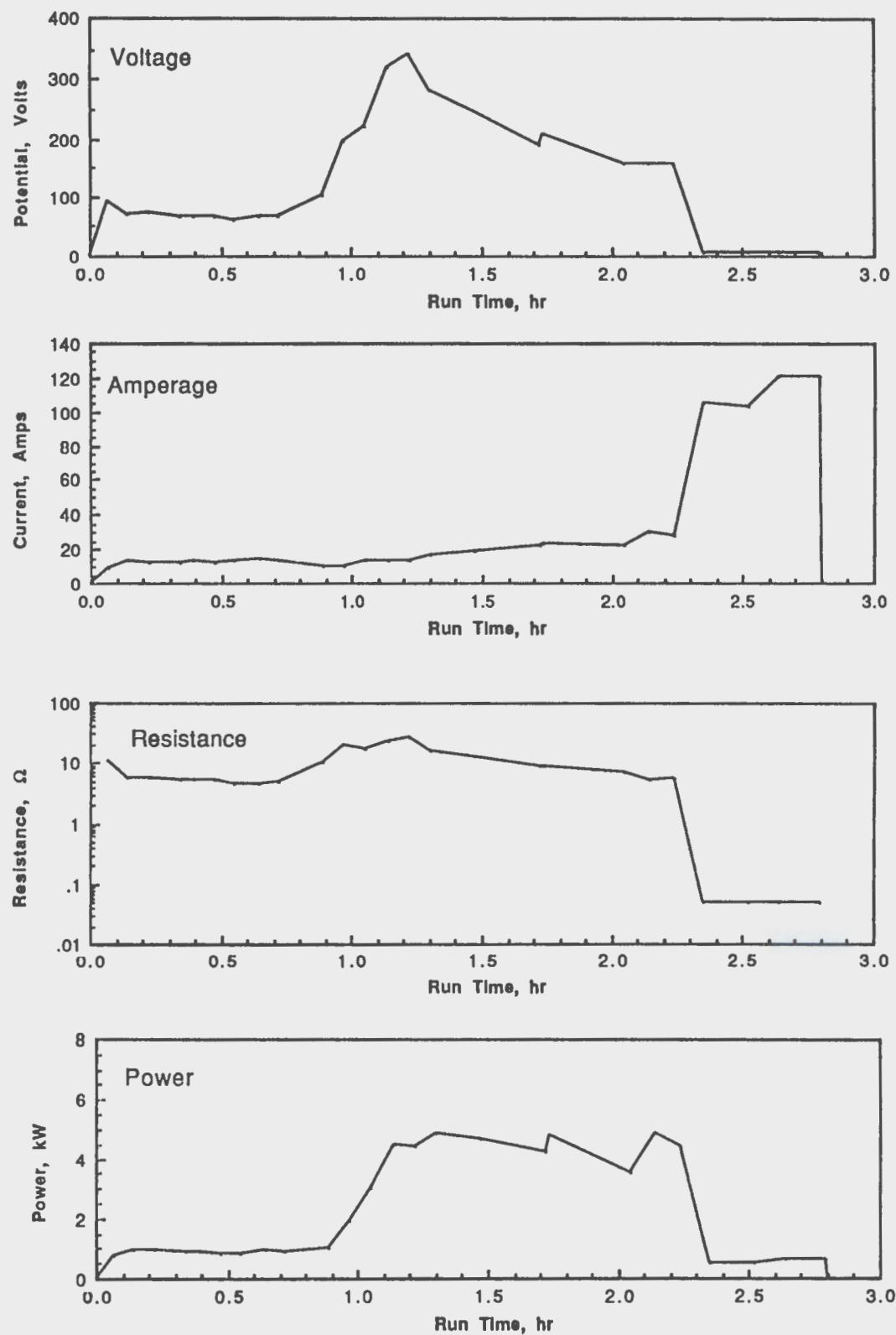
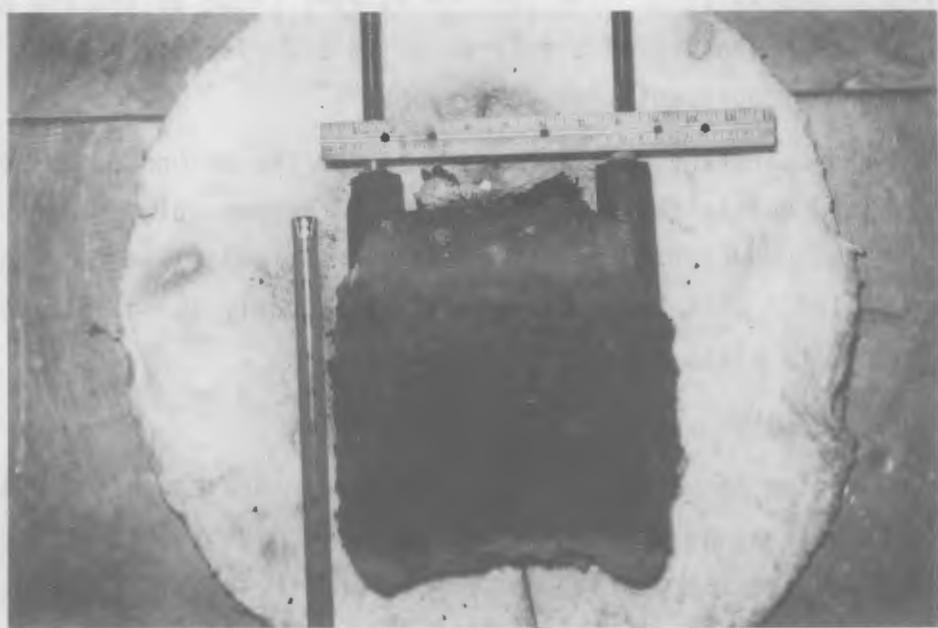
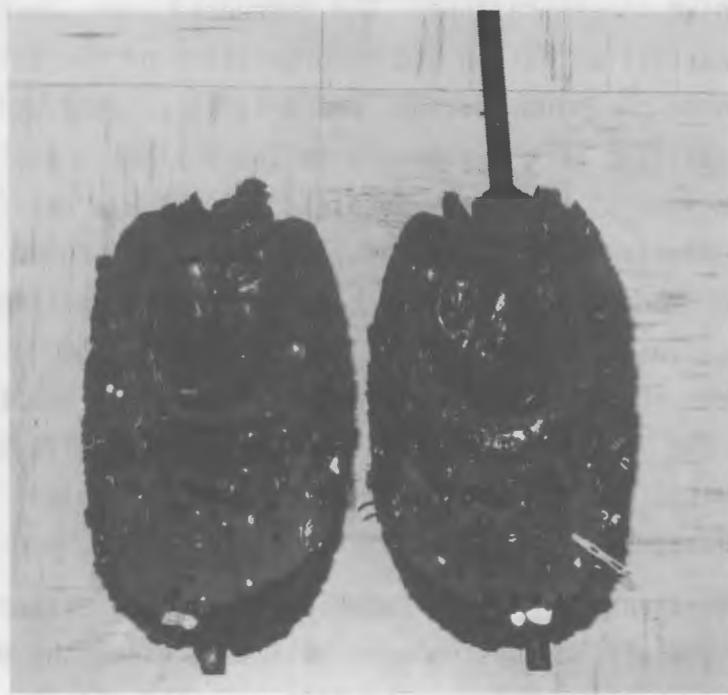


FIGURE 6.8. Electrical Data for BS-INEL-2



**FIGURE 6.9.** Vitrified Product Resulting from BS-INEL-2



**FIGURE 6.10.** Cross Section of the BS-INEL-2 Vitrified Block

was 275 kW/m<sup>2</sup> (25.6 kW/ft<sup>2</sup>). The density is very close to the 286 kW/m<sup>2</sup> (26.6 kW/ft<sup>2</sup>) power density of the large-scale ISV system, implying that the test was run under representative conditions.

Data on the operating melt temperatures for the second bench-scale ISV test were limited due to failure of the type C thermocouple readout after 2.1 h of testing. However, a melt temperature of 1643°C was recorded before the readout failed. This temperature is significantly above 1550°C, implying that a high-quality glass product was produced.

#### 6.4.3 Product Quality

The composition of vitrified soil from the second bench-scale test is shown in Table 6.4, along with the original soil composition, before the addition of metal, sodium tetraborate, asbestos, and EP Toxicity metals. In addition, the soil composition of the pretest mixture of INEL soil, metal, combustibles, and sodium tetraborate is included in Table 6.4. The results show significant increases in iron, chromium, barium, zinc, manganese, arsenic, and lithium concentrations. The increased iron concentration is probably due to partial oxidation and incorporation of the metals into the glass; the increased chromium, barium, and arsenic concentrations were probably due to the addition of heavy metals to the BS-INEL-2 test soil. Interestingly, cadmium (one of the heavy metal additions) was not found in the glass, while arsenic (another heavy metal addition) was found in the glass at a level much lower than that of either barium or chromium (two other heavy metal additives). This implies that all of the cadmium and a portion of the arsenic were either reduced into their metallic forms or volatilized to the off-gas system. The lack of cadmium in the glass was confirmed by x-ray analysis of the vitrified glass sample. (The vitrified glass was not analyzed for silver, the other heavy metal additive in the BS-INEL-2 test soil.)

The composition and loading of materials that were either volatilized or entrained to the ISV off-gas system were estimated using the off-gas sampling system discussed in Section 6.4.1. The filter in the off-gas system was used to estimate the composition and loading of large entrained particulate in the system off gas, and the impinger solutions were used to capture the remaining particulate. In addition, the entrained particulate that plated out in front

TABLE 6.4. Pretest and Posttest Soil and Glass Compositions from BS-INEL-2

Oxide	Composition, wt%		
	Pretest Soil	Pretest Mix	Vitrified Soil
SiO <sub>2</sub>	65.0	58.2	64.3
Al <sub>2</sub> O <sub>3</sub>	12.2	11.0	12.5
Fe <sub>2</sub> O <sub>3</sub>	4.14	3.71	5.70
Na <sub>2</sub> O	1.44	5.36	4.60
CaO	3.35	2.90	3.70
K <sub>2</sub> O	2.6	2.4	2.6
MgO	1.70	1.51	2.18
Cr <sub>2</sub> O <sub>3</sub>	<0.02	<0.02	0.87
BaO	0.09	0.08	0.82
TiO <sub>2</sub>	0.67	0.59	0.72
ZnO	<0.02	<0.02	0.26
MnO <sub>2</sub>	0.10	0.09	0.24
B <sub>2</sub> O <sub>3</sub>	0.16	0.12	0.08
As <sub>2</sub> O <sub>3</sub>	<0.08	<0.08	0.15
ZrO <sub>2</sub>	0.04	0.04	0.07
Li <sub>2</sub> O	<0.01	<0.01	0.05
SrO	0.03	0.02	0.03
NiO	<0.02	0.03	<0.02
V <sub>2</sub> O <sub>3</sub>	0.01	0.01	0.02
CuO	<u>&lt;0.01</u>	<u>&lt;0.01</u>	<u>0.01</u>
Total Oxides	91.5	86.0	97.9
Density, g/cc	NM <sup>(a)</sup>	NM	2.18

(a) NM = not measured.

of the off-gas filters was also collected and filtered. The rinsed filtrate was then added to the material collected in the off-gas impinger, and the filtered residue was added to the filtered particulate material. The total weight and composition of material collected in the filters, impingers, and total off-gas train are shown in Table 6.5. In addition, Table 6.5 contains estimates of the total amount of material that passed through the ISV

TABLE 6.5. Off-Gas Sample Train Compositions and Loadings for BS-INEL-2

<u>Element</u>	<u>Total Filtered Weight, mg</u>	<u>Total Weight in Impingers, mg</u>	<u>Total Weight in Sample System, mg</u>	<u>Total Off-Gas Release, mg</u>
Al	0.081	<0.011	<0.092	<2.79
As	4.7	0.063	4.76	144.3
B	9.48	0.554	10.03	304.1
Ba	<0.0032	<0.0008	<0.004	<0.012
Ca	0.073	0.097	0.17	5.15
Cd	19.19	<0.158	19.35	586.4
Cu	0.032	<0.0025	<0.035	<1.05
Fe	1.168	<0.0017	1.17	35.4
K	7.037	0.24	7.28	220.5
Li	1.907	<0.023	1.93	58.5
Mg	0.162	0.029	0.191	5.79
Mn	0.292	0.101	0.393	11.9
Mo	5.455	0.108	5.56	168.6
Na	19.17	0.402	19.58	593.2
P	1.091	<0.03	<1.12	<34.0
Pb	0.522	<0.019	<0.541	<16.4
Si	2.598	0.881	3.48	105.4
Zn	2.626	0.548	3.17	96.2
Zr	<u>0.031</u>	<u>&lt;0.003</u>	<u>&lt;0.034</u>	<u>&lt;1.03</u>
Total	75.6	<3.08	<78.7	2.38 g

engineering-scale off-gas system. The estimates were made by multiplying the total sample system weights by 30.3. This number is equivalent to the total ISV off-gas system flow rate (600 lpm, or 21.2 scfm) divided by the average flow rate of the off-gas sample train (19.8 lpm, or 0.7 scfm). The total amount of material estimated to pass through the engineering-scale off-gas system was 2.38 g. This amount represents only 0.052 wt% of the vitrified block.

The off-gas data show that significant amounts of cadmium (3.0%) and arsenic (0.73%) were sent to the ISV off-gas system during processing. Other ISV processing tests have shown up to 35% of the cadmium can be volatilized or

entrained to the ISV off-gas system during processing (Buelt et al. 1987). As a result, it is suspected that even more cadmium and arsenic would have been found in the ISV lid and off-gas smears and the insulation blanket sample, if these materials had been analyzed by ICP. It is recommended that these samples be analyzed in the fourth engineering-scale ISV test on INEL soils (ES-INEL-4), currently scheduled for July 1989. The amount of cadmium and arsenic released to the ISV system off-gas is expected to decrease in field operations, where the buried depths of these contaminants is generally greater than 1 m (3.3 ft).

Samples of the soil beneath the BS-INEL-2 vitrified block were also analyzed after testing. This was performed to determine whether there was any migration of EP Toxicity heavy metals to the surrounding soil. The soil samples were taken near the edge of the vitrified block and at four soil positions that correspond to soils that reached estimated maximum temperatures of 250°C, 100°C, 70°C, and 40°C. The soil samples were analyzed using ICP analysis.

The compositions of each soil sample are shown in Table 6.6. The results show that none of the EP Toxicity metals migrated outside of the melt edge. Although barium was present in the five soil samples, its composition was similar to those found in the original INEL soil (see Tables 6.1 and 6.4), and are not due to migration. However, the ICP analyses indicate that some cadmium was present in the crusty soil within 1 in. of the vitrified melt bottom, less than 6 cm (2.5 in.) away from the original contaminated soil placement. This is not in accordance with previous data which showed no movement of cadmium into surrounding soils during ISV processing (Buelt et al. 1987). The presence of cadmium was verified by x-ray analysis of the edge soil sample, which also showed trace amounts of arsenic (below ICP detection limits, however). The concentration of cadmium in the edge soil sample (0.18 wt%) was significantly less than that present in the original 983 mL of contaminated test soil make-up (approximately 1.9 wt%).

Because of the relatively low boiling points of cadmium (765°C) and arsenic (613°C), it is postulated that some of materials may have volatilized before they were encapsulated by the vitrified melt. However, based on the

TABLE 6.6. Posttest Surrounding Soil Compositions from BS-INEL-2

Oxide	Melt Edge	Composition, wt%			
		250°C Isotherm	100°C Isotherm	70°C Isotherm	40°C Isotherm
SiO <sub>2</sub>	67.3	64.7	66.0	65.8	63.5
Al <sub>2</sub> O <sub>3</sub>	12.6	12.5	12.6	12.4	12.7
Fe <sub>2</sub> O <sub>3</sub>	4.76	5.13	4.63	5.25	5.86
CaO	2.68	3.30	3.03	3.17	3.65
K <sub>2</sub> O	3.0	2.5	2.7	1.8	1.7
MgO	1.70	1.95	1.73	1.74	2.20
Na <sub>2</sub> O	1.59	1.63	1.74	1.40	1.67
TiO <sub>2</sub>	0.71	0.82	0.71	0.69	1.07
MnO <sub>2</sub>	0.12	0.32	0.17	0.10	0.12
CdO	0.18	<0.01	<0.01	<0.01	<0.01
BaO	0.10	0.11	0.10	0.10	0.09
As <sub>2</sub> O <sub>3</sub>	<0.08	<0.08	<0.08	<0.08	<0.08
ZrO <sub>2</sub>	0.04	0.07	0.04	0.04	0.04
SrO	0.04	0.04	0.04	0.06	0.05
NiO	0.04	<0.02	0.03	0.03	<0.02
Li <sub>2</sub> O	<0.01	0.03	<0.01	<0.01	<0.01
V <sub>2</sub> O <sub>3</sub>	0.01	0.01	<0.01	<0.01	0.02
CuO	0.02	<0.01	<0.01	<0.01	<0.01
Cr <sub>2</sub> O <sub>3</sub>	<0.02	<0.02	<0.02	<0.02	<0.02
Ag <sub>2</sub> O	<0.01	<0.01	<0.01	<0.01	<0.01
Total Oxides	94.8	93.1	93.5	92.6	92.7
Density, g/cc	0.97	1.11	1.17	1.17	1.17
% Moisture	0.4	5.4	7.0	11.2	13.4

previous data showing no cadmium migration (Buelt et al. 1987), it is expected that the cadmium and arsenic would have eventually been caught up by the vitrified melt or ISV off-gas system if ISV processing had continued to a greater depth. Generally, ISV processing in the field is continued to a depth 15 to 30 cm beyond the contamination depth. The behavior of cadmium and

arsenic in the surrounding soils will be further evaluated during the fourth engineering-scale ISV test on INEL soils (ES-INEL-4).

The durability of the vitrified glass block was tested using TCLP and EP Toxicity procedures. In addition, TCLP analysis was performed on a sample of the molten metal at the bottom of the vitrified block (see Figure 6.7). EP Toxicity testing was not performed on the metal slab because of the significant amount of time necessary to properly prepare the sample. Table 6.7 shows the EP Toxicity and TCLP test results for the vitrified glass and metal, compared to the EP Toxicity concentration limits. The results show that the vitrified product is an excellent waste form that would provide long-term isolation of the EP Toxicity metals present in the vitrified glass.

In addition, x-ray analysis was performed on the molten metal sample to determine if any EP Toxicity materials had reduced to metals and become incorporated in the molten metal pool. The x-ray analysis indicated that arsenic and silver were present in the metal sample. This is due to the highly reductive nature of arsenic and silver and the presence of graphite electrodes in the ISV melt. However, the high quality of the molten metal (as indicated by TCLP analysis) should prevent leaching of EP Toxicity chemicals from the molten metal pool.

TABLE 6.7. TCLP and EP Toxicity Concentrations for the BS-INEL-2 Glass and Metal

<u>Contaminant</u>	<u>EP Tox. Conc., Glass, mg/L</u>	<u>TCLP Conc., Glass, mg/L</u>	<u>TCLP Conc., Metal, mg/L</u>	<u>Allowable Conc., mg/L</u>
Ag	<0.1	<0.1	<0.1	5
As	<1	<5	<5	5
Ba	0.056	0.05	<1	100
Cd	0.35	<1	<1	1
Cr	<0.02	<1	2.7	5
Hg	<0.03	<0.03	<0.03	0.2
Ni	0.09	<1	3	5
Pb	<1	<1	<1	5
Se	<0.01	<0.01	<0.01	1

A cross section of the BS-INEL-2 vitrified block (see Figure 6.10) was analyzed under microscope to determine whether the asbestos fibers had been incorporated in the vitrified product. Imbedded in the cut surface were structures that may have been the original asbestos test material; however, these structures were no longer fibrous, nor did they have the crystallographic properties of any asbestos material. As a result, it appears that asbestos becomes safely incorporated in the vitrified product during ISV processing.

## 6.5 BENCH-SCALE TEST 3 (BS-INEL-3)

### 6.5.1 Test Configuration

The third bench-scale ISV test (BS-INEL-3) was performed to evaluate the ISV product quality of vitrified INEL soil containing materials representative of those found in Pit 9 of the INEL burial site. Pit 9 is the location for the large-scale ISV treatability test at INEL, and is estimated to contain 4 wt% metals (50 wt% carbon steel, 50 wt% ferric oxide) and 1 wt% combustibles (paper/wood). The bench-scale test was performed without the addition of sodium tetraborate to determine the product quality of vitrified INEL soil without chemical additives.

In addition, two different organic sludge/grease mixtures were mixed with cement-like materials and allowed to cure for over 12 h in a 250-mL glass sample bottle with a Teflon® lid. The sample bottle was then placed in the test soil between the two electrodes. The cemented sludge/grease mixtures were representative of those found in the volatile organic wastes that have been disposed of at INEL. The product quality evaluation included a determination of the leachability of the glass and an estimate of the amount of volatile organic migration away from the vitrified block.

Figure 6.11 shows the test configuration for BS-INEL-3. The test used a 15-cm-(6-in.-) thick layer of soil with a uniform concentration of 2 wt% carbon steel, 2 wt% ferric oxide (to simulate rusting carbon steel drums),

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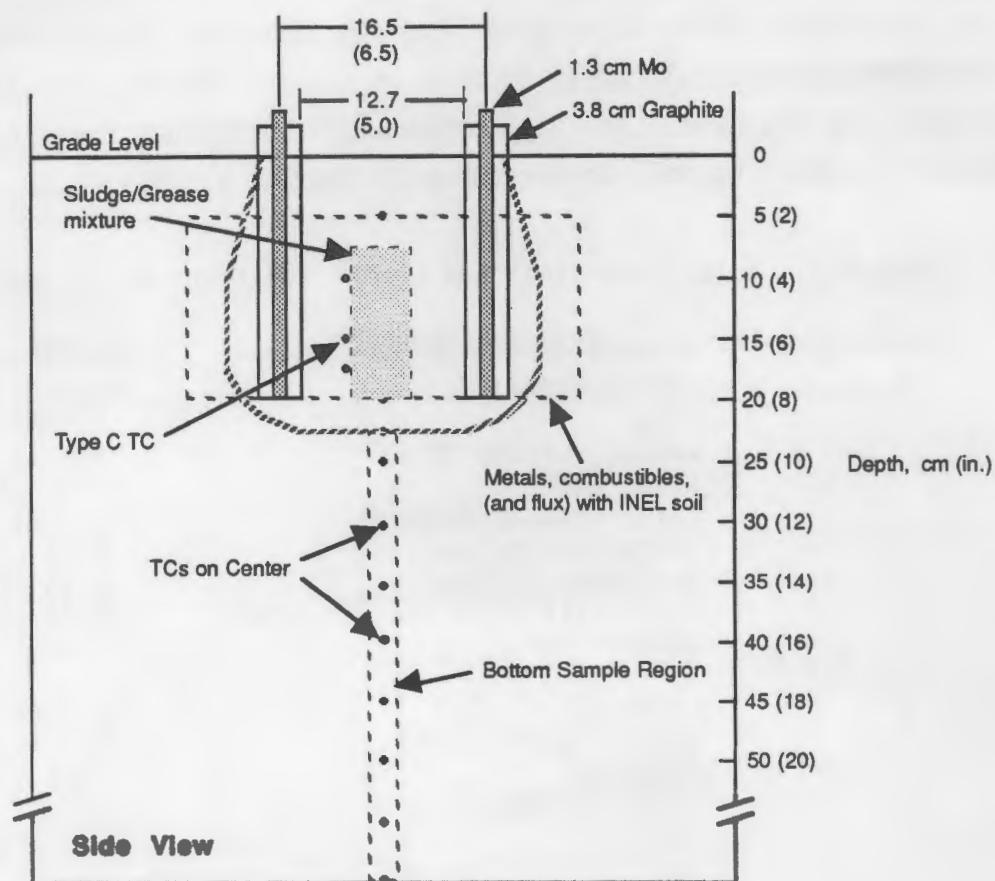
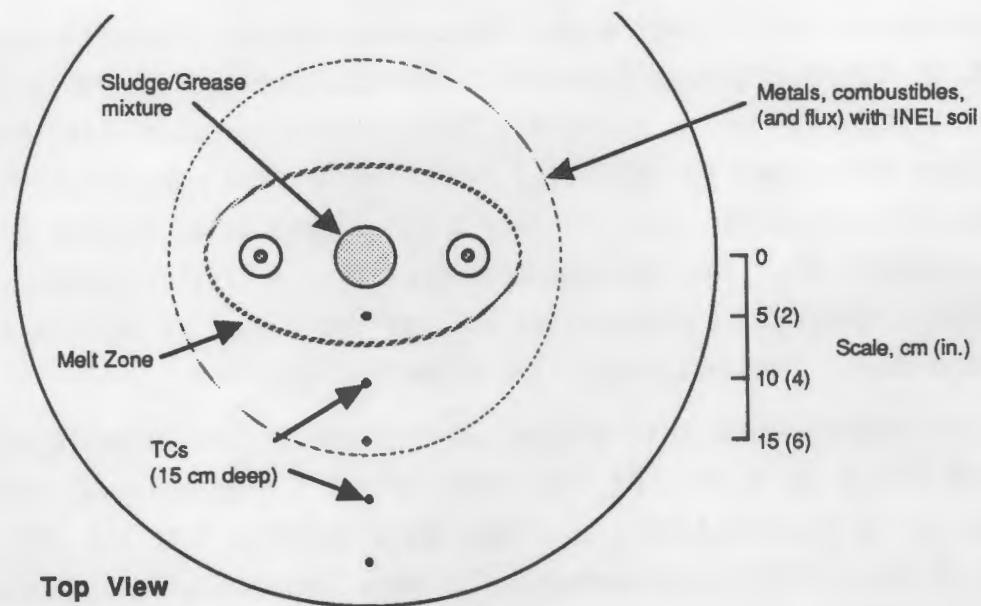


FIGURE 6.11. Thermocouple and Test Soil Locations for BS-INEL-3

0.5 wt% paper, and 0.5 wt% wood. The sample bottle containing approximately 220 mL of cemented sludge/grease mixture was placed at the 20-cm (8-in.) depth, between the two electrodes. This is in accordance with the changes that were proscribed for BS-INEL-2, with the desired concentration of cemented sludge/grease mixture (approximately 5 wt%) based on an assumed glass weight of 9.9 kg (22 lb). The type and amount of each volatile organic added to the cemented sludge/grease mixture, as well as the volume of calcium silicate and Portland cement that was added, are shown in Table 6.8.

The prepared test soil volumes were placed at the 20-cm (8-in.) depth and covered with 5 cm (2 in.) of INEL cover soil. Two molybdenum/ graphite electrodes, at 16.5 cm (6.5 in.) spacings, were inserted into the soil to a depth of 20 cm (8 in.). Type K thermocouples were incrementally positioned along the center line and side of the 55-gal drum to monitor the progress of the melt and surrounding temperature profiles. In addition, a high-temperature Type C thermocouple was placed 14 cm (5.5 in.) below the soil surface, in the radial center of the melt. The high-temperature thermocouple was used to determine the operating melt temperatures during ISV processing.

TABLE 6.8. Volatile Organic and Cement Additions to BS-INEL-3

<u>Batch No.</u>	<u>Chemical Component</u>	<u>Amount</u>
1	Texaco regal oil	13.8 ml
	CCl <sub>4</sub>	8.6 ml
	Unocal soluble 10 oil	5.6 ml
	Ethyl ether	1.95 ml
	1,1,1-Trichloroethane	1.8 ml
	Trichloroethylene	1.33 ml
	Perchloroethylene	1.33 ml
	Micro-cell (calcium silicate)	30.36 g
2	Water	141.4 ml
	Isopropyl alcohol	3.0 ml
	Glacial acetic acid	3.0 ml
	EDTA-2Na	2.59 g
	Portland cement	26.0 g
	Water	12.3 ml

### 6.5.2 Performance Evaluation

The third bench-scale test was performed on March 31, 1989. The purpose of this test was to evaluate the effect of cemented organic materials on ISV processing performance and product quality. The cemented organics were representative of those materials present in the actual INEL soils. In addition, the test evaluated ISV processability without the additions of fluxing agents to the soil. Only 4 wt% metal was added to the soil, with 50% added as carbon steel and the other 50% added as ferric oxide. The amount of metal added to the soil was representative of the concentration of metal in the Pit 9 trench at INEL.

The lowered concentration of metal in BS-INEL-3 resulted in a very smooth test operation. Surges and lapses in current were observed for only 10 min, 1.4 h after the initiating of testing. However, these surges were suspected to be primarily due to voltage arcs and steam pressurizations that occurred in the melt, as water was released from the cemented organic materials. This theory was supported by the large amount of percolation that occurred in the melt as it vitrified the cemented organic materials.

Electrical data for the third bench-scale test are presented in Figure 6.12. The test resulted in a total energy input of 17.5 kWh over 4.5 h of testing, for an average power input of 3.9 kW. The average power input for the last 3.2 h of testing was 5.0 kW.

Vitrification processing proceeded to a depth of over 30 cm (12 in.) and produced an 11.4-kg (25.1-lb) block of vitrified glass over the 4.5-h test period. The energy-to-mass ratio for the vitrified block was 1.2 kWh/kg of soil, which was just above that for typical ISV melts (0.8 to 1.1 kWh/kg). A photograph of the BS-INEL-3 test block is shown in Figure 6.13. The vitrified block was elliptical, with a maximum height of 30 cm (12 in.), and a maximum width of 28 cm (11 in.) by 20 cm (8 in.).

In contrast with the first two bench-scale tests, there were no large void volumes in the BS-INEL-3 vitrified block. This is because of the lowered concentration of metal in the melt, which eliminated the presence of a molten metal pool at the melt bottom and balanced the power distribution throughout

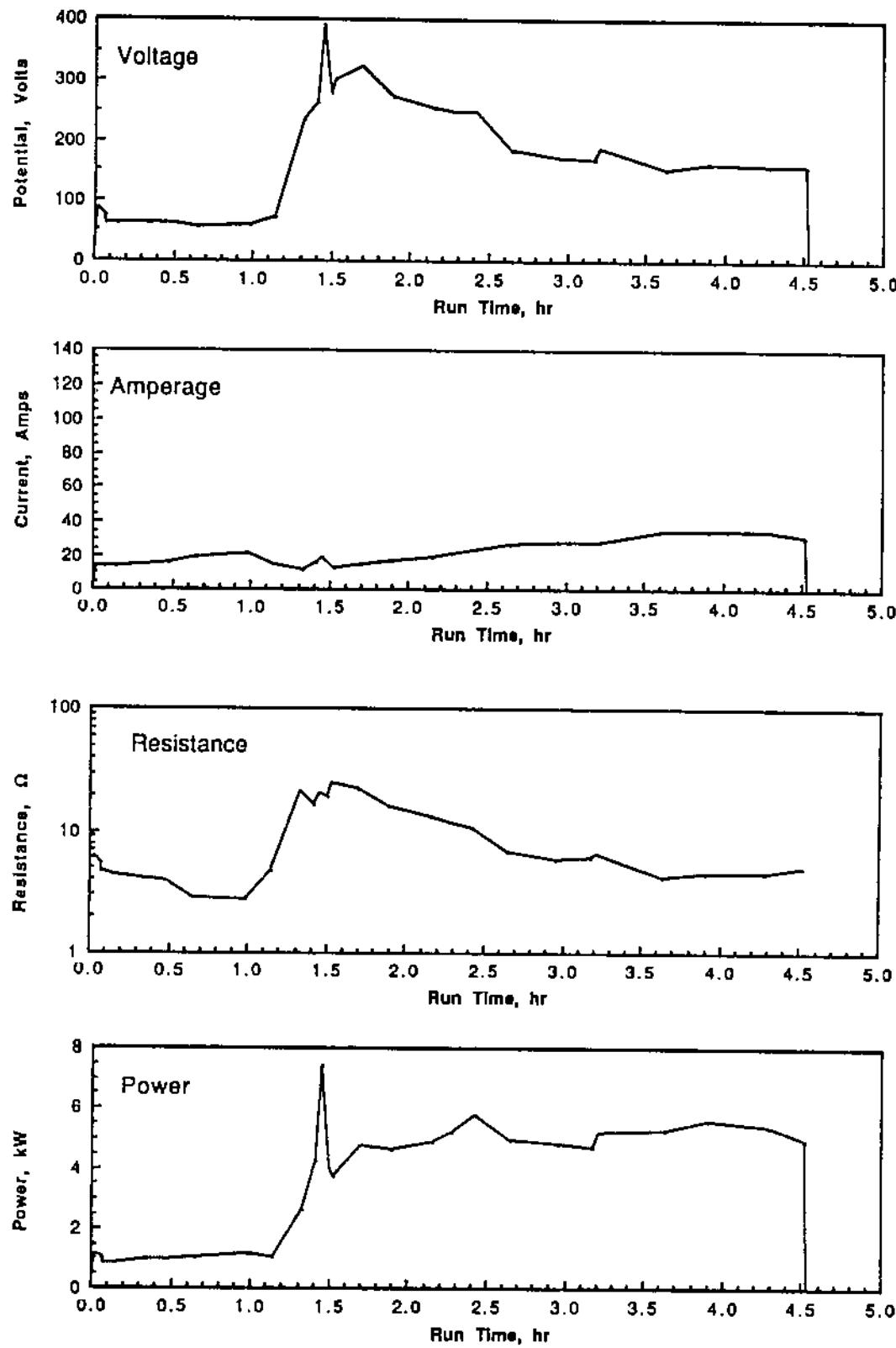


FIGURE 6.12. Electrical Data for BS-INEL-3

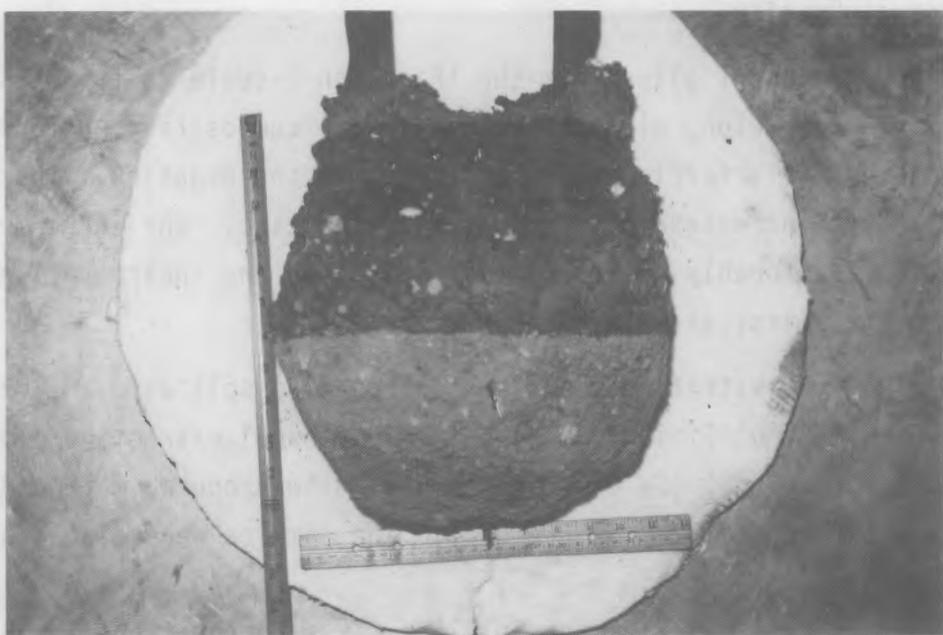


FIGURE 6.13. Vitrified Block Resulting from BS-INEL-3

the vitrified melt (instead of concentrating it around the molten metal pool). The balanced power distribution resulted in a hotter glass surface that was able to subside during ISV processing. Melt subsidence was also aided by the presence of cemented organic materials in the melt, which kept the vitrified glass surface molten during its vigorous off gassing.

During the latter portion of the test, the power level averaged 5.0 kW. Assuming an elliptical melt cross-sectional geometry and a surface cross-sectional area of  $162 \text{ cm}^2$  ( $25 \text{ in.}^2$ ), the actual power density for this test was  $308 \text{ kW/m}^2$  ( $28.6 \text{ kW/ft}^2$ ). The density is very close to the  $286 \text{ kW/m}^2$  ( $26.6 \text{ kW/ft}^2$ ) power density of the large-scale ISV system, implying that the test was run under representative conditions.

Data on operating melt temperatures for the third bench-scale ISV test were not taken. However, it is expected that the operating temperatures for the third bench-scale test were at least as high as those of BS-INEL-1 and BS-INEL-2. As a result, the vitrified glass should be of high product quality.

### 6.5.3 Product Quality

The composition of glass from the third bench-scale test (BS-INEL-3) is shown in Table 6.9, along with the original soil composition, before the addition of carbon steel, ferric oxide, and the cemented organics. The results show significant increases in iron, calcium, and zinc. The increased iron concentration is probably due to partial oxidation and incorporation of the metals into the glass, as with BS-INEL-2.

Samples of the vitrified glass and surrounding soil were analyzed for  $CCl_4$ , TCE, and PCE, utilizing the high-level methanol extraction procedure found in EPA Method 5030 (SW 846, 3rd ed.) and the proposed Method 8021 (proposed update package to SW-846, 3rd ed.). The samples were also analyzed

**TABLE 6.9. Pretest and Posttest Soil and Glass Compositions for BS-INEL-3**

Oxide	Pretest Soil	Posttest Glass
$SiO_2$	69.4	66.8
$Al_2O_3$	13.3	12.4
$Fe_2O_3$	4.41	6.24
$CaO$	2.94	4.77
$K_2O$	3.11	3.0
$Na_2O$	1.64	1.98
$MgO$	1.75	1.91
$TiO_2$	0.72	0.71
$ZnO$	<0.02	0.19
$MnO_2$	0.11	0.13
$BaO$	0.09	0.10
$ZrO_2$	0.07	0.09
$SrO$	0.03	0.03
$Li_2O$	<0.01	0.03
$V_2O_3$	<0.02	0.015
Total Oxides	97.6	98.5
Density (g/cc)	1.25	2.38

for a number of additional volatile organic chemicals as part of Method 8021. Analytical results for the vitrified product sample and the five surrounding soil samples are shown in Table 6.10.

The vitrified product sample was analyzed to verify that there were no residual amounts of the volatile organics left in the vitrified block. This was confirmed by the results (see Table 6.10), which showed that the concentrations of  $\text{CCl}_4$ , TCE, and PCE were below the detection limits of 0.06 ppm. As a result, it is recommended that future engineering-scale tests need not look for the presence of volatile organics in either the vitrified glass and crystalline product or the vitrified metal product.

Samples of the soil beneath the vitrified block were analyzed to determine if there was any thermal transport of volatile organics to the surrounding soil during ISV processing. The soil samples were taken near the edge of the vitrified block and at four locations where the soil reached maximum temperatures of 250°C, 100°C, 70°C, and 40°C, respectively. These four soil locations were approximately 5 cm (2 in.), 13 cm (5 in.), 25 cm (10 in.), and 36 cm (14 in.) away from the bottom surface of the vitrified product. Results from Table 6.10 showed that there was some PCE and (to a lesser extent) TCE in the surrounding soils less than 5 cm (2 in.) away from the melt. However, the concentrations of PCE and TCE were less than 1 ppm, and 0.1 ppm, respectively. This is 10,000 to 100,000 times less than the concentrations of PCE (10,200 ppm) and TCE (9,200 ppm) present in the original cemented sludge/grease mixtures. Based on EPA's definition of de minimus exemption levels for nonspecific sources [40 CFR 261.31 (under F0002)], the low concentrations of

TABLE 6.10. Glass and Surrounding Soil Analysis for BS-INEL-3 (ppm)

<u>Contaminant</u>	<u>Glass</u>	<u>Melt Edge</u>	<u>250°C Isotherm</u>	<u>100°C Isotherm</u>	<u>70°C Isotherm</u>	<u>40°C Isotherm</u>
PCE	bd <sup>(a)</sup>	0.46	0.84	bd	bd	bd
TCE	bd	bd	0.06	bd	bd	bd
$\text{CCl}_4$	bd	bd	bd	bd	bd	bd

(a) bd = below detection limits (0.06 ppm).

PCE and TCE in two of the five surrounding soil samples are not high enough to designate the soils as a hazardous waste material.

The analytical results from the surrounding soil samples must be interpreted in light of the fact that the sampling method may not have been totally appropriate for these constituents. With volatile organics, it is generally necessary to pack the sample container with soil before it is sealed and refrigerated to eliminate any plenum space in the sample container where any trace organics may collect. Unfortunately this was not a part of the sample collection procedures for this test. Improved methods for surrounding soil sampling will be included in the fourth engineering-scale ISV test.

The durability of the vitrified glass block from BS-INEL-3 was tested using TCLP and EP Toxicity procedures. Table 6.11 shows the EP Toxicity and TCLP test results for the vitrified glass and metal compared to the allowable EP Toxicity concentration limits. The results show that the vitrified product is an excellent waste form that would provide long-term isolation of the EP Toxicity metals present in the vitrified glass.

## 6.6 COMBINED BENCH-SCALE TEST EVALUATION

Performance data showing the differences between the three bench-scale tests are summarized in Table 6.12. The data show that the mass vitrified and

TABLE 6.11. TCLP and EP Toxicity Concentrations for the BS-INEL-3 Glass

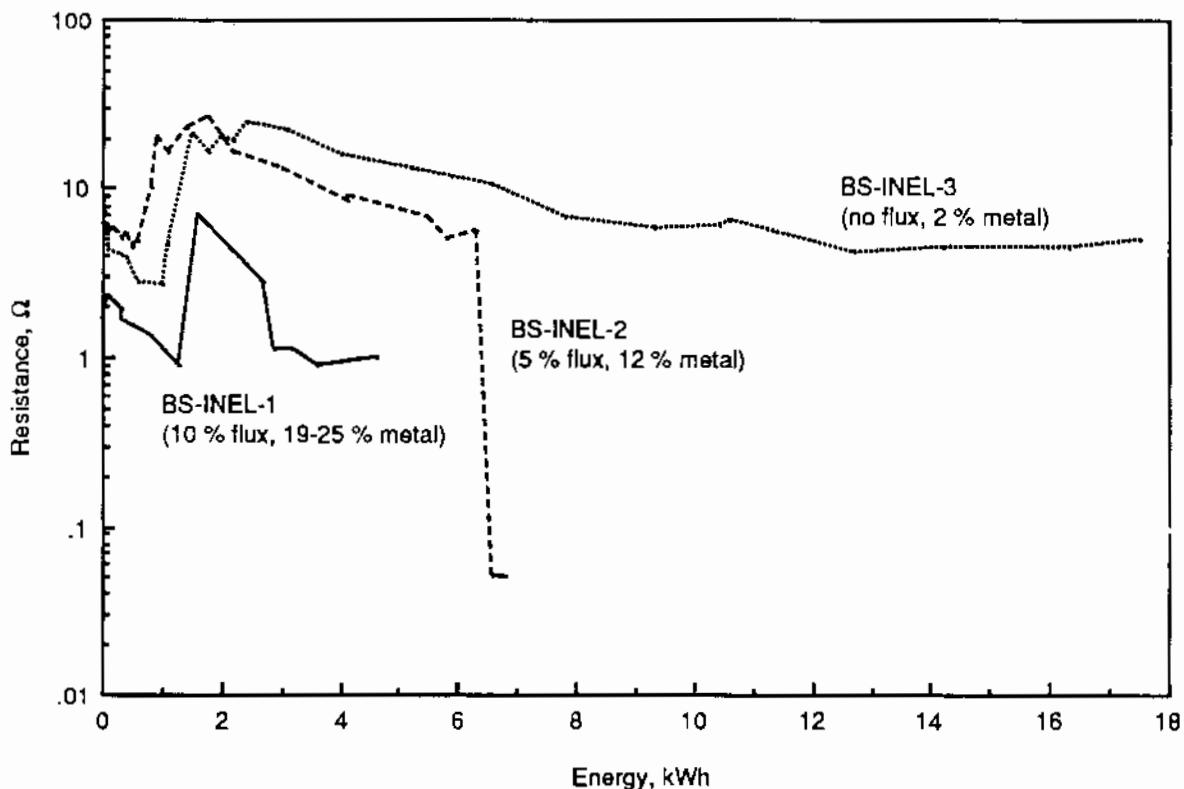
<u>Contaminant</u>	<u>EP Tox Conc., mg/L</u>	<u>TCLP Conc., mg/L</u>	<u>Allowable Conc., mg/L</u>
Ag	<0.1	<0.1	5
As	<1	<5	5
Ba	0.011	<1	100
Cd	<1	<1	1
Cr	<0.02	<1	5
Hg	<0.03	<0.03	0.2
Ni	0.02	<1	5
Pb	<0.06	<1	5
Se	<0.01	<0.01	1

TABLE 6.12. System Performance During Bench-Scale ISV Testing

Parameter	BS-INEL-1	BS-INEL-2	BS-INEL-3
Total run time	3.15 h	2.8 h	4.5 h
Vitrified glass wt	2.04 kg	4.54 kg	11.4 kg
Soil vitrified (est.)	2.46 kg	5.60 kg	14.1 kg
Melt depth	20+ cm (8+ in.)	25+ cm (9+ in.)	30+ cm (12+ in.)
Melt width	11 cm (4.5 in.) x 6 cm (2.5 in.)	23 cm (9 in.) x 15 cm (6 in.)	28 cm (11 in.) x 20 cm (8 in.)
Average power	1.46 kW	2.44 kW	3.89 kW
Total energy	4.6 kWh	6.8 kWh	17.5 kWh
Energy-to-mass ratio	1.9 kWh/kg	1.2 kWh/kg	1.2 kWh/kg
Nominal metals content	19-25 wt%	11.6 wt%	(+2 wt% oxidized metal)
Sodium concentration	9.8 wt%	5.4 wt%	1.5 wt%

average power of each bench-scale test were lower for those tests that had high concentrations of metal and sodium (from sodium tetraborate additions) in the vitrified block. This was expected, since high concentrations of metals will result in pooling of the molten metals between the electrical phases (leading to full and partial shorts), while high concentrations of sodium tetraborate will significantly reduce glass resistivity. This is confirmed by Figure 6.14, which gives the effective resistance of each melt as a function of its total energy input. The low resistance of BS-INEL-1 was primarily due to the presence of 19 to 25 wt% metal and 9.8 wt% sodium in the vitrified block. A similar, though less significant decrease in glass resistivity was noted for BS-INEL-2 when compared with the resistivity of BS-INEL-3. The decreased resistivity can decrease the maximum power input that is possible if the current reaches the maximum transformer rating, and the voltage is still below the transformer rating.

The energy-to-mass ratios for BS-INEL-1, -2, and -3 were 1.9 kWh/kg, 1.2 kWh/kg, and 1.2 kWh/kg, respectively; energy-to-mass ratios for most soils are typically in the range of 0.8 to 1.1 kWh/kg. The high energy-to-mass ratio in BS-INEL-1 and slightly increased ratios in BS-INEL-2 and -3 were probably due to the lowered power input that was caused by metal and sodium



**FIGURE 6.14.** Electrical Resistance Versus Energy Input for the Bench-Scale ISV Tests

tetraborate concentrations in the three melts. As the average power input decreased, the fraction of that power associated with heat losses increased, increasing the energy-to-mass ratio of the vitrified mass.

The resultant shape of the vitrified glass blocks that were produced during bench-scale ISV testing appeared to also be affected by metals concentration. In particular, the vitrified blocks produced from bench-scale ISV tests with high concentrations of metal (BS-INEL-1 and BS-INEL-2) did not exhibit as much horizontal melt growth as the ISV computer model predicted. It appears that this is caused by the presence of a molten pool of metal at the bottom of the melt, which enhances the downward melting rate while limiting horizontal melt growth. The presence of molten metal pools near the bottom of the melt results in a less resistant electrical path between electrodes. The electrical current is then focused at this point of least resistance, which creates a higher power density in the glass near the bottom

of the melt, enhancing the downward growth rate. The increased downward growth rate with high metals concentrations is confirmed by Figure 6.15, which shows the melt depth for all three bench-scale tests as a function of total energy input.

The effect of metals on downward melt growth can also be used to explain the large voids that were formed in the vitrified glass blocks from the first and second bench-scale tests (BS-INEL-1 and BS-INEL-2). The void formation in these melts is a combination of the presence of molten metal pools in the melt and the large reduction in volume (approximately 45%) that is expected to occur in INEL soils. The presence of a metal pool at the bottom of the melt focuses the power distribution toward the melt bottom. As a result, less power is available to keep the glass fluid at the tip of the melt, causing the melt surface to harden faster than normal. The combination of rapid downward melting with accelerated melt surface cooling results in the creation of the large void area in the block as the melt solidifies.

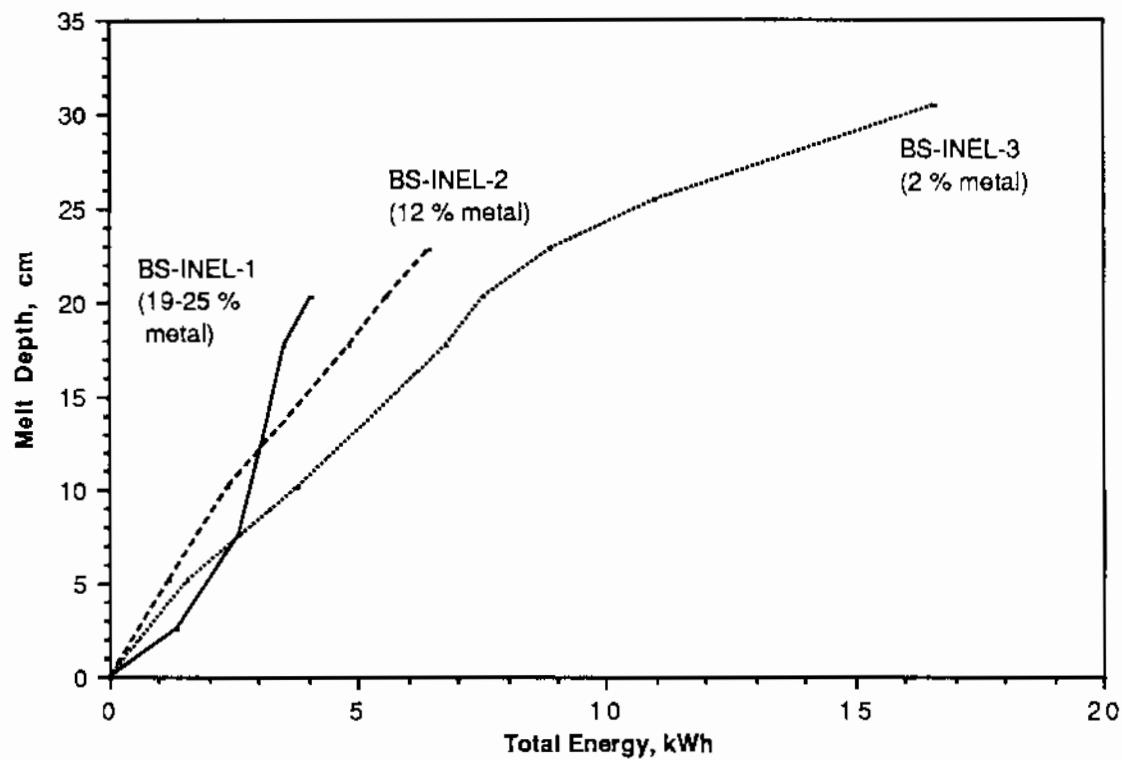


FIGURE 6.15. Melt Depth Versus Energy Input for Each Bench-Scale ISV Test



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