FLUOR DANIEL FERNALD PROOF-OF-PRINCIPLE TEST OF JOULE-HEATED VITRIFICATION

POPT WORK PLAN
FINAL VERSION

Envitco Project #98703
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ACRONYMS

ANS   American Nuclear Society
ASTM  American Society for Testing and Materials
CELS  CELS – Coming Laboratory Services
CERCLA Comprehensive Environmental Response, Compensation and Liability Act
CETL  Clemson Environmental Technologies Laboratory
CFR   Code of Federal Regulations
COA   Certificate of Analysis
COC   Chain of Custody
CSTR  Continuous-stirred Tank Reactor
CWA   Clean Water Act
DOE   Department of Energy
DWPF  Defense Waste Processing Facility
EPA   Environmental Protection Agency
FDF   Fluor Daniel Femald
FEMP  Femald Environmental Management Project
LOD   Loss on Drying
LOI   Loss on Ignition
MSDS  Material Safety Data Sheet
OU4   Operable Unit 4
PFD   Process Flow Diagram
PNNL  Pacific Northwest National Laboratory
POPT  Proof of Principle Test
QA    Quality Assurance
QC    Quality Control
QCA   Quantitative Chemical Analysis
RCRA  Resource Conservation and Recovery Act
RFP   Request for Proposal
scfm  standard cubic feet per minute
SEM   Scanning Electron Microscope
SLS   Soda-lime-silica
SLLS  Soda-lime-lithia-silica
SOP   Standard Operating Procedure
SOW   Statement of Work
SRTC  Savannah River Technology Center
STP   Standard Temperature and Pressure
TCLP  Toxicity Characteristic Leaching Procedure
TD&D  Technology Demonstration and Development
TDS   Total Dissolved Solids
TECO  Toledo Engineering Company
TPD   Tons Per Day
ACRONYMS (cont'd)

TSS  Total Suspended Solids
TVS  Transportable Vitrification System
UTS  Universal Treatment Standards
WAC  Waste Acceptance Criteria
XRD  X-Ray diffraction
1.0 INTRODUCTION

Section C.3.2 of Envitco's Contract Item 001 (FDF Contract No. 98WO002240) requires three (3) activities that must occur prior to the start of any Proof of Principle testing. During the planning phase of the project, (1) a Work Plan and (2) a Quality Assurance/Quality Control (QA/QC) Plan must be developed. Also, (3) the chemical compounds required for the POPT must be purchased and verified.

Section C.3.2.1 of Envitco's Contract Item 001 requires the development of the Work Plan in accordance with the guidance provided in Section C of the Contract. The Work Plan, as developed by Envitco, will be approved by FDF prior to the initiation of any testing. This Work Plan, presented herein, will address the following topics:

- Testing,
- Equipment and materials,
- Sampling,
- Analysis,
- Treatment recipe development,
- Data collection, management, and evaluation,
- Health and safety requirements,
- Waste stream management,
- Reporting,
- Schedule,
- Management and Staffing, and
- Regulatory Compliance.

All activities that are described in this Work Plan will be performed in accordance with QA Impact Level C requirements as defined in the Envitco POPT QA Plan.

1.1 Project Description

1.1.1 Purpose of POPT

The purpose of the POPT is to perform rigorous testing of proven and commercially available remediation technologies to evaluate their potential use for treatment of Silos 1 and 2 residues of Operable Unit 4 (OU4) at the Fermald Environmental Management Project (FEMP). The testing shall focus on meeting the regulatory, processing, storage, transportation and disposal requirements of the Silos 1 and 2 residue. The results of this testing will provide FDF with information on safety, reliability, implementability, cost, and schedule for the...
tested technology. Additionally, this information will be used to support revision of the OU4 Feasibility Study and support the OU4 Record of Decision amendment.

This POPT will employ a mature Joule-heated melter technology in an effort to successfully meet the requirements of the contract between FDF and Envitco, and meet the test specific objectives outlined in Section 1.2 of this Work Plan. Specific issues related to the application of this technology for treatment of the Fernald Silos 1 and 2 waste have been identified. Addressing and resolving these issues through testing with non-radioactive surrogate materials are objectives of the POPT.

1.1.2 Waste Characteristics

The Silo residue is part of OU4 at the Department of Energy (DOE) FEMP in Cincinnati, Ohio. Silos 1 and 2 contain approximately 6,100 cubic meters of residue classified as byproduct material defined under Section 11(e)(2) of the Atomic Energy Act of 1954, as amended. Under this classification, the Silo residue is exempt from regulation by the Resource Conservation and Recovery Act (RCRA).

However, in order for it to be disposed of off-site, the waste must not exhibit a hazardous characteristic. The Silo residue exhibits the characteristic of toxicity due to lead levels in excess of the limits defined in 40 CFR 261. Other significant metals include barium, arsenic and uranium. The Silo residue must be treated so that it no longer exhibits a hazardous characteristic.

The Silo residue is primarily a wet, silty clay with significant concentrations of Ra-226, Pb-210 and Th-230. The Silo 1 and 2 residues also contain a significant amount of sulfur and phosphorous. Radium-226 and the daughter products resulting from its decay, primarily Radon-222, are the primary radiological concern. Radon is relatively mobile and capable of migration through air and water. The radon and lead are therefore the key components to be immobilized prior to disposal.

As part of the Silos 1 and 2 Removal Action under CERCLA, a layer of BentoGrout™ was placed over the residue. This cap was designed to attenuate Radon-222 emissions from the Silos. There are approximately 380 cubic meters of BentoGrout in Silo 1 and 300 cubic meters in Silo 2. The total volume of material to be treated is approximately 6,780 cubic meters.
1.1.3 POPT Facilities Description

The POPT demonstration will be performed at Clemson University's Clemson Environmental Technologies Laboratory (CETL) in Anderson, South Carolina. This facility has been used for numerous other vitrification demonstrations for the Department of Energy, Envitco and Envitco's customers. The CETL facility includes laboratories for crucible melting, bench-scale operations areas, pilot-scale process demonstration areas suitable for the POPT melter operations, and office space for both CETL staff and visiting staff from Envitco.

The facilities used for the full-scale design data effort will be the offices of Envitco, Toledo Engineering Company (TECO), SGN, and Cogema Engineering. Envitco is co-located with TECO in Toledo, OH and will be directly supervising the preliminary design data development for the full-scale Joule-heated melter from their common location. Envitco will also be supervising the layout, material handling, offgas treatment system design and radon control studies conducted by SGN from the Toledo office.

1.1.4 POPT Deliverables

The deliverables generated by Envitco as a result of this POPT will consist of the following:

- Work Plan,
- Envitco POPT QA Plan,
- Weekly reports and telephone conferences,
- Compound Assays,
- Compound Sieve Tests for the materials,
- Prepare surrogates for S1, S2 and DS within specifications and provide samples to FDF,
- Treatment recipes for each of the surrogate formulas,
- Video Tapes of the Demonstration Run,
- Testing documentation and analytical data packages,
- Samples for FDF durability testing and archiving,
- Draft final report including summary design data, and
- Final Report.
1.2 Test Objectives

Specific test objectives of the POPT Demonstration are:

- Perform a demonstration to be run continuously under steady-state conditions over a 72-hour period with no more than 3.5 hours of down-time without melter feeding.

- Prepare a minimum of 5 batches of demonstration surrogate slurry prior to initiation of the 72-hour test.

- Maintain an average target processing rate equivalent to 2,600 kilograms of surrogate slurry (30 wt.% solids) per 24-hour period during the demonstration.

- Operate under conditions that minimize the production of secondary wastes (metals, salts, non-recyclable offgas treatment residues, and wastewater).

- Evaluate the ability to recycle condensed offgas blowdown liquids and scrubbed offgas solids to the melter feed for purpose of secondary waste minimization.

- Collect samples prior to, during, and after the demonstration in accordance with Section 7.0 - Sampling, Data Collection, and Analysis Plan of this Work Plan.

- Analyze representative samples of the treated surrogate in accordance with Section 7.0 - Sampling, Data Collection, and Analysis Plan of this Work Plan.

- Conduct the POPT demonstration in a manner similar to that proposed for the full-scale remediation facility.

- Supply the data requested in Appendix F, Table F1, of the Contract to FDF as part of the Final Report. This data will, at a minimum, include:
  - Process Description including PFD and catalog cut sheets,
  - Mass and Energy Balance for the pre-process batch preparation, in-process vitrification, post-process offgas treatment, drained metals and/or salts, secondary wastes and glass handling systems. The mass and energy balance will address temperatures, pressures, composition, flow rates, and production rates of the system inputs and outputs,
- Production rate calculations including treated surrogate slurry throughput and a schedule of any unplanned downtime with a discussion of the cause of the downtime, and

- Secondary waste streams characterization as described in Section 7.0 - Sampling, Data Collection, and Analysis Plan.

- Provide preliminary full-scale design data identified in Table F2 of the contract.

- Provide to FDF all deliverables as described in Section 1.1 of this Work Plan.
2.0 TREATMENT TECHNOLOGY DESCRIPTION

The treatment technology to be demonstrated by Envitco for the Fernald Silos 1 and 2 POPT demonstration is the Envitco WASTE-VIT® EV-101 which is a Joule-heated, molybdenum electrode, ceramic refractory-lined glass melter. The basic technology is a mature technology widely used in the commercial glass industry. The melter has a design melting capacity of two (2) tons of glass per day operating with dry feed.

Specific Envitco melter design features which are considered relevant to treatment of the Fernald Silo 1 and 2 wastes include:

- High temperature capability to process a range of durable glass formulations with high waste loadings,
- Operating versatility allowing the melter to be run in a cold-top, warm-top, or hot-top mode of operation with either slurry or dry feeds,
- The ability to collect and control the discharge of metallic sludge from the melter,
- The ability to collect and control the discharge of molten salts (sulfates, chlorides, etc.) from the melter, and
- A water-cooled shell and refractory configuration.

The upper melting temperature limit for the melter is ~1500°C. The target operating temperature is 1250 – 1350°C. Lower temperatures may occur adjacent to the refractories depending on the refractory type. However, the refractories operate somewhat cooler than the maximum center glass temperature as a result of water-cooling the external surface. A variety of refractories will be used in the POPT melter to allow the performance of different refractories to be compared. The refractory performance will be evaluated with results and conclusions provided in the Final Report.

For the POPT, the melter will be operated with a partial to full cold-top batch coverage over the melt pool. Cold-top operation will reduce the volatilization of hazardous waste species such as lead and will also reduce volatility losses of other waste and additive components such as alkali metals and boron oxide. Most volatilized species and entrained solids will be removed from the offgas by the offgas treatment system. A preliminary estimate of the quantity of solids to be removed by the offgas system is 1 to 2% of the feed solids content based on operating experience from the Transportable Vitrification System (TVS) (Bechtel, 1998). Blowdown liquids and solids removed by the offgas treatment system can...
potentially be recycled directly to the melter feed. Recycle of these streams from the offgas system will not be demonstrated during the POPT but will be evaluated as part of the full-scale data evaluation. The principal limitation on the ability to recycle the blowdown and solids from the offgas treatment system is the buildup of species that have limited solubility in the product glass. For the Silo 1 and 2 wastes, the component most likely to limit the ability to directly recycle these offgas waste streams is sulfate.

A process challenge for the Silos waste is the potential for PbO-Mo redox reactions to occur between PbO in the glass melt and the molybdenum components of the melter resulting in accelerated electrode wear and the precipitation of metallic Pb in the melter. Controlling melt redox so as not to precipitate Pb metal in the melter will be a process control objective during the POPT.

The melter is equipped with a bottom drain for the discharge of metals should a significant amount of Pb accumulate in the bottom of the melter. It is planned to operate the bottom drain during a late stage of the POPT to determine the amount of any precipitated metal (Pb) present. The bottom metals drain is considered an important feature for processing the Silos 1 and 2 waste because of the potential for metallic lead precipitation, which if allowed to accumulate could eventually lead to electrical shorting and/or melter refractory failure.

The melter also incorporates a side bay “salt tap” which provides the ability to skim and drain any phase-separated layer that may accumulate on top of the glass melt surface. Typical waste components that may be present at concentrations in excess of their solubility limits in glass and may phase segregate to the melt surface include sulfate, phosphate and halides. The resulting phase-separated layer would typically have a low viscosity, be very corrosive to the refractories, and have significant negative impacts if allowed to accumulate.

Sulfate is the only component identified in the Silo 1 and 2 waste compositions that may likely require operation of the salt drain. The salt drain will only be operated if it becomes necessary to remove an accumulation of phase-separated material from the glass surface. The material drained by the salt tap cannot be directly recycled to the melter without further treatment and would therefore likely represent a secondary waste stream. The maximum amount of sulfate containing material that may require draining is estimated to be ~4% of the product glass weight assuming all the sulfate in the S0-D surrogate formulation at 70 wt.% waste loading separated to the salt layer as CaSO₄. The actual amount of sulfate salt is expected to be less than this amount depending on the SO₃ content of the glass and the amount of SO₂ volatilization.
If significant secondary waste streams (metals, salts or offgas treatment residues) that cannot be directly recycled to the feed are produced, they may require additional treatment for recycle or disposal. Expected constituents and quantities of the secondary wastes produced and process options for the minimization or potential elimination of these wastes will be addressed in the Final Report. The development of atypical treatment methods for full-scale secondary wastes is beyond the scope of the current POPT.

The melter features a water-cooled shell on the external faces of the sides and bottom of the glass contact refractories. The water-cooled shell mitigates the risk of a glass leak and reduces refractory wear. The shell and refractories are designed so that glass will freeze prior to reaching the cooling shell. A frozen glass layer will form on the inner surface of the refractories in areas where significant wear or thinning of the refractories occurs, which will limit further refractory wear and glass penetration past the refractories.

The effectiveness of the refractory and shell system design was demonstrated during the 1997 TVS mixed waste demonstration at Oak Ridge (Bechtel, 1998). During this demonstration the melter was operated for over four months from late July through October 1997. The melter was partially drained at the end of the demonstration and the refractory inner surfaces below the melt line were examined. No significant visible corrosion or wear of the exposed refractories was observed.

The melter has a separate drain bay connected to the main melter chamber by a submerged throat which prevents any unmelted feed, or phase-separated salts or metals from the top or bottom of the melt pool, from entering the drain bay. Power and temperature in the main melting chamber are independently controlled to allow temperatures and viscosities to be separately optimized for melting and glass pouring. The glass pouring rate is controlled by heating or cooling the drain orifice with a backup mechanical drain control capability provided for emergency shut off.
3.0 PROOF OF PRINCIPLE TREATMENT RECIPE DEVELOPMENT

Two treatment recipes are to be developed for each of the three waste surrogate compositions provided in the Contract SOW as revised by Contract Modification No. 01 and Contract Modification No. 03. These treatment recipes are defined as follows:

- Treated Silo 1 waste surrogate that meets the following leaching requirements:
  1. Less than 50% of the TCLP limits (Glass S1-T), and
  2. Less than the UTS limits (Glass S1-U);

- Treated Silo 2 waste surrogate that meets the following leaching requirements:
  1. Less than 50% of the TCLP limits (Glass S2-T), and
  2. Less than the UTS limits (Glass S2-U);

- Treated Demonstration Surrogate - a combination of Silo 1 simulant spiked with heavy metals present in Silo 2 residue - that meets the following leaching requirements:
  1. Less than 50% of the TCLP limits (Glass S0-D), and
  2. Less than the UTS limits (Glass S0-U).

3.1 Facilities Utilized for Development Work

The surrogate and slurry feed preparation activities and the glass recipe laboratory development work will be performed at the Clemson Environmental Technologies Laboratory (CETL). The Femald POPT 72-hour melting demonstration trial will also be performed at CETL. Suitable equipment and quality systems are in place to ensure accuracy in preparation and sample integrity for this development work. The glass recipe laboratory has a high temperature Deltech furnace for performing the crucible melts. An equipment list is provided in Section 6.0 of this Work Plan for the recipe development work.

The Clemson staff will provide laboratory assistance, but all of the development work, including the glass fabrication experiments, will be conducted by and/or supervised directly by Envitco engineers. Envitco will develop candidate formulations for testing, perform data evaluation, and be responsible for final recipe selection and reporting. Supporting glass analyses will be performed by CELS - Corning Laboratory Services (CELS). Compuchem will be used for final TCLP measurements. Other laboratories may be used for non-critical screening test measurements.
3.2 Experimental Plan Overview

The basic approach to the POPT glass formulation is as follows:

- Choose initial glass recipes based on previous formulations found in literature that exhibit similar leachability and pre-defined processability requirements,
- Melt first series of glasses using waste loading vs. alkali/alkaline additives,
- Characterize the visually acceptable glasses (glass homogeneity, viscosity, etc.),
- Perform second series of melts to determine sensitivity to redox state,
- Define glass formulations.

Figure 3.2-1 shows an overview of the experimental plan to develop the recipes described in Section 3.0. Each box has been labeled with a number. This number corresponds to the following sections which describe the recipe development work plan.
Figure 3.2-1 Glass Recipe Development Flow Diagram

1. Review past silo Glass Formulation
2. Define Processing Requirements
3. Select Glass System(s) for initial testing (SLS and SLLS)
4. First Series Melts (3-5 alkali/alkaline levels for S0-D, S1-T and S2-T)
5. Select best glasses (Visc, Redox, TCLP)
6. Second Series Melts (Vary Redox Additives)
7. Define S0-D, S1-T and S2-T Recipes
8. POPT Demo
9. Prepare Surrogates
10. Prepare dry S0, S1 and S2 surrogate batches
11. Melt Additional Formulations
12. Reject/Eliminate Formulation
13. Additional Melts - Formulated for UTS Durability
14. Define S0-U, S1-U and S2-U Recipes
15. Prepare Treatment Recipe Samples for S0, S1, S2 Glasses

Severe foaming, or phase separation

Little/no foaming or phase separation, acceptable viscosity

Pass TCLP

Fail TCLP
3.3 Surrogate Preparation and Validation

Three activities will be performed prior to the initiation of the crucible scale glass melting activities. These activities include the confirmation of surrogate compounds, validation of the surrogate recipe mixes, and preparation of surrogate batches for treatment recipe development.

3.3.1 Compound Assays and Material Certificates

Envitco will purchase the chemical compounds required for the POPT in accordance with the specifications in Table C4 of the Contract SOW and will provide material quality documentation as described in Section C.2.3 of Contract Item 001. Envitco will provide to FDF the vendor-supplied material assays and the CELS sieve analyses results of the silica at least two (2) weeks prior to the initiation of testing. Envitco will not perform additional analyses or testing of chemicals or materials purchased with supplier certifications.

An inspection of the vendors assays will be conducted prior to procurement in order to ensure proper purity. An inspection of the materials upon receipt will also be performed to establish that it is the proper compound and has an MSDS with the shipment.

3.3.2 Surrogate Mix Validation

Envitco will prepare each of the three (3) surrogates as a blended mixture with 30 wt.% moisture in accordance with instructions and recipes provided in Modification No. 01 of the Contract (Box No. 9A in Figure 3.2-1). This activity will provide assurance that the raw materials and resulting surrogate mixes used for the treatment recipe development work and the POPT demonstration will yield credible data. A three-liter sample of each 30 wt.% moisture mix will be made available to FDF for verification against the Contract requirements. FDF approval is required prior to initiating the preparation of the recipe development surrogates (Box No. 9 of Figure 3.2-1). Samples will also be archived at the CETL facility for reference. FDF will respond within seven (7) working days of receipt of the slurry sample at the analytical laboratory.

The following steps, as provided in Contract Modification No. 01, will be followed to prepare the 30% water surrogates:

1. Weigh out the dry chemicals and place in suitably sized container.
2. When all powdered chemicals, other than the organics, have been added, blend well. Break up all lumps so that no lump is larger than that of the coarsest particle i.e., the coarse silica.

3. Add the organics and continue blending. Note: The organics must be completely mixed with the dry chemicals and absorbed by them.

4. Add the 30 wt.% water and mix thoroughly. Note: The finished product should ball like clay at this moisture content. A sheen of organics on the surface of the mixture is typical.

5. Collect samples for surrogate preparation validation testing and analysis.

The 30 wt.% moisture surrogates will not be made into 70 wt.% moisture surrogates because the crucible melting work will be performed with dry materials as noted in the following section.

3.3.3 Surrogate Preparation for Crucible Testing

As shown in Figure 3.2-1 Box No. 10, the preparation of the crucible testing surrogate will occur after FDF’s approval of the 30 wt.% moisture surrogate. The treatment recipe surrogate blend will be prepared from the dry raw materials used in the proportions confirmed in the surrogate mix validation exercise. All treatment recipe work will be conducted using only dry surrogate and not the 30 wt.% moisture mix. Master batches of each of the dry mix surrogate recipes (including BentoGrout additions) will be prepared to minimize variations in surrogate mix compositions used in different crucible melts. These larger master batches will be split into smaller units using sample splitters (rifflers) to avoid component segregation problems. Additive weights for individual crucible melts will be normalized to match the actual split surrogate weights. Glass-making additives will be blended with the dry surrogate splits for glass formulation melts.

3.4 Glass Formulation Issues and Targets

3.4.1 Previous and Target Glass Formulations

Initial literature review has indicated that durable glasses can be formulated in the soda-lime-silica (SLS) and lithia (Li2O) substituted SLS (SLLS) glass systems with high waste loadings on the order of ~80 wt.% (Box No. 1 in Figure 3.2-1). These glass systems can accommodate the levels of PbO, BaO and CaO contained in the three surrogate compositions and appear to present a lower risk of phase separation than expected in the borosilicate glass system. There is also a considerable amount of experience successfully processing SLS and SLLS mixed waste formulations in the TVS and other Envitco melters.
Therefore, the initial formulations for testing will be developed in these glass systems. It is anticipated that the only glass-making additives that will be used in the initial crucible melt series will be Na$_2$CO$_3$, Li$_2$CO$_3$ and CaCO$_3$. Additives initially used for redox adjustment will be NaN$_3$ as an oxidant and sugar and/or powdered carbon as reductants. Additions of Al$_2$O$_3$ may be evaluated in later formulations as a means to increase the durability to meet the UTS limits.

The primary concern for the glasses to pass the TCLP is the release of Pb. The other metals of interest are easily incorporated into the glass with low leaching characteristics. A SLS glass prepared by Pacific Northwest National Laboratory (PNNL) by adding Na$_2$O to a K-65 Silos 1 and 2 surrogate composition passed TCLP with Pb releases measured in the 0.81 to 1.2 mg/L range compared to the POPT limit of 2.5 mg/L (Merrill, 1993). A SLLS glass prepared by Savannah River Technology Center (SRTC) to melt at 1050°C added only Na$_2$O and Li$_2$O to a K-65 (Silos waste) surrogate composition and passed TCLP with Pb releases measured in the 1.27 to 1.62 mg/L range (Jantzen, 1998). The ability to formulate a higher melting temperature SLLS glass that meets the 0.75 mg/L UTS release limit for Pb, using less alkali addition than the low melting SRTC formulation, and possibly added Al$_2$O$_3$, is a reasonable expectation.

3.4.2 Processing Requirements and Targets

In addition to the glass durability objectives identified in Section 3.0, there are several processability issues (Box No. 2 of Figure 3.2-1) that need to be addressed in the treatment recipe development and during the actual POPT demonstration. The principal processability issues to be considered are:

- Composition control within a durable glass forming envelope,
- Glass melting temperature and viscosity,
- Liquidus temperature,
- Glass redox potential as measured by the Fe$^{+2}$/ΣFe ratio,
- Glass solubility limits for waste components, and
- Materials compatibility.

Preliminary target ranges for glass melting temperature, melt viscosity, liquidus temperature, and redox are summarized in Table 3.4-1. The rationale for choosing these target property ranges as a starting point for the treatment recipe development activities is provided in the following discussion.
Table 3.4-1 Preliminary target glass properties and operational ranges

<table>
<thead>
<tr>
<th>Glass property</th>
<th>Range</th>
<th>Processing concern</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting temperature, $T_m$</td>
<td>1250–1350°C</td>
<td>Refractory corrosion, volatility, melt rate</td>
</tr>
<tr>
<td>Viscosity</td>
<td>20–100 poise</td>
<td>Pourability, corrosion, melt rate</td>
</tr>
<tr>
<td>Liquidus temperature, $T_L$</td>
<td>$T_m - T_L &gt; 200^\circ$</td>
<td>Crystallization in melter</td>
</tr>
<tr>
<td>Redox</td>
<td>0.1$&lt;$[Fe$^{2+}$/ΣFe]$&lt;$0.4</td>
<td>Foaming, metals (Pb), molybdenum wear</td>
</tr>
</tbody>
</table>

Initial glass melts will be formulated to have target melting temperatures in the 1250–1350°C range, with melt viscosity in the 20–100 poise range. This temperature range is expected to provide higher melting rates and flexibility to formulate more durable, high waste loading compositions. Above 100 poise viscosity, the glass melting and refining (gas bubble removal) rates and glass pourability are adversely reduced. At melt viscosities below 20 poise, the glass is excessively corrosive to the melter refractories. At higher temperatures the redox equilibria become more reducing (higher Fe$^{2+}$/ΣFe ratios) increasing both melt gas release and the risk of metals (Pb) precipitation.

Liquidus temperature ($T_L$) is the highest temperature at which the glass composition will devitrify or form crystalline phases. Crystalline phases likely to be a concern for the candidate SLS and SLLS waste glass compositions include wollastonite, pseudo-wollastonite, and spinels. Accumulation of crystalline sludges in the melter will limit melter performance and melter life. To ensure that crystalline sludges do not accumulate in the melter, glass composition and temperature will be controlled so that the melting temperature ($T_m$) is at least 200°C greater than the glass liquidus temperature ($T_L$) to allow for potential temperature gradients in the melter. The $T_m - T_L > 200^\circ$C requirement is greater than the $T_m - T_L > 100^\circ$C requirement that is used for the Defense Waste Processing Facility (DWPF) because greater temperature gradients are likely to occur in the EnviTco melter design which uses rod rather than large plate electrodes and runs the refractories cooler relative to the peak melting temperatures in the center of the melter. However, the molybdenum electrode melter being used for the POPT has the capability to process higher liquidus temperature glass formulations since an inconel™ plate electrode melter, such as DWPF, has a melting temperature limit of ~1150°C. Since waste loading may be limited by liquidus temperature constraints, the ability to process at higher temperatures provides increased flexibility for achieving higher waste loadings.

Redox ratio is the ratio of the concentration of Fe$^{2+}$ to the total amount of iron ($\Sigma$Fe) present in the glass (Fe$^{2+}$/ΣFe). The redox ratio is used as an indicator of the oxidation state of all of the other transition metals present in the glass. Molybdenum electrode Joule-heated melters such as the EV-101 typically
operate under slightly reducing conditions such that a portion of the Fe present in
the glass is reduced from the Fe$^{3+}$ to the Fe$^{2+}$ oxidation state. The redox of the
glass will be targeted to be in the 0.1<$[Fe^{2+}/ΣFe]$<0.4 range. If the glass is too
oxidizing ($Fe^{2+}/ΣFe <$ 0.1), melt foaming may occur as a result of redox reactions
that liberate gases such as SO$_2$ and O$_2$. Foamed glass has poor heat transfer
characteristics and can cause a cooler viscous crust to form over the glass melt
pool. Such crusts are difficult to melt and tend to increase and rise as additional
gas is generated from below. An oxidized glass is also more corrosive to the
molybdenum electrodes. A glass that is too reducing ($Fe^{2+}/ΣFe > 0.4$) may
precipitate metals or metal sulfides that if allowed to accumulate in the melter
could cause electrical power shorting. Another issue with overly reducing melt
conditions is that precipitated metals such as Ni can cause eutectic melting of
electrodes and other molybdenum components in the melter.

A particular concern for the waste surrogate compositions being used for the
POPT is reduction of PbO and the precipitation of metallic Pb in the melter. The
presence of ~0.9 wt.% kerosene in the demonstration surrogate waste may
require the addition of oxidant additives to the feed to prevent the glass from
becoming overly reducing. Initially, NaNO$_3$ will be used as an oxidant additive if
an oxidant additive is required. MnO$_2$ may also be used as an oxidant additive if
the required NaNO$_3$ additions result in excessive NO$_x$ emissions. Sugar and/or
carbon will be used as reductant additives if reductant additives are required.

Approximate glass solubility limits for several troublesome components that can
limit the waste loading in SLS-based glasses are given in Table 3.4-2. Oxide
components such as Cr$_2$O$_3$, Fe$_2$O$_3$, FeO, and MnO can crystallize and precipitate
forming heavy sludge accumulations in the melter if their solubility limits are
exceeded. TiO$_2$ forms very fine crystals that can form nucleation sites for
precipitation of other crystalline phases. CuO and NiO may be reduced causing
metal precipitation in the melter. Depending upon the cooling rate, fine
phosphate crystals may precipitate in the glass on cooling as the $P_2O_5$ content
increases beyond ~2%, resulting in a milky or muddy appearance for the glass.

Other waste components such as Cl, F and SO$_4$ may separate if their glass
solubilities are exceeded forming a low viscosity molten salt layer on top of the
glass melt pool, which is very corrosive to the refractories at the melt line. A salt
tap is provided on the POPT melter for removal of molten salts should they
accumulate on top of the glass. Sulfate is the single component in the POPT
surrogate compositions considered most likely to phase separate as an molten
salt layer if it exceeds the glass solubility limit and is not decomposed and
volatilized. A portion of the phosphate could also potentially segregate to a
molten salt layer as alkali/alkaline phosphates.
Table 3.4-2. Approximate Glass Solubility Limits

<table>
<thead>
<tr>
<th>Component</th>
<th>Solubility limit (wt.%)</th>
<th>Processing concern</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr₂O₃</td>
<td>&lt;0.5</td>
<td>Cr₂O₃ precipitation</td>
</tr>
<tr>
<td>CuO</td>
<td>&lt;1.0</td>
<td>Cu metal precipitation</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>&lt;20% (Fe₂O₃ + FeO + MnO)</td>
<td>Spinel precipitation</td>
</tr>
<tr>
<td>FeO</td>
<td>&lt;20% (Fe₂O₃ + FeO + MnO)</td>
<td>Spinel precipitation</td>
</tr>
<tr>
<td>MnO</td>
<td>&lt;20% (Fe₂O₃ + FeO + MnO)</td>
<td>Spinel precipitation</td>
</tr>
<tr>
<td>NiO</td>
<td>&lt;1.0%</td>
<td>Ni metal, eutectic melting of Mo</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>&lt;2–10%</td>
<td>Glass phase separation, devitrification</td>
</tr>
<tr>
<td>TiO₂</td>
<td>&lt;1.0%</td>
<td>Promotes glass devitrification</td>
</tr>
<tr>
<td>Cl</td>
<td>&lt;0.6%</td>
<td>Molten salt layer separation</td>
</tr>
<tr>
<td>SO₃</td>
<td>&lt;1%</td>
<td>Molten salt layer separation</td>
</tr>
<tr>
<td>F</td>
<td>&lt;2%</td>
<td>Molten salt layer separation</td>
</tr>
</tbody>
</table>

Materials compatibility issues will be evaluated as part of the POPT development work. It is known that a low melting temperature Mo-rich eutectic exists in the Mo-Ni binary system that can cause damage to electrodes and other molybdenum parts in the melter if the Fe⁺²/ΣFe ratio exceeds 0.5 causing NiO in the waste composition to be reduced to Ni metal. Prior mixed waste processing experience with the TVS and pilot-scale testing has shown that very little wear of internal molybdenum components occurs when melt redox is controlled within the target 0.1<Fe⁺²/ΣFe<0.4 range. Ni is not expected to be a problem for the POPT demonstration at the low concentrations in which it is present.

Although there are no Mo-rich low melting eutectics in the Mo-Pb binary system, the glass melting temperatures are much greater than the 327°C melting temperature of pure Pb and the potential for dissolution or attack of Mo parts in contact with significant accumulations of molten Pb will be assessed.

3.5 Crucible Melting Activities

The glass formulation development strategy is outlined as a logic flow diagram in Figure 3.2-1. An overall goal of the glass formulations will be to maximize waste loadings in processable formulations that meet the treated product requirements specified in the contract SOW. These product requirements include:
- Uniform, homogeneous appearance,
- Compressive strength of > 50 psi,
- Contain no free liquids,
- Leachability less than 50% of the TCLP limits (S0-D, S1-T and S2-T glasses),
- Leachability less than the UTS limits (S0-U, S1-U and S2-U glasses),
- No fine particulate material (<1 wt.% of <10 microns diameter and <15 wt.% of <200 microns diameter), and
- Not exhibit any hazardous characteristics.

In order to meet the timeline of the overall POPT project, development of the S0-D formulation for use in the POPT melter demonstration will be given first priority. The S1-T and S2-T recipes will be developed in parallel with the S0-D recipe. Development of the S0-U, S1-U and S2-U recipes to meet the more stringent UTS durability requirements will occur at a later stage and take advantage of the experience gained in developing the glasses to meet the 50% of TCLP release limits.

### 3.5.1 TCLP Recipe Development (S0-D, S1-T, S2-T)

The first series of crucible melts (Box Nos. 4, 5, and 11 in Figure 3.2-1) will focus on glass formation, melt viscosity, the need for redox control and durability verification measurements. The crucible melts will be performed in uncovered, fused silica crucibles. This approach yields high production from the laboratory and large amounts of data in a short time. Other crucible types that may be used if problems arise using silica crucibles are alumina or high-temperature clay.

Volatility losses and some dissolution of crucible material into the glass may occur shifting the glass composition from the target batch composition. To correct for such composition shifts, properties measured for the glasses will be correlated to the analyzed composition rather than the batch composition when appropriate. Up to fifteen (15) of the first series melts will be analyzed to evaluate the shift in volatile species and silica concentration that occurs during the crucible melting process. The fusion factors determined in these tests will be applied during subsequent melts to better predict the optimum recipes for the remaining glasses. This should improve the ability to produce laboratory glasses that match the glass produced during the POPT demonstration run and the proposed full-scale system.
Preliminary plans are to vary alkali/alkaline to surrogate ratios in the first melt series (3 to 5 levels) using each of the three surrogate waste recipes. These glasses will initially be melted at a single temperature (tentatively 1300°C) but temperature may also be used as a variable to maximize the waste loading. The resulting glass melts will be cast into disks for visual examination and photographic recording. A visual, experience-based judgement by Envitco engineers as to whether the glass viscosity is in the general usable range will be made and recorded in a log book for each melt. Other visual observations such as homogeneity will also be recorded in a logbook for inclusion with the Final Report.

Melts exhibiting excessive foaming (Box No. 11 in Figure 3.2-1) may be reformulated and additional melts performed. Samples of one or more of the glasses that appear to have melt viscosity in the proper range will be analyzed in accordance with Section 7 of the Work Plan (Box No. 5 in Figure 3.2-1). If the initial observations of the melts appear promising, samples may also be analyzed for liquidus temperature. Any glasses that fail TCLP will be rejected as possible recipes (Box No. 12 in Figure 3.2-1). Should all of the first series melts not meet the TCLP requirements, additional melts will be performed at lower waste loadings and/or include durability enhancing additives such as alumina. Multiple melts of a single composition may be required to provide sufficient sample quantities.

The glasses that pass the durability and processability requirements will be used in a second series of melts (Box No. 6 in Figure 3.2-1). Preliminary plans for a second series of melts would focus on variations in redox additive additions. These tests may also provide verification of PbO reduction redox threshold and additional data on potential melt foaming issues. Additional crucible melt series will be planned to further optimize the formulations or to address specific processability related issues that may be indicated based on the initial crucible melt tests.

The glass formulation that has the highest waste loading and still meets the durability and processability requirements will be chosen as the treatment recipe for the S0-D, S1-T and S2-T surrogates (Box No. 7 in Figure 3.2-1). The POPT demonstration will use the S0-D recipe (Box No. 8 in Figure 3.2-1).

3.5.2 UTS Recipe Development (S0-U, S1-U, S2-U)

After the TCLP recipe glasses are formulated, the UTS glass development will proceed. Additional melts will be performed based on the SLS and SLLS glass systems with possible additions of Al₂O₃ to increase durability (Box No. 13 in Figure 3.2-1). Lower waste loading than the TCLP recipes will also be
investigated. The S0-U, S1-U and S2-U recipes will be chosen using the same processability criteria as the TCLP recipe development and the stricter UTS durability requirements (Box No. 14 in Figure 3.2-1).

3.5.3 Glass Samples for FDF Testing

After the treatment recipes have been selected, each glass will be manufactured in multiple melts to provide sufficient amounts of glass to meet the requirements of Table G2 in the Contract. Samples of the six final glass formulations will be supplied to FDF for testing and archival storage as specified in Table G2 of the Contract (Box No. 15 in Figure 3.2-1). Section 7.0 - Sampling, Data Collection and Analysis Plan describes the sampling in more detail.

3.6 Development of the Surrogate Melter Feed Slurry

The selection of specific glass-forming additives for addition to the surrogate waste allows some control of the rheological properties of the final melter feed slurry. This flexibility will be used to develop melter feed having maximum solids content and having acceptable rheological properties. The rheological properties, including viscosity and sheer characteristics, will be evaluated through visual observation of mixing tests in the laboratory.
4.0 TESTING AND DATA RATIONALE

4.1 Rationale for Sampling Points and Sampling Frequency

The rationale for the selection of sampling points and the frequency of sampling is based on experience gained in previous melter demonstration programs (Wilson, 1996). The sampling points and frequency are established to provide data to support the mass and energy balance, which in turn provides the basis for the full-scale process design. The sampling strategy also fulfills the contract requirements for process sampling of the surrogate and glass product. The sampling plan is designed to fulfill the Data Requirements as defined in Section 7.1 of this Work Plan.

Special considerations have been applied to the sampling plan to address the fact that the process is a combination of continuous unit operations and batch unit operations. This is presented below in Table 4.1-1 Process Staging and Sequence.

<table>
<thead>
<tr>
<th>Unit Operation</th>
<th>Process Type</th>
<th>Transfer To</th>
<th>Period/Inventory</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surrogate Blend</td>
<td>Batch</td>
<td>Feed Blending</td>
<td>~2000 kg/batch</td>
<td>At least 5 batches</td>
</tr>
<tr>
<td>Feed Blending</td>
<td>Batch</td>
<td>Melter</td>
<td>~2500 kg/tank</td>
<td>Combined surrogate and feed batches</td>
</tr>
<tr>
<td>Melter</td>
<td>~CSTR</td>
<td>Pouring</td>
<td>1800 kg/melter</td>
<td></td>
</tr>
<tr>
<td>Pouring</td>
<td>Continuous</td>
<td>Container</td>
<td>~250 kg/container</td>
<td></td>
</tr>
</tbody>
</table>

The sequence of the batch process surrogate preparation, batch process feed blending, and continuous process melter operation results in the loss of the identity of the individual surrogate batches. This voids any possibility of tracking the demonstration based on the surrogate batch identification.

In an effort to conform to the requirements of the contract, which dictates that 10 samples must be taken to represent the original SOW's 10 batches processed, Envitco will conduct sampling on a periodic basis once the POPT demonstration has started its 72-hour duration. To achieve the minimum 10 samples, a sampling period of 7.2 hours would be required. Envitco will conduct process sampling on approximately a 3-hour interval to ensure that:
a) sufficient samples are available to represent the process,
b) additional samples are available for analysis in the event of sample loss or analytical difficulties, and
c) additional samples are available for analysis in the event of interferences or anomalies that may make the data or sample unacceptable.

Sampling on a time basis will be acceptable for the POPT demonstration as the entire 72-hour demonstration will be conducted under steady-state conditions. Steady-state is defined as a chemical steady-state where the feed and glass are of similar composition and that feed is continuously being input to the melter. The process optimization period will be used to process at least 3 volumes of glass prior to starting the POPT demonstration to bring the system to chemical steady-state conditions. Therefore, when feed is being input to the melter, the system is at steady-state and any samples taken during the POPT will be indicative of steady-state conditions.

The Surrogate Preparation and Melter Feed Preparation processes are described in further detail in Section 5.1.1 of this Work Plan.

The samples selected for analysis will be identified per Section 7.0 of this Work Plan. The samples analyzed will represent the POPT demonstration period. This will provide complete and accurate data for assessing the process performance and confirming the steady-state operating conditions of the process.

4.2 Identification of and Rationale for Analytical Methods

The primary goal of the analytical work is to provide sufficient, quality data to perform the proper evaluations required to meet the data requirements. Table 4.2-1 lists the analyses that will be performed and the analytical method for that analysis. Since the rationale for choosing the list of analyses is to meet the data requirements, the requirements that are met by each analysis are also shown. The data requirements will be addressed in Section 7.1 of this Work Plan.
### Table 4.2-1 Analytical Methods

<table>
<thead>
<tr>
<th>Line No.</th>
<th>Analysis</th>
<th>Analytical Method</th>
<th>Data Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Toxicity - TCLP</td>
<td>EPA 1311 and 6010/7000s</td>
<td>Product Performance</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2nd Waste Characterization</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Verification of Surrogate</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Develop Treatment Recipe</td>
</tr>
<tr>
<td>2</td>
<td>Corrosivity</td>
<td>EPA 9045B</td>
<td>2nd Waste Characterization</td>
</tr>
<tr>
<td>3</td>
<td>Reactivity</td>
<td>EPA 9010/9030</td>
<td>2nd Waste Characterization</td>
</tr>
<tr>
<td>4</td>
<td>Ignitability</td>
<td>EPA 1010</td>
<td>2nd Waste Characterization</td>
</tr>
<tr>
<td>5</td>
<td>Compressive Strength</td>
<td>ASTM C 773</td>
<td>Product Performance</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Develop Treatment Recipe</td>
</tr>
<tr>
<td>6</td>
<td>Loss on Drying (LOD)</td>
<td>ASTM D 2216 and CELS # 424002-002</td>
<td>Waste Loading</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Mass Balance</td>
</tr>
<tr>
<td>7</td>
<td>Loss on Ignition (LOI)</td>
<td>CELS # 424003-000</td>
<td>Waste Loading</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Mass Balance</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Full-scale Design</td>
</tr>
<tr>
<td>8</td>
<td>Quantitative Chemical Analysis</td>
<td>Various CELS Procedures Depending on Chemistry</td>
<td>Mass Balance</td>
</tr>
<tr>
<td></td>
<td>(QCA)</td>
<td></td>
<td>Develop Treatment Recipe</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2nd Waste Characterization</td>
</tr>
<tr>
<td>9</td>
<td>Glass Density</td>
<td>ASTM C 693</td>
<td>Bulking Factor</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Mass Balance</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Full-scale Design</td>
</tr>
<tr>
<td>10</td>
<td>Slurry Density (70 wt. % moisture)</td>
<td>CETL SOP # 002</td>
<td>Full-scale Design</td>
</tr>
<tr>
<td>11</td>
<td>Surrogate Density (30 wt. % moisture)</td>
<td>EM-1110-2-1906 App. II (Adapted)</td>
<td>Verification of Surrogate</td>
</tr>
<tr>
<td>12</td>
<td>Particle Size Determination (sieve analysis)</td>
<td>ASTM D 422</td>
<td>Product Performance</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Verification of Surrogate</td>
</tr>
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<td>13</td>
<td>Total Suspended Solids (TSS)</td>
<td>CWA 160.2</td>
<td>2nd Waste Characterization</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(Wastewater)</td>
</tr>
<tr>
<td>Line No.</td>
<td>Analysis</td>
<td>Analytical Method</td>
<td>Data Requirement</td>
</tr>
<tr>
<td>---------</td>
<td>--------------------------------------------</td>
<td>-----------------------------</td>
<td>-----------------------------------</td>
</tr>
<tr>
<td>14</td>
<td>Total Dissolved Solids (TDS)</td>
<td>CWA 160.1</td>
<td>2nd Waste Characterization (Wastewater)</td>
</tr>
<tr>
<td>15</td>
<td>Glass Resistivity</td>
<td>ASTM C 965</td>
<td>Develop Treatment Recipe</td>
</tr>
<tr>
<td>16</td>
<td>Glass Viscosity vs. Temperature</td>
<td>ASTM C 965</td>
<td>Develop Treatment Recipe</td>
</tr>
<tr>
<td>17</td>
<td>Glass Oxidation State</td>
<td>CELS # 425007-000, CELS # 412006-000</td>
<td>Full-scale Design</td>
</tr>
<tr>
<td>18</td>
<td>Glass Annealing Point</td>
<td>ASTM C 336</td>
<td>Full-scale Design</td>
</tr>
<tr>
<td>19</td>
<td>Glass Liquidous Point</td>
<td>ASTM C 829</td>
<td>Develop Treatment Recipe</td>
</tr>
<tr>
<td>20</td>
<td>Offgas Reference Data</td>
<td>EPA Methods 1, 2, 3, 4</td>
<td>Mass Balance</td>
</tr>
<tr>
<td>21</td>
<td>Offgas Particulate Loading</td>
<td>EPA Method 5</td>
<td>Mass Balance</td>
</tr>
<tr>
<td>22</td>
<td>Offgas Sulfur Dioxide</td>
<td>EPA Method 6C</td>
<td>Mass Balance</td>
</tr>
<tr>
<td>23</td>
<td>Offgas Nitrous Oxides</td>
<td>EPA Method 7E</td>
<td>Mass Balance</td>
</tr>
<tr>
<td>24</td>
<td>Offgas Volatile Metals</td>
<td>EPA Method 29</td>
<td>Mass Balance</td>
</tr>
<tr>
<td>25</td>
<td>Crystalline Content and ID by X-ray Diffraction (XRD)</td>
<td>Lab Procedures</td>
<td>2nd Waste Characterization</td>
</tr>
<tr>
<td>26</td>
<td>Phase Identification by Scanning Electron Microscopy (SEM)</td>
<td>Lab Procedures</td>
<td>2nd Waste Characterization</td>
</tr>
</tbody>
</table>
The following list presents the analytes for the quantitative chemical analysis (QCA).

As  Al  Ba  Ca  Cr  Fe
K   Li  Mg  Mo  Na  Ni
P   Pb  S  Se  Si  V  Zn

This list may be modified as a result of the recipe development if the chosen glass additives contain any new elements that are not shown on the list.
5.0 PROCESS DESIGN AND TESTING PROCEDURES

5.1 Discussion of Design/Configuration

The POPT demonstration will be conducted using a fully integrated vitrification test unit at CETL. The system consists of the melter feed preparation, melter, auxiliary services (power, water, air, etc.), offgas treatment, secondary waste handling, and glass handling. The core treatment technology is the melter which will vitrify the waste into a durable waste form.

The melter to be used for the POPT is EnviTco's WASTE-VIT® EV-101. The melter has a design throughput of two (2) tons per day (TPD) of glass based on a dry soda-lime-silica glass batch. The melter is capable of operating between 1000°C and 1500°C. The melter is designed with three (3) processing areas, each having an independent drain orifice. The main melt chamber is used to melt the waste plus glass additives and to homogenize the glass. It is heated by the use of molybdenum electrodes and a propane combustion system in the plenum. Volatility is limited by the use of a cold-cap (layer of unmelted feed) formed on top of the glass pool. The other two chambers are for the individually controlled draining of salts and glass.

5.1.1 Description of Operations and Equipment

5.1.1.1 Surrogate Preparation

The preparation of surrogate will occur at CETL in Anderson, SC. The surrogate preparation schedule will be provided to FDF prior to initiation of surrogate manufacturing.

Manufacturing the surrogate slurry will occur at least 24 hours prior to the initiation of the 72-hour test. The system consists of a mixing tank and a feed tank. Mixers and recirculation pumps are integrated into the system for mixing and transferring abrasive slurries.

Each batch of surrogate (70 wt.% water) will be made according to EnviTco-prepared batch sheets which will serve as a recipe log for surrogate manufacturing. These will be included in the Final Report. The surrogate will be manufactured according to the following procedure as defined in Contract Modification No. 01:

1. In accordance with the surrogate recipe defined in Contract Modification No. 01 and the batch sheet, add the desired amount of water into the mix tank.

2. Turn on the mix tank.
3. Add the bentonite while stirring/agitating.

4. Blend at high speed until the contents are well mixed.

5. Maintain the stirring for 24 hours to allow the bentonite to fully hydrate.

6. In a separate container, add the organics to the fine silica.

7. Allow the silica to absorb the organics.

8. Weigh the dry chemicals and add to the bentonite/water mixture with sufficient agitation to keep all chemicals suspended.

9. Add the organics/silica mixture and continue blending for 24 hours.

Note: The slurry may thicken over time.

The slurry will then be transferred and stored in holding tanks. The surrogate slurry will be re-suspended and homogenized prior to transfer into the mix tank for addition of the glass forming additives.

5.1.1.2 Melter Feed Preparation

The melter feed system will be inspected and tested during the Pre-POPT process optimization period as described in Section 5.1.1.3.

The melter feed preparation will occur simultaneously with the POPT melter demonstration operation. Each batch of surrogate slurry (70 wt.% water) will be transferred back into the mix tank where the glass-forming additives will be combined with the surrogate slurry. Each mixed batch of melter feed will then be transferred to the feed tank. Upon transfer of the melter feed to the feed tank, a new batch of surrogate slurry will be transferred into the mix tank and the melter feed preparation process will start again. Each transfer of surrogate slurry will be recorded in the test log.

When the feed tank is more than half empty, a new melter feed batch will be transferred to the feed tank. This will continue until the conclusion of the POPT Demonstration. Each transfer of melter feed to the feed tank will be recorded in the test log.

Based on successful implementation of slurry blending and feeding the Transportable Vitrification System, the melter feed slurry will be recirculated continuously from the feed tank to the vicinity of the melter and back into the feed tank. A small slip-stream of melter feed will be metered from the recirculation line. A metering pump will be used to control the feed rate to the melter. The
feed rate will be determined by pump calibration and/or direct monitoring of the slip-stream flow rate to the metering pump.

Buildups in the system will be monitored through the use of pressure sensors, flow meters, and monitoring of the pump speed. If a buildup occurs in the line, the back pressure and pump speed will increase to maintain the same flow rate. These monitoring points will be logged through the data acquisition system or on the log sheets used by the operators and technicians. Buildups, clogs and settling will be investigated and any problems will be corrected during the pre-POPT process optimization period described in Section 5.1.1.3.

5.1.1.3 Melter Operations

The POPT Demonstration will be performed using a Joule-heated, ceramic-lined, cold-top, slurry-fed melter. The melter will run for a total of 72-hours for the POPT demonstration. A period of process optimization will occur prior to initiation of the POPT demonstration run. This will include feed, melter, and offgas treatment systems checkout, training for technicians and melter operation to achieve steady-state operating conditions. Envitco engineers will operate the system with the assistance of CETL technicians. For the 72-hour POPT demonstration, Envitco engineers will provide direct control and oversight of the melter operations 24 hours per day for the entire 72 hours (3 days).

5.1.1.3.1 Pre-POPT Process Optimization

During the days leading up to the 72-hour POPT test, a period of process optimization with demonstration surrogate will occur. The four (4) primary objectives of this period are to 1) inspect melter and peripheral systems 2) start and check the melter and peripheral systems, 3) establish the target operating parameters, and 4) process at least three (3) melter volumes of surrogate glass in order to reach a chemical steady-state.

The melter will be started and all systems will be checked for proper operation. Systems such as the feed, offgas, and melter services will be operated at various points within their operational ranges to verify minimum and maximum capabilities.

The starting target operating parameters for the process optimization period are based on the processability requirements and previous vitrification tests with the EV-101 melter. These targets are subject to change depending on the final glass recipe that is developed prior to the initiation of the demonstration. Table 5.1-1 shows the key parameters that will be monitored and adjusted accordingly.
Table 5.1-1 Initial Optimization Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Initial Target</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass Temperature</td>
<td>1250 – 1350°C</td>
</tr>
<tr>
<td>Plenum Pressure</td>
<td>-0.1&quot;</td>
</tr>
<tr>
<td>Cold-top Coverage</td>
<td>&gt;75%</td>
</tr>
<tr>
<td>Feed Rate</td>
<td>~1.3 l/min</td>
</tr>
<tr>
<td>Glass Pour Rate</td>
<td>~44 kg/hr</td>
</tr>
<tr>
<td>Plenum Temperature</td>
<td>~350 – 550°C</td>
</tr>
<tr>
<td>Glass Viscosity</td>
<td>20 – 100 Poise</td>
</tr>
<tr>
<td>Redox Ratio (Fe²⁺/Fe_total)</td>
<td>0.1 – 0.4</td>
</tr>
</tbody>
</table>

Steady-state conditions for the melter will be defined as reaching a state where the chemistry of the feed is similar in oxide composition as the glass that is exiting the melter. The melter roughly responds like a continuous-stirred tank reactor. Therefore, in order to reach chemical equilibrium, three (3) melter volumes will be processed prior to the initiation of the 72-hour POPT demonstration. Once a chemical equilibrium has been reached and process parameters have been refined and confirmed, the demonstration will begin. From the start of the POPT demonstration forward, steady-state operation will be defined as having continuous feed input to the melter.

The information and experience gained during the process optimization period will be used by Envitco to help develop the preliminary full-scale design data.

5.1.1.3.2 Main Melt Chamber

Surrogate is introduced and melted into glass in the main melt chamber. The slurry is fed through the roof of the melter and distributed to allow coverage of a significant portion of the melt surface. The main glass in the main melt chamber is heated through the use of in-glass electrodes. The plenum above the glass is heated by a combustion heater located in the roof and heat losses from the glass. The propane combustion system is used to start the melter and provide sufficient plenum heat to control the offgas exit temperature. The plenum pressure is maintained under vacuum by the offgas treatment equipment to prevent the release of gases into the work-area surrounding the melter.

Once operating, the glass is maintained in a molten state through the use of joule-heating. Joule-heating is the result of passing current through the glass.
which has a certain resistance. The temperature of the melter is monitored through the use of in-glass and plenum thermocouples. Operators can adjust the power input to the melter in order to maintain the glass temperature.

The POPT demonstration will employ the cold-top approach in order to minimize power consumption (heat input) and reduce volatility. Under normal cold-top operation, feed will cover nearly the entire surface of the molten glass. The slurry will build up a layer of unmelted feed, commonly referred to as a cold-cap, that will help reduce heat losses and volatility. The unmelted feed layer acts as an insulating blanket over the glass. The cold-cap reduces volatility by condensing the volatile species and returning them to the glass. The overall result is a very high retention of volatile elements, such as lead, in the final waste form.

5.1.1.3.3 Metals Drain

The Silo 1 and 2 residues contain high levels of lead which, under certain conditions, could be reduced to metallic sludge phases that will separate from the glass. However, should metallic species separate from the glass, the main melt chamber has a sloped bottom for the collection of reduced metal species. The bottom of the main melt chamber has a dedicated drain to purge any such collected material. Independent draining of possible reduced metal species increases the melter life by reducing downward drilling of metals into the refractory and mitigates the risk of short circuiting due to the accumulation of metals.

One goal of this POPT is to minimize secondary waste which means minimizing production of any metallic sludges. The precipitation of metallic species will be minimized by adjusting the glass chemistry and redox state of the glass. However, should they form during the POPT demonstration, the metals drain will be opened between the 48th and 72nd hour of the demonstration. The metals drain material will be sampled and analyzed according to Section 7.0 of this Work Plan.

5.1.1.3.4 Glass Drain

The main melt chamber is connected to a glass drain bay. The drain bay is used to condition the glass to the proper temperature for pouring the glass into disposal containers. Thermocouples in the drain bay glass and plenum allow the operators to monitor and control the power input, and hence the temperature, independent of the main melt chamber. Drain control is improved because the glass drain bay orifice does not have as much hydraulic head above it and the pouring temperature can be more precisely controlled.
The glass is drained through an orifice at the bottom of the drain bay. The glass flow can be started, stopped and controlled by adjusting the heating and cooling of the drain orifice. As the glass is heated, it will become less viscous and flow easily through the orifice. As the glass is cooled, it will become increasingly more viscous and eventually stop flowing through the orifice. Operators will monitor thermocouples in the drain orifice mechanism and the drain rate (see Section 5.1.1.3.8) in order to adjust the heat applied/removed from the orifice.

5.1.1.3.5 Salts Drain

When the solubility of the salts is exceeded in the glass, they will separate from the glass and float on top of the glass surface. The batch layer on top of the glass may also become supersaturated with the salt species if the amount of salt being produced is not volatilized quickly enough. When the solubility of the glass and the saturation of the batch layer have been exceeded, the salt will then migrate to the lowest point, or seek its own level. The excess salt is then "skimmed" from the melt surface while holding back the batch layer to prevent batch from entering the salt drain chamber. The molten salt is then drained through an orifice as described below.

The main melter chamber is connected to a salt drain bay. The salt drain bay allows for the collection and draining of molten salts during steady-state operations. Once the salt is collected, it is drained through an orifice into steel collection containers. Removing excess salt from the main melter chamber will reduce refractory wear, as molten salt is extremely corrosive to refractory. Removing the salt as a separate phase will also help reduce energy consumption in the main melt tank because energy is not being used to volatilize all of the salt into the offgas.

The primary salt-forming concern with the Silo waste is sulfur. The PbSO₄ and BaSO₄ in the surrogate will decompose in the melter resulting in PbO, BaO, and SO₃ and/or SO₄. The PbO and BaO will participate in the glass structure along with ~1 to 2% of SO₃. However, if the sulfur solubility limit is exceeded, SO₄ will then preferentially combine with Ca or Na to form CaSO₄ and NaSO₄. The formation of CaSO₄ was confirmed in the Vitrification Pilot Plant operations conducted by FDF (Fu, et al, 1996).

A goal of the POPT demonstration is to minimize secondary waste. In order to minimize the production of salt species, salt solubility will be investigated in the recipe development phase. The salt drain will be used to remove the salt as a separate phase should it form on the surface of the glass.
5.1.1.3.6  Auxiliary Melter Services

The melter system consists not only of the melter but includes the power supply, services (utilities) pallet and controls. These systems provide monitoring and control of the electrical energy input, cooling water, and protective gas.

The power supplies are capable of automatically maintaining a constant power input to the melter. The operator can adjust the power input to effect changes in the glass temperature. Key parameters of the power supply are displayed on the control panels and are recorded in the data acquisition system.

The services pallet provides cooling water and nitrogen protective gas to the melter. The cooling water is primarily used in the water-cooled shell surrounding the melter. The shell helps reduce refractory wear by lowering the operating temperature of the refractory and helps to mitigate glass leaks by freezing the glass before it can reach the exterior of the melter. Nitrogen is used to protect certain molybdenum parts from oxidation. Any molybdenum parts that are exposed to air above a few hundred degrees Celsius are purged with nitrogen. The cooling water and nitrogen are monitored for flow, temperature, and pressure as required. High and low level alarms are integrated into the control system to warn an operator of unacceptable conditions.

5.1.1.3.7  Product Handling

The glass wasteform will be cast directly into 30 gallon drums during the POPT demonstration. The 30 gallon drums will be held inside a 55 gallon drum with sand filling the space between the two drums. This arrangement was used for previous tests and allows the integrity of the 30 gallon drum to be maintained while at temperature. After the glass has cooled, the 30 gallon drum is covered with a lid and removed from the 55 gallon drum by the use of a forklift and drum hooks. A production-scale glass handling system would be highly automated and the glass cast directly into containers matched to the full-scale transport and disposal requirements.

The glass handling equipment is arranged with roller-conveyors to index the drums under the drain. Drums will typically remain on the conveyor to cool for a period of a few hours prior to moving into an area for additional cooling and removal of the 30 gallon drum. The conveyor directly under the glass drain is equipped with load cells to monitor the weight of the glass container.

Salts that are removed through the salt drain are cast into steel containers. The salt containers are then removed by hand using personal protective equipment suitable for the hot container. The containers are covered and allowed to cool. The salt drain rate is then calculated by weighing the containers over a time.
basis and by collecting samples over a period of time and returning the sample to the collection container.

Metallic sludges that are drained from the bottom of the melter will be collected in the same manner as the glass. The purpose of the metallic sludge sample collection is to determine if any metallic precipitation occurred in the melter, to characterize the precipitates for determination of secondary waste generation, and to provide material balance input related to metallic precipitation accumulation in the melter. The metals drain will only be opened long enough to either observe that only glass is pouring through the orifice or the glass level in the melter is unacceptably low. The collection of the metals drain material will either be in a drum similar to the glass collection container or quenched for easy collection of samples. The exact method will be determined prior to the POPT demonstration.

5.1.1.3.8 Offgas Treatment

Offgas generated during the vitrification of the melter feed is treated by the offgas treatment system. A fan draws the melter exhaust gases through the offgas treatment train and maintains a constant negative pressure within the melter. The exhaust gases are conditioned by a film cooler and an electrostatic precipitator prior to release to the atmosphere.

Sample points are provided at the entrance and exit of the offgas treatment system for the performance of EPA standard methods (Method 5, Method 29, etc.). Pressure monitors, pitot tubes and thermocouples mounted throughout the system provide flow characterization data. When coupled with the data collected by the EPA standard methods, the physical and chemical characteristics of the offgas stream may be integrated into the material and energy balance.

5.1.1.3.9 System Throughput Considerations

Operating at lower than design production rates has been proven with other Envitco melters. The design throughput of the EV-101 melter is 2 TPD of glass from a dry soda-lime-silica (SLS) feed (similar to bottle glass). The production rate of the POPT demonstration is currently targeted at 1 TPD of glass from slurry feed. The difference in glass production is due to the water content of the slurry feed. Adjusting the glass drain rate to 1 TPD is merely a function of adjusting the temperature of the drain orifice as noted previously. Should the melt rate be lower than anticipated, the glass drain rate can be reduced to reflect the lower melt rate.
5.1.2 Pre-Treatment Requirements

Glass-making additives will be incorporated into the surrogate slurry to produce melter feed slurry. This slurry will then be fed to the melter in order to produce glass. Other than the incorporation of glass-making additives, the surrogate waste slurry will not undergo any treatment prior to being vitrified.

5.1.3 Testing Methodology

The Testing Methodology to be applied during the POPT will be based on structuring the testing program to provide the data necessary to carry out the required design studies on the full-scale treatment facility. This includes identification of the required sampling points, sampling intervals, process parameter data requirements, and process controls to ensure that the demonstration mimics the continuous, full-scale process.

As the test is conducted in two phases (bench-scale development, and the POPT melter demonstration), two methodologies are required to match the actual testing environment.

Bench-scale Testing: The bench-scale development program will require systematic glass formulation studies with analysis of fundamental glass characteristics and performance indices. The glass formulations to be tested will initially be based on existing performance data. The composition region will be expanded as necessary to address the specific composition and performance requirements as defined in the contract.

The testing program will be structured into a matrix which will provide a plan for controlled variation in the waste loading, glass-making additives, and processing conditions. The objective of the matrix is to optimize the glass performance while maintaining acceptable glass characteristics matched to the melter capabilities. Some flexibility exists at this stage, as the design basis for the melter will ultimately be matched with the glass characteristics, within a set of general bounds for Joule-heated melter design.

Analysis of initial glasses will provide insight to the more promising composition regions, which may result in refinement of the test matrix. As the composition region is refined, the focus will shift from glass characteristics to glass performance, though characteristics will still be assessed for each glass.

The glasses selected from the bench-scale development program will be those compositions that showed the best combination of characteristics and performance, while safely meeting the glass performance requirements as
specified in the contract. The glass that is chosen from the demonstration surrogate testing will then be utilized in the melter demonstration test.

See Section 3.0 of this Work Plan for a detailed description of the recipe development and processing issues.

**Melter Demonstration:** The period prior to the 72-hour witnessed demonstration will be used for process development and optimization. This will establish the process conditions and limits, parameter set-points and ranges, and other process controls that will be applied throughout the POPT testing program.

The process controls determined during the process development and optimization program will be applied throughout the melter demonstration to ensure that the data generated is based on accepted, predetermined operating conditions. This will help ensure that the demonstration data is correct and valid, and matches the intended process. The overall methodology for the test includes a combination of pre-process and in-process control functions, as well as data collection and post-process feedback to the extent possible.

### 5.1.4 Secondary Treatment Requirements

The glass wasteform will not require any secondary treatment. It is cast directly into the disposal container. A production-scale treatment system can be configured to cast directly into a container designed to meet the handling and disposal requirements of the full-scale system.

Based on previous melter demonstrations, possible secondary wastes that may require treatment prior to disposal include:

- Offgas solids,
- Wastewater,
- Salts,
- Metallic species (elemental metal, metal sulfides, etc.), and
- Used refractory and other melter parts.

A goal of the POPT is to minimize secondary waste by minimizing offgas residues, maximizing salt solubility in the glass, and preventing metallic sludge formation. Secondary waste treatment of POPT demonstration residues has not been defined at this time. The exact characteristics of the secondary waste will determine the treatment requirements for disposal. Should the secondary wastes be hazardous, treatment and disposal will be performed according to Clemson University's hazardous waste disposal system. The disposition of secondary wastes will be addressed in the Final Report.
5.2 Test Procedures

The POPT will be conducted in accordance with Standard Operating Procedures (SOPs), as reviewed and revised by Envitco for activities conducted during the recipe development and demonstration runs. Table 5.2-1 presents a list of existing CETL procedures that will be reviewed and/or revised for use on this project.

CETL SOPs cover the following areas:

Purpose – why the SOP was written,
Scope – what the SOP applies to,
Responsibilities – to whom the SOP applies (specifically and generally),
Procedure – step-wise instructions on how to perform the SOP,
Records – what records are required and where they are stored,
Safety – safety issues, precautions, or specific instructions,
References – any documents that support the SOP, and
Attachments – any forms or ancillary information specific to the SOP.

Copies of the CETL SOPs are not included with this Work Plan due to their review status. When the procedures are approved for use, copies of the procedures will be made available to FDF. The SOPs will be located in a convenient location for the operators to use during the demonstration and can be reviewed by the FDF witnessing engineer(s) at that time.
### Table 5.2-1 CETL SOPs

<table>
<thead>
<tr>
<th>CETL SOP No.</th>
<th>SOP Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>002</td>
<td>Density Measurements of Slurries</td>
</tr>
<tr>
<td>003</td>
<td>Density Measurements of Glass</td>
</tr>
<tr>
<td>007</td>
<td>Chain of Custody</td>
</tr>
<tr>
<td>008</td>
<td>Control of Standard Operating Procedure Documents</td>
</tr>
<tr>
<td>009</td>
<td>Sample Handling Procedure</td>
</tr>
<tr>
<td>010</td>
<td>Standardization of Balances and Scales</td>
</tr>
<tr>
<td>011</td>
<td>Outside Storage of Canisters and Drums</td>
</tr>
<tr>
<td>012</td>
<td>Determining the Moisture Content in Feed Materials</td>
</tr>
<tr>
<td>013</td>
<td>Determining Loss on Ignition</td>
</tr>
<tr>
<td>014</td>
<td>Glass Batch Preparation</td>
</tr>
<tr>
<td>015</td>
<td>Envitco Furnace Start-up Procedure</td>
</tr>
<tr>
<td>016</td>
<td>Envitco Furnace Hot Hold Procedure</td>
</tr>
<tr>
<td>017</td>
<td>Envitco Furnace Shutdown Procedure</td>
</tr>
<tr>
<td>019</td>
<td>Melt Chamber Glass Sampling Procedure</td>
</tr>
<tr>
<td>020</td>
<td>Feed Rate/Pull Rate Balance for Envitco Melter</td>
</tr>
<tr>
<td>021</td>
<td>Melter Drain Stream Sampling</td>
</tr>
<tr>
<td>024</td>
<td>Control of Non-conforming Items and Activities</td>
</tr>
<tr>
<td>025</td>
<td>Issuing and Archiving Research Notebooks</td>
</tr>
<tr>
<td>029</td>
<td>Weighing</td>
</tr>
<tr>
<td>030</td>
<td>Density of Glass by Bouyancy</td>
</tr>
<tr>
<td>034</td>
<td>Verification, Reporting, and Filing of Analytical Results</td>
</tr>
<tr>
<td>035</td>
<td>Verification of Calculations</td>
</tr>
<tr>
<td>036</td>
<td>Slurry Feed System: Operation and Maintenance</td>
</tr>
<tr>
<td>037</td>
<td>Envitco Offgas System: Operation and Maintenance</td>
</tr>
<tr>
<td>040</td>
<td>Receiving and Inspection</td>
</tr>
<tr>
<td>041</td>
<td>Data Acquisition System</td>
</tr>
<tr>
<td>042</td>
<td>Thermocouple Calibration</td>
</tr>
</tbody>
</table>
5.3 Process Control Plan

The process will primarily be controlled through monitoring of key variables and the use of instructions or SOPs being implemented by operators and technicians. Example SOPs were listed in Section 5.2 and the target parameters of key variables for the start-up of the melter were identified in Section 5.1. Envitco engineers will be operating the melter with assistance from CETL technicians. Technician training will be conducted in accordance with Section 14.3 of this Work Plan.

The process controls will be applied through automatic, manual, and administrative methods. Automatic controls are those devices that are integrated into the melter design to automatically maintain processing conditions (i.e., power input and temperature). Manual controls will also be adjusted by the operator based on experience, instructions and training. Manual controls are generally applied to parameters that are clearly monitored and adjusted to a set range (e.g. temperature, flow rate). A third control is administrative, which is applied to subjective characteristics, where it is difficult or impossible to establish definitive bounds. The controls will be applied to variables such as feed distribution, power distribution, and other parameters that will not or can not be automated or bounded as described above. These controls will be guided by the Envitco engineer(s) responsible for the demonstration.

Some SOPs establish acceptable ranges of operation for process control variables. Standard operating ranges for variables that are waste or glass-chemistry-specific will be established during the process optimization period before the demonstration run. These parameters will be inserted into the appropriate SOPs and all operators will be instructed as to their existence and use.

Certain safety interlocks also exist within the melter control system. These interlocks are automatic and can only be reset if the limits of the interlock are met. For example, if cooling water is lost and the supply pressure is insufficient, the melter power will be turned off to prevent an overheating situation.

Sampling will be placed under administrative controls through the use of operator instructions that will define all of the sample requirements prior to the testing program. This will include instruction on sample size, sample identification, storage and handling, records, and other requirements as necessary.

The following table summarizes the anticipated control parameters for the POPT demonstration run.
Table 5.3-1 Control Parameter Summary

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Control</th>
<th>Method</th>
<th>Indication</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surrogate Composition</td>
<td>Administrative</td>
<td>Prequalification</td>
<td>Chem. Analysis</td>
</tr>
<tr>
<td>Feed Composition</td>
<td>Administrative</td>
<td>Batch Sheets with verification</td>
<td>Chem. Analysis</td>
</tr>
<tr>
<td>Feed Rate</td>
<td>Administrative, Manual</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Magnetic flow monitor</td>
</tr>
<tr>
<td>Temperature (melter)</td>
<td>Administrative, Manual</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Thermocouple/ readout</td>
</tr>
<tr>
<td>Temperature (plenum)</td>
<td>Automatic, Manual</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Thermocouple/ readout</td>
</tr>
<tr>
<td>Feed Distribution</td>
<td>Administrative</td>
<td>Visual Observation</td>
<td></td>
</tr>
<tr>
<td>Glass Level</td>
<td>Automatic, Administrative</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Level Gauge/ readout</td>
</tr>
<tr>
<td>Melt Rate</td>
<td>Administrative, Manual</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Visual, feed rate correlation</td>
</tr>
<tr>
<td>Glass Pour Rate</td>
<td>Manual</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Automated weigh vs. time integration</td>
</tr>
<tr>
<td>Cooling Water System</td>
<td>Administrative, Manual</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Process interlocks, flow monitors, setpoint control</td>
</tr>
<tr>
<td>Offgas Status</td>
<td>Manual, Automatic</td>
<td>Operator Instruction, Parameter Limits</td>
<td>Plenum Pressure</td>
</tr>
</tbody>
</table>

Due to the test nature of the EV-101 melter, most controls are manual. The control system for the POPT demonstration is not as sophisticated as production-scale systems. Envitco has supplied automated systems for previous vitrification units. The TVS included a computerized control system capable of remotely manufacturing melter feed, operating the melter, and monitoring the offgas treatment system which contained its own automated control system. The TVS control system was also capable of automatically controlling the key process variables of power input, melter plenum pressure, feed rate and glass drain rate. This experience will be used to provide preliminary full-scale design data in conjunction with the manual control experience of the POPT demonstration.

5.4 Test Logs

Three types of test logs will be included with the Final Report. Test logs will be in the form of copies of the melter demonstration notebook, vitrification system round sheets and print-outs of the information gathered by the data acquisition system.
The melter demonstration notebook is a research notebook that is issued to the POPT project for use in recording the activities that occur during the POPT demonstration run. Entries in this book are made by the technicians and engineers that are performing the demonstration.

Vitrification system round sheets are used to periodically log data that is not monitored by the automatic data acquisition system and to assist in making sure key variables of the system are being monitored. These round sheets can often be used to note trends in system performance and provide an opportunity to change process control parameters before they reach an alarm condition. Copies of the round sheets will be included with the Final Report. The following is a list of key variables included on the round sheet for the POPT demonstration:

- Date and Time
- Operators Initials
- Melter Power Parameters (A, V, kW)
- Cooling-Water Flow and Temperature
- Protective Gas Flow and Pressure
- Feed System Parameters (feed rate, temperature, etc.)
- Offgas System Parameters (plenum pressure, exit temperature, etc.)

The information that is gathered by the automatic data acquisition system will be included in the Final Report. Graphs representing the data will be presented along with summaries of the data.

5.5 Video Tapes

Standard VHS video tapes will be used to record the entire 72-hour POPT demonstration run. The video tapes will be treated like any other sample for the POPT. They will be controlled according to the Sampling, Data Collection and Analysis Plan. They will be included with the transfer of samples that will occur concurrent with the Final Report.
6.0 EQUIPMENT AND MATERIALS

The following equipment and materials will be used during the Treatment Recipe Development phase of the POPT:

- Deltech Crucible Melting Furnace (model DT-31-12-RS)
- Electronic Weigh Scales
- Slurry Viscometer
- Miscellaneous Lab Ware (sample dishes, flasks, etc.)
- Sample Containers
- Silica Crucibles (alumina and high-temperature clay, as necessary)
- Silo Surrogate
- Glass-Making Additives (soda, lime, lithium, etc.)

The following equipment and materials will be used during the POPT demonstration run:

- Envitco WASTE-VIT® EV-101 Melter
- Air Pollution Control Equipment
  - film cooler
  - electrostatic precipitator
- Slurry Preparation Equipment
  - surrogate holding tanks with mixers
  - mixing tank
  - feed tank with mixer
  - recirculation and transfer pumps
- Glass / Product Handling Equipment
  - roller conveyors
  - 30 and 55 gallon drums
  - forklift
- Sampling Equipment
  - stainless steel glass sampling plate
  - stainless steel sample pans
  - sample containers
- Demonstration Surrogate
- Glass Making Additives
- Utilities (electricity, water, compressed air, nitrogen)
7.0 SAMPLING DATA COLLECTION AND ANALYSIS PLAN

The objective of the Sampling, Data Collection, and Analysis Plan is to provide a sufficient level of confidence that the data collected from the POPT will support the data requirements. The following is a description of the data requirements, samples and data that will be collected during the POPT. The types of analyses to be performed are also included on the summary tables to provide a cross reference to Section 4.0 of this Work Plan.

7.1 Sample Points and Data Requirements

7.1.1 Data Requirements

In order to assess the effectiveness of Joule-heated vitrification for the Silos 1 and 2 residues, data must be generated to evaluate the product performance, waste loading and bulking factor. The testing will also provide data for the full-scale process design. These requirements will be met through testing and data collection in the following areas:

1. Development of treatment recipes that meet the following criteria:
   - Leach at less than 50% of the TCLP limits
   - Leach at less than the UTS limits
   - Exhibit a compressive strength greater than 50 psi
   - Homogeneous physical form
   - Minimize dusting and particulate matter
   - Possess suitable properties for vitrification based on the following properties:
     - Resistivity
     - Viscosity
     - Liquidus point
     - Annealing point

2. Obtain verification of crucible melts composition compared to the batch preparation sheets

3. Develop a rheologically suitable slurry melter feed

4. Develop a mass balance through compositional analysis and monitoring of system inputs and outputs

5. Develop an energy balance based on system inputs and outputs

6. Determine the Bulking Factor

7. Determine the Waste Loading

8. Meet the product performance criteria for the demonstration glass as noted above for the recipe development glass

9. Produce full-scale process input data to meet Section C, Table F2 of the contract including:  

000048
4. Characterize secondary waste including:
   - Spent melter components
   - Offgas treatment filters and residues
   - Possible metals and salt drain material
   - Wastewaters

7.1.2 Sample Points

A narrative description of each of the sample points is presented in the following subsections. The sample points will be further refined based on the process optimization work prior to the initiation of the demonstration run. Any changes to the sampling points, analyses and frequency noted here will be transmitted to FDF prior to commencing the demonstration run.

7.1.2.1 Treatment Recipe Development

The treatment recipes for the three (3) surrogate wastes will be developed at the laboratory scale. One (1) set of glass formulations will be developed to pass the Toxic Characteristic Leaching Procedure (TCLP) with imposed limits of less than 50% of the 40 CFR 261 limits. A second set of glasses will be developed to meet the Universal Treatment Standards (UTS) assuming that the stricter durability requirements mandate a change in the glass chemistry.

The surrogate preparation and recipe development schedules will be provided to FDF once they are finalized. This will allow FDF to inspect and collect samples at FDF's discretion. Any samples collected by FDF will be analyzed by FDF at their cost.

Table 7.1-1 - Recipe Development Sample Summary summarizes the sample points, frequency, and data requirement. A brief description of each sample family, corresponding to Table 7.1-1, is presented below.

Surrogate Sampling - Raw Materials, Surrogate Mix, and Dry Lab Surrogate

The raw materials used to manufacture the surrogate recipes will be purchased with a vendor-supplied certificate of analysis (COA) and will not be analyzed for chemical composition. The COA will be used for verifying chemical purity. One (1) sample of each silica source will be taken and analyzed for particle size distribution to verify compliance with Section C, Appendix C of the contract. One (1) sample of each raw material will also be tested for loss on drying (LOD) at
105°C. The resulting water content results will be used as correction terms for the development of the batch sheets.

Silo 1, 2 and Demonstration surrogate mixes that simulate the in-situ silo residue will be made in accordance with Section C, Appendix C of the contract. A three (3) liter sample of each surrogate mix will be provided to FDF for verification. Samples of the surrogate mixes will then be archived until the conclusion of the POPT.

The Silo 1, 2 and Demonstration surrogates used for the treatment recipe development will be made in single large batches of dry chemicals. The formulas developed from the surrogate mixes plus Bentogrout™ will be used without the addition of water. Each of the three (3) dry surrogates will be sampled. A single sample of each recipe will be sent to CELS - Coming Laboratory Services (CELS) for verification of chemical makeup with a second sample archived until the conclusion of the POPT.

Table 7.1-1 – Recipe Development Sample Summary

<table>
<thead>
<tr>
<th>Sample Point</th>
<th>Analysis Quantity</th>
<th>Analytical Methods</th>
<th>Supports Data Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw Materials</td>
<td>4</td>
<td>COAs, Sieve Testing, LOD</td>
<td>Verification of surrogate</td>
</tr>
<tr>
<td>Surrogate Mix</td>
<td>3</td>
<td>LOD, Density, Plasticity, TCLP</td>
<td>Verification of surrogate</td>
</tr>
<tr>
<td>Dry Lab Surrogate</td>
<td>3</td>
<td>QCA, LOI, LOD, TCLP</td>
<td>Development of treatment recipes</td>
</tr>
<tr>
<td>TCLP &amp; UTS Series Glasses</td>
<td>15</td>
<td>QCA, Redox, Viscosity</td>
<td>Development of treatment recipes and recipe database</td>
</tr>
<tr>
<td>TCLP &amp; UTS Series Glasses</td>
<td>45</td>
<td>TCLP</td>
<td>Development of treatment recipes</td>
</tr>
<tr>
<td>Treatment Recipe Glass</td>
<td>18</td>
<td>TCLP, QCA, Annealing PL</td>
<td>Development of treatment recipes</td>
</tr>
<tr>
<td>Treatment Recipe Glass</td>
<td>6</td>
<td>ASTM C-773</td>
<td>Development of treatment recipes – compression testing</td>
</tr>
<tr>
<td>Treatment Recipe Glass</td>
<td>6</td>
<td>Viscosity, Resistivity, Liquidus</td>
<td>Full-scale Design</td>
</tr>
<tr>
<td>Treatment Recipe Glass</td>
<td>0</td>
<td>FDF defined</td>
<td>FDF durability evaluation</td>
</tr>
</tbody>
</table>

LOD: loss on drying
QCA: quantitative chemical analysis
COA: Certificates of Analysis
TCLP: Toxicity Characteristic Leaching Procedure
Batch Sampling

Sampling of the surrogate plus glass additives (batch) will not be done. It is assumed that the combination of dry chemicals to the batches will be sufficiently controlled using batch sheets with established ranges of error. Batch chemistry confirmation will be done through glass analysis.

TCLP and UTS Series Glasses

Each glass that is homogenous, as determined by visual observation, will be sampled. Samples of the glass will be taken from a portion of the molten glass that is poured into a mold. The remainder of the sample will be archived until the conclusion of the POPT. The sample will be size-reduced to meet TCLP requirements.

Approximately forty-five (45) total samples from the TCLP and UTS recipe development will be sent for TCLP analysis. Fifteen (15) samples will be split and a portion sent to CELS for quantitative chemical analysis. This will provide verification of glass chemistry based on the batch sheets and surrogate analysis as noted in Section 3.0. These samples will also represent the glass region that forms an acceptable product.

Treatment Recipe Glass

After the glasses have been fabricated and tested according to Section 3.0, the best performing glass for each recipe will then be manufactured in crucibles to provide samples of sufficient quantity for analysis. An FDF-approved laboratory will be used to perform the TCLP on three (3) independently melted samples of each recipe. The leachate of each of the three (3) samples will be analyzed twice. This will result in eighteen (18) total samples and thirty-six (36) data points, assuming that both TC and UTS recipes are required.

One (1) sample of each recipe will be sent to CELS for analysis by ASTM C 773 Modified (entitled Standard Test Method for Compressive (Crushing) Strength of Fired Whiteware Materials). CELS will prepare ten (10) test specimens from each sample resulting in ten (10) data points for each recipe. Assuming that six (6) different recipes are tested, the total number of compressive strength data points totals sixty (60).

Various tests for properties, such as electrical resistivity and high temperature viscosity, will be conducted to provide Joule-heated melter design information. CELS will be used to perform the various design input tests. See Section 3.0 of this Work Plan for further detail on processability issues.
A sufficient quantity of each treatment recipe glass will be manufactured in crucibles to meet the Appendix G, Table G2 requirements for FDF durability testing samples.

7.1.2.2 Proof of Principle Testing

Sampling for the POPT demonstration will commence after achieving steady-state during the process optimization activities. The POPT surrogate preparation and POPT demonstration schedules will be provided to FDF once they are finalized. This will allow FDF to inspect and collect samples at FDF's discretion. Any samples collected by FDF will be analyzed by FDF at no cost to the Seller.

The random samples noted in each of the following sections will be chosen prior to initiation of the POPT. With concurrence from FDF, Envitco will choose three (3) of the sample periods from the population described in each section at which time sufficient surrogate, feed, glass, or secondary waste will be collected. The sample period that is chosen will then be applied to the sampling of each stream so that the mass balance will have better correlation to a defined period of time. Having all of the samples collected on a consistent, defined time interval will also allow for correlation of the data being collected with the sample compositions and properties.

Figure 7.1-1 depicts the POPT process flow with the sampling points identified for each input/output. The sample points referred to in Table 7.1-2 correspond to the sample points identified in Figure 7.1-1 and are described briefly.
FIGURE 7.1 - Sample Point Flow Diagram

GLASS FORMING ADDITIVES

PT. 2: GLASS FORMING ADDITIVES

GLASS FORMING ADDITIVES

SURROGATE BLENDING

STAGING OF MANUFACTURED SURROGATE

FEED BLENDING (SURROGATE + ADDITIVES)

MIX TANK

PT. 3A: MELTER FEED SAMPLE FOR MAT'L BALANCE (EVERY 3 HRS)

BLENDED FEED TANK

PT. 1: SURROGATE SAMPLE FOR AUTHORIZATION TO PROCEED

PT. 1A: SURROGATE SAMPLE FOR ARCHIVE (EACH BATCH)

PT. 3: FEED SAMPLE FOR MAT'L BALANCE

PT. 6: GLASS PT. 4: (EVERY 3 HRS) (FLOW, T)

TO STACK

OFFGAS TREATMENT SYSTEM

PT. 7: OFFGAS SAMPLES (UNTREATED) (FLOW, T, P)

PT. 7: OFFGAS SAMPLES (UNTREATED) (FLOW, T, P)

EV-101 MELTER

PT. 8: GLASS SAMPLE (EVERY 3 HRS) (FLOW, T)

PT. 4: SALT SAMPLE (FLOW)

PT. 8: OFFGAS SOLIDS RESIDUE SAMPLE (EVERY 3 HRS)

PT. 5: PRECIPITATED METALS SAMPLE (FLOW)
# Table 7.1-2 – POPT Demonstration Sample Summary

<table>
<thead>
<tr>
<th>Sample Point</th>
<th>Sample Type</th>
<th>Sample Quantity</th>
<th>Analysis Quantity</th>
<th>Analytical Methods</th>
<th>Supports Data Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>POPT Surrogate</td>
<td>10</td>
<td>3</td>
<td>LOD, LOI, TCLP, QCA, density</td>
<td>Waste Loading, Bulking Factor, Mass Balance</td>
</tr>
<tr>
<td>2</td>
<td>Glass Forming Additives</td>
<td>Each Additive</td>
<td>N/A</td>
<td>Archive</td>
<td>Archive</td>
</tr>
<tr>
<td>3</td>
<td>Melter Feed</td>
<td>24</td>
<td>3</td>
<td>LOD, LOI, QCA, density, Viscosity</td>
<td>Waste Loading, Bulking Factor, Mass Balance</td>
</tr>
<tr>
<td>6</td>
<td>Treated Surrogate Glass</td>
<td>24</td>
<td>3</td>
<td>TCLP</td>
<td>Random Samples Required By Section</td>
</tr>
<tr>
<td>6</td>
<td>Treated Surrogate Glass</td>
<td>189</td>
<td>0</td>
<td>FDF defined</td>
<td>FDF durability evaluation</td>
</tr>
<tr>
<td>6</td>
<td>Treated Surrogate Glass</td>
<td>24</td>
<td>12</td>
<td>TCLP, QCA, density, redox</td>
<td>Waste Loading, Bulking Factor, Mass Balance, Product Performance</td>
</tr>
<tr>
<td>6</td>
<td>Treated Surrogate Glass</td>
<td>TBD</td>
<td>3</td>
<td>Sieve</td>
<td>Product Performance</td>
</tr>
<tr>
<td>6</td>
<td>Treated Surrogate Glass</td>
<td>24</td>
<td>3</td>
<td>ASTM C 773</td>
<td>Product Performance</td>
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<td>7</td>
<td>Offgas – pre pollution control equipment</td>
<td>3</td>
<td>3</td>
<td>Methods 1 through 8 &amp; 29</td>
<td>Mass Balance, Offgas Equipment Design at Full-scale, Permitting Data</td>
</tr>
<tr>
<td>10</td>
<td>Offgas – post pollution control equipment</td>
<td>3</td>
<td>3</td>
<td>Methods 1 through 8 &amp; 29</td>
<td>Mass Balance, Offgas Equipment Design at Full-scale, Permitting Data</td>
</tr>
<tr>
<td>8</td>
<td>Offgas Treatment Residue</td>
<td>24</td>
<td>3</td>
<td>QCA, TSS, TDS, TCLP, CFR 261</td>
<td>Mass Balance, Characterization of Secondary Waste</td>
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<tr>
<td>5</td>
<td>Metal Drain</td>
<td>5</td>
<td>5</td>
<td>TCLP, QCA, XRD, density</td>
<td>Characterization of Secondary Waste, Mass Balance</td>
</tr>
<tr>
<td>4</td>
<td>Salt Drain</td>
<td>24</td>
<td>3</td>
<td>TCLP, QCA, density</td>
<td>Characterization of Secondary Waste, Mass Balance</td>
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Table 7.1-2 – POPT Demonstration Sample Summary (cont’d)

<table>
<thead>
<tr>
<th>Sample Point</th>
<th>Sample Type</th>
<th>Sample Quantity</th>
<th>Analysis Quantity</th>
<th>Analytical Methods</th>
<th>Supports Data Requirement</th>
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</thead>
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<tr>
<td>EV-101</td>
<td>Electrodes</td>
<td>3</td>
<td>3</td>
<td>SEM</td>
<td>Characterization of</td>
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<tr>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>Secondary Waste and</td>
</tr>
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<td></td>
<td>Support of Full-scale</td>
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<td></td>
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<td></td>
<td>Design</td>
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<tr>
<td>EV-101</td>
<td>Refractory</td>
<td>3</td>
<td>3</td>
<td>TCLP</td>
<td>Characterization of</td>
</tr>
<tr>
<td></td>
<td></td>
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<td></td>
<td></td>
<td>Secondary Waste</td>
</tr>
<tr>
<td>EV-101</td>
<td>Bottom of Melter Glass</td>
<td>3</td>
<td>3</td>
<td>XRD, SEM, TCLP, QCA</td>
<td>Characterization of</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Secondary Waste</td>
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<tr>
<td>Various</td>
<td>Wastewater</td>
<td>TBD</td>
<td>3</td>
<td>TSS, TDS, TCLP, QCA</td>
<td>Characterization of</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Secondary Waste,</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Mass Balance</td>
</tr>
</tbody>
</table>

LOD: loss on drying
TSS: total suspended solids
TDS: total dissolved solids
XRD: X-ray Diffraction
QCA: quantitative chemical analysis
SEM: scanning electron microscope
TCLP: Toxicity Characteristic Leaching Procedure

Surrogate Sampling – Sample Point 1

Five (5) batches of surrogate slurry will be made prior to the 72-hour test. Verification of the surrogate preparation will be done by FDF via analysis of the first batch manufactured. None of the subsequent batches will be verified prior to treatment. It is assumed that if the method to manufacture the first batch is acceptable, it will be acceptable for all subsequent batches. All batch sheets will be provided to FDF for verification of surrogate preparation as part of the Final Report. The subsequent batches will be sampled for Seller analysis.

One (1) sample will be taken for each surrogate batch and analyzed or archived until the conclusion of the POPT. Three (3) samples, chosen at random, will be sent to an FDF-approved laboratory for determination of loss on drying (LOD) at 105°C per ASTM D 2216, and dry and wet density. Loss on ignition (LOI) at 900°C and chemical composition determination will be performed at CELS.

Glass Forming Additive Sampling – Sample Point 2

Glass-forming additives chosen during the treatment recipe development phase will be used in the demonstration run. Samples of each glass-forming additive will be taken and archived for the duration of the project.
Melter Feed Sampling – Sample Point 3 and 3A

The melter feed will be a slurry consisting of the surrogate slurry, Bentogrount and glass additives in the form of powders, granules or liquids. The melter feed slurry will be sampled every three (3) hours during the POPT. This will result in twenty-four (24) total samples. Three (3) samples, chosen at random prior to the test, will be sent to an FDF-approved laboratory for determination of loss on drying (LOD) at 105°C and dry and wet density. Loss on ignition (LOI) at 900°C and chemical composition determination will be performed at CELS. The remaining samples will be archived until the conclusion of the POPT.

The feed shall also be monitored for the mass balance. Data collected will include the temperature in the feed tank and flow rate into the melter.

Treated Surrogate – Sample Point 6

One (1) sample of the treated surrogate will be taken from the glass pour stream every three (3) hours during the POPT. This will result in twenty-four (24) glass samples corresponding to the twenty-four (24) melter feed samples. The following sections describe the sampling and analyses to be performed.

Random TCLP Samples

Three (3) glass samples will be chosen at random per Section C.4.2.2 - Samples. These samples will be sent to an FDF-approved laboratory for TCLP testing. The leachate from each sample will be analyzed twice, resulting in a total of six (6) data points.

FDF Durability Samples

Three (3) of the twenty-four (24) sample periods will be randomly selected, with FDF’s concurrence, for collection of FDF durability testing samples. A sufficient quantity of glass will be collected to manufacture the samples noted in Section C, Table G2 of the contract.

Treated Surrogate Performance Samples

A series of tests will be performed on the treated surrogate samples in accordance with the contract for appearance, compressive strength, standing liquid content, TCLP, dusting and particulate content, RCRA characteristics and FDF durability tests.
Appearance

According to Section C.4.2.3.1 Paragraph A of the contract, the treated surrogate residue shall appear uniform and homogeneous. The treated surrogate residue appearance will be logged on the sampling log sheet when the sample is taken from the glass pour stream. Visual (non-magnified) observation will be used to determine if the sample is homogeneous glass, non-homogeneous (color variations or streaks) but fully glass-like, or non-homogeneous and non-glass-like. Each of the twenty-four (24) samples will have an appearance evaluation.

Compressive Strength

According to Section C.4.2.3.1 Paragraph B of the contract, the treated surrogate should exhibit a compressive strength of at least 50 psi per ASTM C 39. As a more appropriate test for glass, Envitco will provide compressive strength results from the ASTM C 773 test developed for ceramic materials. The compressive strength of the glass will be greater than 50 psi upon testing via ASTM C 773.

Based on historical data developed by Envitco, the compressive strength of the glass should be greater than 10,000 psi. Based on this experience, only three (3) randomly selected samples will be sent to CELS for compressive strength determination. The CELS methodology includes making ten (10) test specimens from each sample. This would result in thirty (30) compressive strength data points for evaluation.

Liquid Content

According to Section C.4.2.3.1 Paragraph C of the contract, the treated surrogate should contain no standing liquids per ANS 55.1 testing. No samples of the treated surrogate will be tested by the ANS 55.1 method. No standing nor free liquids will be associated with the treated surrogate as it is processed at temperatures in excess of 1000°C and will be sampled using a dry collection device.
According to Section C.4.2.3.1 Paragraph D of the contract, the treated surrogate will be deemed acceptable if the leachate concentrations are less than 50% of the RCRA limit. In addition to the eight (8) standard TCLP metals, the concentrations of Antimony (Sb), Beryllium (Be), Nickel (Ni), Thallium (Tl), Vanadium (V), and Zinc (Zn) will be determined in the leachate. TCLP testing for toxic organic material will not be conducted.

Twelve (12) of the twenty-four (24) samples will be randomly selected to be sent for TCLP analysis. A duplicate analysis of the leachate will be performed resulting in twenty-four (24) data points. All TCLP samples will be sent to an FDF-approved laboratory.

Dusting/Particulate

Section C.4.2.3.1 Paragraph E of the contract requires the treated disposal package contain no more than 1 wt.% of less-than 10 micron diameter particles, nor more than 15 wt.% of less-than 200 micron particles. The contents of three (3) treated surrogate collection containers, chosen at random, will be analyzed for particle size distribution. The contents of the container will be emptied, and all particles less than ¼ inch (6.35 mm) will be sent for a sieve analysis at CELS using ASTM Method D 422. A determination will be made as to how much material is less than 200 microns and how much is less than 10 microns.

RCRA Characteristics

Section C.4.2.3.1 Paragraph F of the contract requires that the treated waste form shall not exhibit any hazardous characteristics as defined by 40 CFR 261 Subpart C. Nor shall the treated waste be listed as hazardous waste.

The glass wasteform will not be sampled and analyzed to determine the hazardous characteristics as defined in 40 CFR 261.20 through 261.23. Subsection 261.24 is the requirement for toxicity which is addressed separately in the product performance requirements. The following is the rationale to support this assumption:

Subsection 261.21 – Glass is not an alcohol-containing liquid. Glass is not capable, under standard temperature and pressure, of causing fire through friction, absorption of water nor spontaneous chemical changes because glass is a smooth solid that does not react...
vigorously with water and does not undergo spontaneous chemical changes. Glass is not an ignitable compressed gas as defined in 49 CFR 173.300. Glass is not an oxidizer as it is already an oxidized matrix of metallic and non-metallic elements.

Subsection 261.22 - The glass will not be corrosive as it is not a liquid with pH of <2 or >12.5 nor will glass corrode steel at 55°C as defined in the Subsection.

Subsection 261.23 - The glass wasteform will not be reactive. Glass is a very stable material which will not react violently with, nor release gases when in contact with, water. The glass will not be explosive nor will the glass contain cyanides or sulfates that will generate toxic gases when subjected to conditions where the pH is between 2 and 12.5.

Subsection 261.24 - Toxicity will be tested in accordance with the SOW. The concentration of the eight standard metals will be determined along with Antimony, Berillium, Nickel, Thallium, Vanadium, and Zinc.

Mass/Energy Balance Input

The twelve (12) samples of treated surrogate that are chosen for TCLP analysis will be split to have a portion sent to CELS for quantitative chemical analysis (QCA) and redox state via iron oxidation ratio determination. The composition of the glass will provide input to the mass balance and verify that the target glass composition developed at the lab scale is being manufactured.

The density of the twelve (12) samples sent for QCA will also be determined. This will be used as input to the mass balance and for the Bulking Factor determination.

Offgas Sampling – Sample Points 7 and 10

The offgas will be sampled at two (2) locations; prior to any offgas treatment equipment and at the exhaust stack after the treatment equipment. The temperature and pressure of the offgas will be monitored at a series of points along the treatment train. Offgas flow rates will be measured at the sampling points as standard procedure prior to sampling the offgas stream. The data will feed into the mass/energy balance and will be used in the full-scale remediation equipment design.
Offgas sampling will occur between the 24th and 72nd hours of the test. Three (3) samples will be taken at both the melter exit and the stack using each of the following EPA methods:

- Reference Methods 1, 2, 3 and 4
- Method 5 for particulate loading
- Method 6C for sulfur dioxide
- Method 7E for NOx
- Method 8 sulfur dioxide and sulfur trioxide, and
- Method 29 for volatile metals (including Mo)

A final report will be provided by TRIGON, the offgas sampling contractor. The report will include:

- Summary of results,
- Sampling and analytical procedures,
- Location of sampling ports and point,
- Example calculations,
- Field data sheets,
- Laboratory data, and
- Calibration data.

Secondary Waste – Sample Points 4, 5, 8 & 9

It is anticipated that offgas treatment residues, salt drain material and metals drain material will be generated during the POPT. At the conclusion of the POPT, other secondary wastes such as spent refractory, electrodes, and spent offgas filters will be generated. Samples of each of these streams will be collected as part of the POPT to support characterization of secondary waste and for input to the mass balance.

Offgas Treatment Residue

The offgas treatment residue is expected to be a dry powder. This powder will be collected during the POPT. Samples of the powder will be collected every three (3) hours along with the melter feed and glass. Three (3) samples will be chosen at random for analysis. The powder will be analyzed by QCA, TCLP and 40 CFR 261 Hazardous Characteristic tests.
Metals Drain

The metals drain will be opened for a short period between the 48th and 72nd hour of the test. A sample will be taken of the material that first exits the drain orifice. A second sample will be taken during the drain. The timing of the second sample will depend on the flow rate and observation of the material drained. Three (3) samples will also be taken from the collection container. If there are separate phases in the drained material, a composite sample will be made and a sample of each of the phases will be taken. Only two (2) analyses of separated phases are included in the POPT.

A total of five (5) metals drain samples; the two (2) drain samples, the composite sample from the two (2) drain samples and the two (2) separated phase samples, will be characterized via TCLP, QCA, x-ray diffraction (XRD) and density measurements as required by the makeup of each sample.

Salt Drain

The salt drain material will be sampled every three (3) hours along with the melter feed, glass, and offgas treatment residue. If no salt is draining from the salt drain during the sampling period, no sample will be taken and will be so noted in the sample log. A total of twenty-four (24) samples will be scheduled to be taken. Three (3) of the salt drain samples will be chosen at random for analysis. Testing will consist of TCLP, QCA and density measurement.

If a continuous salt drain occurs, the temperature and flow rate will be recorded in the POPT log along with the glass temperature and flow rate. This will support the mass/energy balance.

Spent Refractory and Electrodes

Upon completion of the test, six (6) samples of the refractory from the melter will be taken. The samples shall be collected from various areas of the melter once the glass has been removed. The six (6) samples will be analyzed by TCLP to determine if they are hazardous. The amount of refractory materials and their composition will be known prior to the POPT in order to support the characterization of secondary waste.

The electrodes will be photographed, weighed and measured to assess wear and wear patterns. Formation of secondary alloys, or 'sweating', will be observed and recorded. Three (3) electrode samples will be analyzed by scanning electron microscope (SEM) for characterization purposes. The bulk composition and the amount of electrode material will be known prior to the
POPT in order to support characterization of secondary waste and provide full-scale design input regarding electrode corrosion.

**Other Secondary Wastes**

The glass in the melter will be removed upon conclusion of the POPT in order to examine the bottom of the melter. Any visually observed phases that had separated from the glass will be sampled. Depending on the visual inspection, the sample may be analyzed via X-ray diffraction (XRD) to determine crystalline content, quantitative chemical analysis via dissolution or alkali fusion followed by spectrographic or atomic emission methodologies, TCLP or scanning electron microscopy (SEM). A total of three (3) of each of the analyses listed is included in this Work Plan. Any other samples and analysis will be the responsibility of FDF.

**Wastewater**

Any secondary waste streams that are aqueous liquids will be sampled as noted above for offgas treatment residues. The wastewater will be analyzed at an FDF-approved laboratory for TCLP, TSS, TDS and QCA which will include concentrations of As, Ba, Cd, Cr, Pb, and Se. This is in accordance with Section C.4.3.3 Wastewater Control of the contract.

**Archives**

All samples that are not sent for analysis will be archived in a secure location at the Seller's test facility. Only treated surrogate samples will be transferred to FDF upon completion of the POPT. All other archived samples will be disposed of unless FDF determines that they should be transmitted to FDF with the treated surrogate samples.
7.1.2.3  POPT Data Collection

Table 7.1-3 - Data Collection Summary summarizes the data points recorded during the demonstration run.

<table>
<thead>
<tr>
<th>Data Point</th>
<th>Frequency</th>
<th>Data Quantity</th>
<th>Supports Data Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melter Feed Rate</td>
<td>Hourly</td>
<td>72</td>
<td>Mass Balance, Full-scale Design Data</td>
</tr>
<tr>
<td>Furnace Temps.</td>
<td>Hourly</td>
<td>72</td>
<td>Energy Balance, Full-scale Design Data</td>
</tr>
<tr>
<td>Offgas Parameters</td>
<td>Hourly</td>
<td>72</td>
<td>Energy Balance, Full-scale Design Data</td>
</tr>
<tr>
<td>Melter Plenum Pressure</td>
<td>Hourly</td>
<td>72</td>
<td>Energy Balance, Full-scale Design Data</td>
</tr>
<tr>
<td>Glass Pour Rate</td>
<td>Hourly</td>
<td>72</td>
<td>Mass Balance, Full-scale Design Data</td>
</tr>
<tr>
<td>Melter Power</td>
<td>Hourly</td>
<td>72</td>
<td>Energy Balance, Full-scale Design Data</td>
</tr>
<tr>
<td>Offgas Equipment Parameters</td>
<td>Hourly</td>
<td>72</td>
<td>Energy Balance, Mass Balance, Full-scale Design Data</td>
</tr>
</tbody>
</table>

7.2  Sampling Logs

All samples will be logged on a master sample log sheet. Each sample collected will be given an individual, distinct number and will be labeled with the sample number, date, time, approximate sample size, batch number (if applicable) and sampler's initials. After the log sheet and sample label have been filled in, the sample will be transferred to the secure sample storage area where the master log is stored.

The sample log sheet will be arranged to allow easy use by the operators. Each sample period will be shown with the samples required during that period. It will be the responsibility of the supervising engineer to insure that all of the samples are collected as noted on the log sheet.

Any special circumstances regarding sampling will be recorded in the POPT demonstration log book. Copies of any entries regarding sampling will be included with the sample log sheets in the Final Report.
7.3 Sample Chain of Custody

When a sample is ready for analysis or transfer out of the secure sample storage area, a Chain of Custody Record (COC) is generated. COCs are sequentially numbered and stored in the secure sample storage area.

The COC contains the following information:

- Name of the laboratory and contact person,
- Sample number,
- Date and time, when the sample was collected with sampler’s initials,
- Analysis being requested,
- Sample container description,
- Person transferring sample with data and time transferred,
- Recipient’s name with date and time of receipt,
- Delivery method and tracking number, and
- Comments regarding any special care.

A copy of all COCs that are used for the POPT will be transferred to FDF with the Final Report.

7.4 Analytical Laboratory Logs

The logs from the analytical laboratories will include the standard data reports generated as a result of the testing. Compuchem and CELS will provide logs based on their standard data package guidance which includes information on the following:

- sample number,
- analysis date, time, and method,
- QA/QC data including:
  - blanks,
  - spikes,
  - calibrations,
  - detection limits
- results.

Oxford Laboratories, Inc. will perform the analyses on the offgas samples. Oxford Laboratories will provide logs based on their standard data package which includes information on the following:

- written report,
- analytical narrative including:
- sample number,
- analysis date, time, and method,
- QA/QC data including:
  - blanks,
  - spikes,
  - calibrations,
  - detection limits
- results and calculation sheets.

Copies of the vendor laboratory logs and the offgas sampling final report will be included with the POPT Final Report.

7.5 Analytical Laboratory Procedures

Each laboratory will follow their own internal procedures. CELS will follow their own Procedure No. 108001-002 entitled CELS Operating Procedure which covers the following areas of operation:

- Inquiry – call reports and pricing
- Sample Receipt and Input
- Sample Tracking
- Technical and Quality Issues and Requirements
- Data Output
- Invoicing
- CELS Accounts System
- Laboratory Information Management System (TSLIMS) Procedures
- Accounting Procedures
- Staff Information

Compuchem will follow their internal procedures as follows:

- Receiving Samples
- Storing Samples
- Handling Sample Extract Requests
- Preparing Holding Blanks
- Checking and Recording pH of Metals, Cyanides, Phenols, and Wet Chemistry Water Samples
- Ensuring Sample Security
- Reading and Recording Sample Temperature
- Various standard SOPs regarding business practices per the QA Plan.
The offgas emissions sampling contractor, Trigon, will adhere to its QA Manual that includes the necessary information to insure quality sampling regarding:

- Personnel Qualifications
- Personnel Training
- Procedural Compliance
- Sample Acquisition and Custody Documentation
- Instrument Maintenance
- Equipment Calibration and Records.
8.0 DATA MANAGEMENT PLAN

The purpose of data management is to ensure the collected data is properly controlled and available for analysis, evaluation and interpretation. For the POPT, project data will be collected in various forms: project notebooks, electronic files, log sheets and analytical data packages. The original data will be stored in secure files at CETL and Envitco, as appropriate.

Project notebooks will be used for the recipe development and demonstration run portions of this project. All project notebooks are uniquely numbered and permanently bound with sequentially numbered pages. The notebook will be a project-specific notebook which will be assigned to the individuals working on the project.

All records management and reporting for the analyses performed during the Proof of Principle Test program will follow the POPT Quality Assurance Plan.
9.0 DATA ANALYSIS, EVALUATION, AND INTERPRETATION

The analysis, evaluation, and interpretation of the collected data will be used to determine the performance of the POPT.

9.1 Mass and Energy Balance – Primary Waste Stream

Process data and analytical results generated by subcontract labs will be used to generate a material and energy balance for the demonstration phase of the POPT. The material and energy balance will be presented in two phases.

The first phase will be an overall material and energy balance. The process balance identifies the major product and secondary wastes generated from the treatment of the demonstration surrogate. Incoming surrogate waste characteristics, treated offgas characteristics, treated surrogate characteristics, secondary waste stream characteristics and utility requirements (including water, electrical, and combustion gases) will be calculated.

The second phase will produce individual material and energy balances for each major unit operation. Individual unit operation material and energy balances depict inlet, outlet (especially secondary waste streams), and energy consumption/production.

Around the melter system, the tie component method will be used in conjunction with traditional material balance measurements. Use of the tie component method supplements direct measurement of offgas emissions (Whyatt, 1996). When used in conjunction with empirical material balance methods (i.e., measurement of masses of feed and glass materials combined with direct offgas measurements), the tie component method provides a more complete and accurate material balance of the melter system.

Preliminary Material Balance

A preliminary material balance using the revised Demonstration Surrogate (Contract Modification No. 01 – June 26, 1998) was used to predict the quantity of treated surrogate (glass), secondary waste, and offgas emissions that may be produced during the POPT. Table 9.1-1 presents the estimated input and output streams for the EV-101 on an oxide basis. (Note: The Stream ID on Table 9.1-1 corresponds to the sample points identified in Figure 7.1-1.)
<table>
<thead>
<tr>
<th>Component</th>
<th>Stream Description</th>
<th>Sodium Carbonate</th>
<th>Calcium Carbonate</th>
<th>Lithium Carbonate</th>
<th>Sulfur Carbonate</th>
<th>Melt Feed</th>
<th>POFT Product</th>
<th>POFT Salt Drain</th>
<th>Vitrification Offgas</th>
<th>Metal Sludge Drain</th>
</tr>
</thead>
<tbody>
<tr>
<td>O2</td>
<td>kg/hr</td>
<td>kg/hr</td>
<td>kg/hr</td>
<td>kg/hr</td>
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</tbody>
</table>

1) Material balance based on the demo surrogate composition submitted to EnviCO by FDF on 6/25/66.
2) TNV5 signifies Total Non-Volatile Oxides.
3) Partitioning to the salt phase was based on Low-Level Melt Demonstrations and a sulfate solubility limit of 1.0 w% in glass.
4) Based on a Soda-Line-Silica glass system.
5) The frequency of the metal drain will be variable. Composition will also be variable.

000069
The major inputs in Table 9.1-1 are grouped into Demonstration Surrogate (Stream 1) and Glass Forming Additives (Streams 2A-2C). The major inputs sum to produce the Melter Feed (Stream 3). Outputs are represented by POPT Product (Glass – Stream 6), POPT Salt Drain (Salt – Stream 4), Metal Sludge Drain (Precipitated Species – Stream 5), and Vitrification Offgas (Stream 7).

A SLLS glass was used as the target for the preliminary material balance using only calcium carbonate, lithium carbonate and sodium carbonate as the glass forming additives. At a surrogate slurry feed rate of 110.78 kg/hr (on an oxide basis), which is equivalent to 2600 kg/24-hr on a wet basis, a glass drain rate of 37.6 kg/hr is predicted. This corresponds to a waste loading, as defined in the contract, of 81%. Assuming a glass density of 2800 kg/m³ (2.8 g/cm³), the corresponding bulking factor is calculated to be:

<table>
<thead>
<tr>
<th>In-situ Density*</th>
<th>Bulking Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Demo Surrogate</td>
<td>1.78 g/cm³</td>
</tr>
<tr>
<td>Silo 1 Surrogate</td>
<td>1.57 g/cm³</td>
</tr>
<tr>
<td>Silo 2 Surrogate</td>
<td>1.73 g/cm³</td>
</tr>
</tbody>
</table>

* Values taken from Contract Modification No. 03 and exclude the Bentogroat contribution.

9.2 Mass and Energy Balance – Secondary Waste Streams

Secondary waste streams are identified and included as an integral part of the overall and individual unit operation material and energy balances. Estimates of the quantity of secondary waste generated from the melter are presented below.

POPT Salt Drain

An estimate of the production rate of salt expected from the processing of the demonstration surrogate is presented in Table 9.1-1. When extrapolated over the entire demonstration, approximately 24 kg of salt, as SO₃, is predicted for the 72-hour demonstration. The preliminary material balance assumes sulfate, as SO₃, is soluble in the glass up to a solubility limit of 1.00 wt.% and any remaining sulfate partitions to the salt and offgas phases. The actual solubility of sulfate in the glass will be determined during the treatment recipe development and may differ from the value chosen for the preliminary material balance.

Metal Sludge Drain

For the purposes of the preliminary material balance, the quantity of metallic species drained from the melter was assumed to be zero. In the event metallic
species precipitate during the demonstration, samples and process data will be collected according to Section 7.0 of this Work Plan.

Offgas Emissions

An estimated offgas composition is presented in Table 9.2-1. Table 9.2-1 represents contributions from melter in-leakage, the propane/air burners, instrument air, and purge gases. Only the major volatile components to the offgas are presented in Table 9.2-1. The offgas contribution from the vitrification of melter feed (excluding in-leakage, combustion products, instrument air, and purge gas) is presented in Table 9.1-1 – Vitrification Offgas (Stream 7). Based on the preliminary material balance, approximately 420 scfm (273 K, 1 atm) of offgas is expected during the POPT demonstration. The total gaseous products presented in Table 9.2-1 represents a conservative estimate of the volumetric flow rate at the melter exhaust exit.

Table 9.2-1 – Preliminary Offgas Estimate

<table>
<thead>
<tr>
<th>Gaseous Components</th>
<th>Total Melter In-Leakage Air</th>
<th>Total Melter Instrument Air</th>
<th>Total Nitrogen Purge</th>
<th>Melter Feed to Offgas</th>
<th>Propane/ Air Burners</th>
<th>Total Gaseous Products</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ft³/min</td>
<td>ft³/min</td>
<td>ft³/min</td>
<td>ft³/min</td>
<td>ft³/min</td>
<td>ft³/min</td>
</tr>
<tr>
<td>NOx (as NO₂)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.04</td>
<td>0.00</td>
<td>0.04</td>
</tr>
<tr>
<td>NH₃</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
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<td>0.00</td>
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<tr>
<td>N₂</td>
<td>64.73</td>
<td>111.49</td>
<td>0.66</td>
<td>0.02</td>
<td>96.95</td>
<td>273.85</td>
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<tr>
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<td>0.04</td>
<td>0.00</td>
<td>1.97</td>
<td>15.28</td>
<td>17.32</td>
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<td>0.00</td>
<td>56.07</td>
<td>20.37</td>
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<td>0.00</td>
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<tr>
<td>SO₂ (as SO₃)</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.07</td>
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<td>0.78</td>
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<td>2.12</td>
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<tr>
<td>Total</td>
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<td>142.87</td>
<td>0.66</td>
<td>58.16</td>
<td>132.60</td>
<td>418.42</td>
</tr>
</tbody>
</table>
Lead and Molybdenum Products

Analysis of the glass, offgas, and any precipitate residues will be coupled with observations of electrode wear characteristics to assess the interactions of the molybdenum electrodes with the lead in the glass. This analysis will compliment the material balance and provide data for the scale-up of the system.

9.3 Data Evaluation

The criteria identified in Section 7.0 of this Work Plan will be used as the basis for evaluation of the POPT. The analytical results will be evaluated against the following programmatic goals:

- Produce up to six waste glass formulations that meet or exceed the durability requirements of:
  - less than 50% of the TCLP limits for metals, and/or
  - less than the Universal Treatment Standards.
- Demonstrate that a continuous, Joule-heated vitrification process can produce a waste glass that consistently meets or exceeds the criteria of:
  - uniform, homogeneous appearance,
  - compressive strength greater than 50 psi, as measured by ASTM C773,
  - durability at less than 50% of the toxicity characteristic for the eight (8) RCRA metals: As, Ba, Cd, Cr, Pb, Hg, Se, and Ag, and
  - immobilization of fine particulate surrogate so that the treated surrogate (i.e., glass) contains:
    - no more than 1 wt.% of less than 10 μm diameter particles, or
    - no more than 15 wt.% of less than 200 μm diameter particles
- Develop design data for a full-scale remediation facility.

9.4 Data Interpretation

In conjunction with process observations and operation logs, the results of the performance evaluation will be interpreted against the criteria of:

- Does the result indicate proper operation (i.e., was the test performed within specified parameters)?
- Does the result match anticipated or predicted performance?
- Can a satisfactory explanation be given for an unanticipated result?
- What improvements in design, operation, and/or equipment can be identified from the result?

The interpretive results will then be transferred, as applicable, to the design data effort for the full-scale treatment process.
10.0 HEALTH AND SAFETY REQUIREMENTS FOR POPT ACTIVITIES

POPT activities will be performed in accordance with CETL/Clemson University health and safety guidelines.
11.0 WASTE STREAM MANAGEMENT

Untreated surrogate, treated surrogate, and any secondary wastes will be analyzed prior to disposition. If the waste fails the requirements for disposal at a sanitary landfill, the materials will be handled, stored, and disposed in accordance with Clemson University's hazardous waste program as described in the Hazardous Waste Management Manual.

11.1 Regulatory Issues Specific to Testing Facility

CETL is an approved mixed-waste, TD&D laboratory for conducting treatability studies on hazardous and radioactive materials. As a result, CETL is licensed, permitted, and authorized by state and federal regulatory agencies to conduct demonstrations such as the Proof of Principle.
12.0 REPORTS

Reports generated as a result of this POPT will be managed according to the POPT QA Plan. The following reports will be generated as a result of this POPT:

- Weekly Telephone Call Reports
- Weekly Written Reports
- Final Report

12.1 Weekly Teleconferences

To ensure a regular dialogue and cohesive project interaction, telephone conferences will be conducted weekly, between FDF project personnel and POPT technical and management personnel, on Tuesdays at 09:00 Eastern time. Telephone conferences will discuss testing status, project progress, and upcoming testing activities. Additional telephone conferences will be conducted on an as-needed basis with at least 24-hour advanced notification, if possible. Documentation of telephone conferences, in the form of Envitco Telephone Call Reports, will detail subjects discussed and be included as an attachment to the Final Report.

12.2 Weekly Written Reports

Project-interaction documentation shall include one (1) to three (3) page weekly reports. The weekly reports shall be submitted to the FDF through the ECDC (Attn: Melissa Crews) via fax at (513) 648-3468 each Monday by 16:00 Eastern time. In addition to the fax transmittal, the original weekly report will be mailed to:

Fluor Daniel Femald
P.O. Box 538704
Cincinnati, OH 45253-8704
Attn: ECDC, MS 52-7

Information documented in the weekly reports shall include, as a minimum:

- Progress in respect to the Work Plan and schedule;
- Activities attempted during the period;
- Results of all attempts, including failures (root causes);
- Issues;
- Conclusions;
- Synopsis of weekly telephone conference; and
- Plans for the next two (2) weeks
12.3 Final Report

At the conclusion of the POPT, a draft final report will be submitted for FDF review and concurrence. Upon receipt of FDF's review comments, the draft final report will be revised in accordance with the project schedule and issued as final.

The structure of the final report will follow the final report outline provided in Appendix E of the contract as follows:

1.0 Executive Summary
2.0 Proof of Principle Test Description
3.0 Test Process Design and Procedures
4.0 Sampling and Analysis
5.0 Results and Discussion
6.0 Design Data
7.0 Conclusions

Information specific to the POPT, such as testing documentation (operating logs), analytical data packages, teleconference and weekly reports, and samples will be included with the draft Final Report as attachments.
13.0 SCHEDULE

Schedule 13.0 – 1 – Joule-heated Vitrification with Molybdenum Electrode (Rev. 0) presents the estimated POPT schedule based on a June 4, 1998 award date. Schedule 13.0 –1 provides a schedule that meets the forty-two (42) week POPT draft final report deliverable and completes the POPT within the forty-eight (48) week period of performance.

13.1 Milestones

Table 13.1-1 – Joule-heated Vitrification POPT Key Milestones

<table>
<thead>
<tr>
<th>Activity</th>
<th>Activity Duration (weeks)</th>
<th>Project Duration (weeks)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Award Contract</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Prepare Work Plan and QA/QC Plan</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>FDF Review and Comment on Work Plan and QA/QC Plan</td>
<td>4</td>
<td>7</td>
</tr>
<tr>
<td>Address FDF Comments on Work Plan and QA/QC Plan</td>
<td>2</td>
<td>9</td>
</tr>
<tr>
<td>FDF Review and Comment on Re-submitted Work Plan and QA/QC Plan</td>
<td>2</td>
<td>11</td>
</tr>
<tr>
<td>Address Final Comments</td>
<td>1</td>
<td>12</td>
</tr>
<tr>
<td>FDF Reviews and Approves Work Plan and QA/QC Plan</td>
<td>2</td>
<td>14</td>
</tr>
<tr>
<td>Perform Proof of Principle Testing</td>
<td>24</td>
<td>38</td>
</tr>
<tr>
<td>Prepare Draft Final Report w/Testing Documentation and Analytical Data Packages</td>
<td>9*</td>
<td>41</td>
</tr>
<tr>
<td>FDF Review of Draft Final Report</td>
<td>4</td>
<td>45</td>
</tr>
<tr>
<td>Address FDF Comments on Draft Final Report</td>
<td>2</td>
<td>47</td>
</tr>
<tr>
<td>Submit Final Report w/Archived Samples</td>
<td>0</td>
<td>47</td>
</tr>
<tr>
<td>Perform Presentation of Final Report</td>
<td>1</td>
<td>48</td>
</tr>
</tbody>
</table>

* POPT delineation between lab-scale and demonstration allows early beginning for report preparation, such that only three (3) weeks are required to update the Draft Final Report with demonstration results.
<table>
<thead>
<tr>
<th>ID</th>
<th>Task Name</th>
<th>Start</th>
<th>Finish</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Silos 1 &amp; 2 Proof-of-Principle Test Program</td>
<td>Thu 6/4/98</td>
<td>Mon 8/3/99</td>
</tr>
<tr>
<td>2</td>
<td>Pre-performance Activities</td>
<td>Thu 6/4/98</td>
<td>Fri 9/11/98</td>
</tr>
<tr>
<td>71</td>
<td>FDF Comment on Work &amp; QA / QC Plans</td>
<td>Thu 6/25/98</td>
<td>Thu 7/23/98</td>
</tr>
<tr>
<td>72</td>
<td>Address FDF Comments on Work &amp; QA / QC Plans</td>
<td>Fri 7/24/98</td>
<td>Thu 8/6/98</td>
</tr>
<tr>
<td>73</td>
<td>FDF Review &amp; Comment on Re-submitted Work &amp; QA / QC Plans</td>
<td>Fri 8/7/98</td>
<td>Thu 8/20/98</td>
</tr>
<tr>
<td>74</td>
<td>Address Final FDF Comments on Work &amp; QA / QC Plans</td>
<td>Fri 8/21/98</td>
<td>Thu 8/27/98</td>
</tr>
<tr>
<td>76</td>
<td>FDF Reviews and Approves Work &amp; QA / QC Plans</td>
<td>Fri 8/28/98</td>
<td>Fri 9/11/98</td>
</tr>
<tr>
<td>78</td>
<td>Surrogate Material Acquisition</td>
<td>Mon 6/29/98</td>
<td>Mon 8/31/98</td>
</tr>
<tr>
<td>82</td>
<td>FDF Reviews and Approves Raw Material Quality</td>
<td>Tue 9/1/98</td>
<td>Tue 9/8/98</td>
</tr>
<tr>
<td>83</td>
<td>Performance Activities</td>
<td>Mon 9/14/98</td>
<td>Fri 2/19/99</td>
</tr>
<tr>
<td>84</td>
<td>Surrogate Preparation / Surrogate Mix Validation</td>
<td>Mon 9/14/98</td>
<td>Fri 10/2/98</td>
</tr>
<tr>
<td>87</td>
<td>Lab Scale Development - Treatment Recipe</td>
<td>Mon 9/14/98</td>
<td>Fri 2/19/99</td>
</tr>
<tr>
<td>107</td>
<td>Demonstration of Process</td>
<td>Mon 11/2/98</td>
<td>Mon 2/8/99</td>
</tr>
<tr>
<td>138</td>
<td>Design Data for Full-scale Remediation Facility</td>
<td>Mon 10/19/98</td>
<td>Fri 3/12/99</td>
</tr>
</tbody>
</table>
13.2 Duration


13.3 Hold Points

In addition to the hold points identified as part of the Milestones (Section 13.1), the following hold points are also required:

<table>
<thead>
<tr>
<th>Hold Point</th>
<th>Requirement</th>
<th>Schedule Relationship</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surrogate Raw Material</td>
<td>Submit Compound Assays and Sieve Tests for FDF Approval</td>
<td>FDF Approval</td>
</tr>
<tr>
<td></td>
<td></td>
<td>09/01/98 through 09/08/98</td>
</tr>
<tr>
<td>Surrogate Mix Validation</td>
<td>FDF Collects 1 kg samples for Approval of Surrogate Mix</td>
<td>Surrogate Mix Validation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>09/21/98 through 10/02/98</td>
</tr>
<tr>
<td>Surrogate Slurry Hold Time</td>
<td>Minimum of 24-hours to Allow Complete Hydration of the Surrogate Slurry</td>
<td>During Surrogate Slurry Prep</td>
</tr>
<tr>
<td></td>
<td></td>
<td>11/23/98 through 12/08/98</td>
</tr>
</tbody>
</table>

13.4 Witnessing Visits

FDF may witness performance of the POPT at any point in the development and testing effort provided the visit does not hinder safe operations. To ensure safe operations and provide time for scheduling and training, FDF shall notify Envitco of witnessing visits at least 24-hours in advance.
14.0 MANAGEMENT AND STAFFING

14.1 Project Management

The programmatic management of the POPT project will be performed by Envitco. Envitco will assign project personnel to manage and interface with its subcontractors. Point of contact relationships between Envitco and FDF, as well as between Envitco and its subcontractors, are depicted in the POPT QA. The following personnel are defined as the key Envitco POPT management team:

- Project Manager - David M. Bennert
- Principle Investigator - Robert A. Wilson
- QA Management Representative - Douglas H. Davis

This team will interface through the Project Manager with Ms. Mary Morse, FDF Contract Technical Representative, regarding all technical matters. As defined in Envitco’s Quality Management System, the QA Management Representative will have the authority to directly contact FDF should the need arise. However, when possible, all QA correspondence will be initiated through the Project Manager.

Contractual matters will be handled by Mr. Irving M. Williams, Envitco Sales Manager through Mr. William Hensley, FDF Contract Administrator.

14.2 Staffing

Staffing for the recipe development and POPT demonstration run will be provided by Envitco and CETL. Envitco engineers will be on-site during the recipe development and the demonstration run. CETL will provide sufficient technicians and administrative staff necessary to conduct the testing.

Envitco will manage its key subcontractors who will perform the preliminary design of the full-scale facility equipment. Toledo Engineering, SGN, and Cogema Engineering will provide the staff required to generate the design data and layouts required for the full-scale facility.

14.3 Training

Training will be conducted in accordance with the POPT Training Matrix included in the POPT QA Plan.
15.0 REGULATORY COMPLIANCE

The Clemson Environmental Technologies Laboratory is fully authorized by federal and state regulatory agencies to handle hazardous, radioactive and mixed waste materials.

15.1 Licenses

Although the POPT will not involve radioactive materials, CETL is licensed to handle radioactive and mixed-waste materials.

Clemson Environmental Technologies Laboratory
Radioactive Material License

<table>
<thead>
<tr>
<th>Material</th>
<th>License Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic Numbers 2 - 92</td>
<td>2 Curies</td>
</tr>
<tr>
<td>Atomic Numbers &gt;92</td>
<td>100 millicuries</td>
</tr>
<tr>
<td>Tritium</td>
<td>10 Curies</td>
</tr>
<tr>
<td>Source Material</td>
<td>1000 kilograms</td>
</tr>
<tr>
<td>Special Nuclear Material</td>
<td>350 grams U-235</td>
</tr>
<tr>
<td></td>
<td>200 grams U-233</td>
</tr>
<tr>
<td></td>
<td>200 grams Pu</td>
</tr>
</tbody>
</table>

CETL also operates under a Hazardous Waste Treatability Exemption in accordance with 40 CFR 261.4.

15.2 Permits

CETL is authorized to conduct treatability studies, such as the POPT, by the South Carolina Department of Health and Environmental Control.
16.0 REFERENCES


Envitco Quality Assurance Manual, Document No. 98702 QA DHD QAD 19980620 1345 1 Q.

Envitco POPT Quality Assurance Plan, Document No. 98703 QA DMB QAD 19980918 1100 O Q.


