

DELIVERABLE REPORT



Thermal treatment for radioactive waste minimisation and hazard reduction

Initiative: EU Horizon 2020 - Euratom Research and training programme

Grant Agreement No: 755480

Start date: 01.06.2017 Duration: 36 Months

Project Coordinator: VTT Technical Research Centre of Finland Ltd

WP No: 3

Deliverable No: D3.5

Title: Test Report on Demonstration of Geomelt Technology for THERAMIN Project

NNL document ref: EU10302/06/10/03

Lead beneficiary: NNL

Contributors: NNL, ARE, CEA, USFD, VTT, VUJ

Dissemination level: Non-Confidential

Due date of deliverable: 31/01/19

Actual submission date: 25/01/19





History Chart			
Type of revision	Document name	Partner	Date




	Name	Signature	Date
Author	J I Roe, K M Burns		23-1-19
Checked By	C Scales		23-1-19.
Approved By	S Clarke		23/01/19



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Acknowledgement

This project has received funding from the European Union's Horizon 2020 Euratom research and innovation programme under Grant Agreement No 755480.



THERAMIN Project Partners

Andra	Agence nationale pour la gestion des déchets radioactifs – France
ORANO	ORANO – France
CEA	Commissariat à l'énergie atomique et aux énergies alternatives – France
GSL	Galson Sciences Limited – UK
FZJ	Forschungszentrum Juelich GmbH – Germany
LEI	Lithuanian Energy Institute – Lithuania
NNL	National Nuclear Laboratory – UK
ONDRAF/NIRAS	Organisme National des Déchets RAdioactifs et des matières Fissiles enrichies – Belgium
SCK•CEN	The Belgian Nuclear Research Centre – Belgium
USFD	University of Sheffield – UK
VTT	Teknologian Tutkimuskeskus VTT Oy (VTT Technical Research Centre of Finland Ltd)
VUJE	VUJE a.s. – Slovakia



THERAMIN End User Group

Andra	Agence nationale pour la gestion des déchets radioactifs – France
CEA	Commissariat à l'énergie atomique et aux énergies alternatives – France
EDF	Electricité de France – France
Fortum	Fortum Oyj – Finland
IGD-TP	Implementing Geological Disposal of Radioactive Waste Technology Platform
Nagra	Die Nationale Genossenschaft für die Lagerung Radioaktiver Abfälle – Switzerland
ONDRAF/NIRAS	Organisme National des Déchets RAdioactifs et des matières Fissiles enrichies – Belgium
RWM	Radioactive Waste Management Ltd – UK
Sellafield	Sellafield Ltd – UK
TVO	Teollisuuden Voima Oyj – Finland



List of acronyms

HLW	High Level Waste
ILW	Intermediate Level Waste
LLW	Low Level Waste
PCM	Plutonium Contaminated Material
NNL	National Nuclear Laboratory
THERAMIN	Thermal treatment for radioactive waste minimisation and hazard reduction
TRL	Technology Readiness Level
ICV	In Container Vitrification
TH01	Theramin Experiment 1
TH02	Theramin Experiment 2
SIXEP	Site Ion Exchange Plant
FGMSP	First Generation Magnox Storage Pond
CRB	Cast Refractory Box
DF	Decontamination Factors
SMF	Sintered Metal Filter
SL	Scrub Liquor
IR	Infrared Camera
CMgS	Corroded Magnesium Sludge



1. Introduction

This report describes two thermal treatment experiments carried out by NNL using the GeoMelt ICV system at Sellafield.

High Level Waste (HLW) and Intermediate Level Waste (ILW) in the UK have been successfully immobilised over the past few decades. HLW has been treated using vitrification and some operational ILW streams via immobilisation in a cement matrix. Low Level Waste (LLW) uses other processes e.g. compaction, grouting and subsequent disposal in the Low Level Waste Repository (LLWR).

Cementitious encapsulation for ILW was selected as a baseline treatment technology as it is simple, readily available and is a low cost material compatible with wet wastes. However, ILW destined for cementation does not undergo pre-treatment. In the absence of this stage organic plutonium contaminated material (PCM) and/or reactive metals retain chemical reactivity within the cementitious product; thus additional considerations are required for the storage and final disposal of the product.

Thermal treatment offers an alternative waste treatment process. The application of heat in a thermal process can pacify the reactivity of the waste metals and can provide significant volume reduction, this leads to a product that is potentially more stable and more cost effective for storage and disposal.

The UK National Nuclear Laboratory (NNL) is partaking in a European Union (EU) funded research programme; "Thermal treatment for radioactive waste minimisation and hazard reduction" (THERAMIN). The THERAMIN project has identified a range of waste streams across the EU that are potentially suitable for thermal treatment. There are a wide range of technologies which could be utilised to demonstrate the thermal treatment of such waste streams.

The THERAMIN; project is a European Commission (EC) programme of work jointly funded by the Horizon 2020 Euratom research and innovation programme and European nuclear waste management organisations (WMOs). Twelve European WMOs and research and consultancy institutions from seven European countries are participating in THERAMIN.

The overall objective of THERAMIN is to demonstrate the efficacy of thermal treatment in providing improved safe long-term storage and disposal of intermediate-level wastes (ILW) and low-level wastes (LLW). The work programme provides a vehicle for coordinated EU wide research and technology demonstration designed to provide improved understanding and optimisation of the application of thermal treatment in radioactive waste management programmes across Europe, which may eventually move technologies higher up the Technology Readiness Level (TRL) scale. The THERAMIN project is being carried out in five work packages (WPs). WP1 includes project management and coordination and is being led by VTT. WP2 evaluates the potential for thermal treatment of particular waste streams across Europe; this WP is led by Galson Sciences Limited (GSL). In WP3, the application of selected thermal treatment technologies to radioactive waste management is demonstrated and evaluated; this WP



is led by NNL. In WP4, the disposability of the thermally treated waste products is assessed; this WP is led by ANDRA. WP5 concerns synthesis of the project outcomes and their dissemination to other interested organisations.

NNL and Veolia Nuclear Solutions have formed a collaboration to establish an active GeoMelt In-Container Vitrification (ICV) system at Sellafield. This facility is currently used to demonstrate the treatment of a wide range of UK based waste streams. As part of the THERAMIN project, two appropriate waste streams have been selected for thermal treatment demonstration using the GeoMelt system.

The streams selected for demonstration using are:

- TH01- A cementitious stream representative of sea dump drums or failing cement wastes packages
- TH02- A sludge stream made up of a naturally occurring zeolite (clinoptilolite), sand, Magnox storage pond sludge and miscellaneous contaminants known to arise in a range of UK feed streams.

These two specific waste streams were selected for this experimental programme based on the output from THERAMIN WP2 as being of interest across the EU. Thermal treatment of SIXEP clinoptilolite and FGMSP waste types (similar to TH02) has previously been demonstrated as separate waste streams using the GeoMelt facility. There is an opportunity to demonstrate co-processing using clinoptilolite as the major glass former, thus improving the waste incorporation and efficiency of the process.

Two key fission products of interest in ILW are radioactive caesium and strontium. In order to carry out representative experiments; the cementitious stream (TH01) was dosed with 25MBq of Cs-137 and the sludge stream (TH02), 25MBq of Cs-137 and 16MBq of Sr-85, both sufficient to assess the decontamination factors (DFs) across the GeoMelt process. None active surrogates, europium and cerium oxides were used to represent the presence of actinides in the waste.

Samples of the vitrified product and materials captured in the off gas abatement system were taken to provide data to calculate the material balances within the process. This report specifically addresses the operation of the treatment process; chemical analysis of the product glass and off gas samples will be presented in a subsequent report.

2. Description of the Technology

2.1. System Description

The GeoMelt system installed at the NNL Central Laboratory is shown in Figure 1.

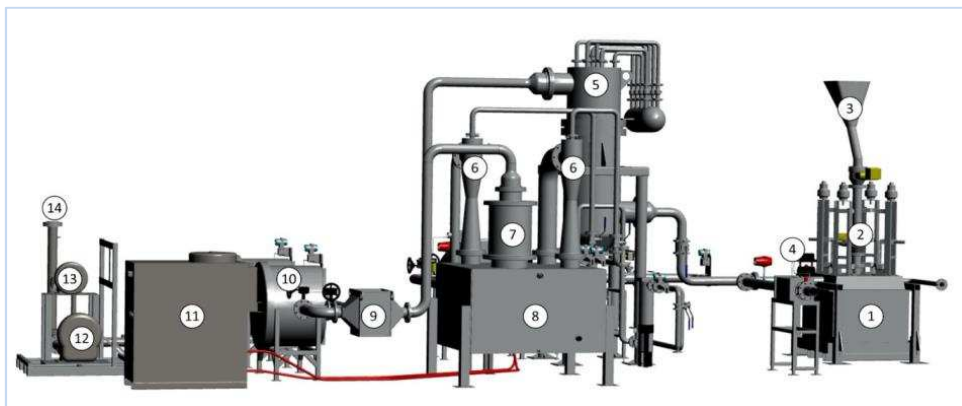


Figure 1: GeoMelt ICV System

The system is divided into 6 zones: 1) ICV Treatment; 2) Off-gas Pre-Treatment; 3) Cooling System; 4) Wet Scrubbing; 5) Off-gas Final Conditioning; and 6) Off-gas Discharge.

2.2. Zone 1: ICV Treatment

Zone 1 includes a 150 kW Scott-Tee transformer that provides power to the ICV melter. The Scott-Tee transformer converts alternating current (AC) 3-phase line power to two independent AC phases that provide the electrical power to the melt. The transformer supplies continuously variable voltage through two independently controlled outputs, each capable of 1500 amperes (A) maximum at 50 volts (V) and 75 kilowatts (kW) from 50 through 750V. Each transformer output is connected to a pair of graphite electrodes vertically embedded in the waste in a square array as shown in Figure 2. The transformer converts the incoming three phase line voltage to two single phases on each of two secondary windings. Each secondary winding has six voltage taps. GeoMelt ICV operates on joule heating. The joule-heating principle operates by internal resistance heating of conductive material as an electrical current pass through it. The electrical resistance decreases as the molten mass grows; therefore, to maintain a power level high enough to melt all of the contained waste, an increase in current to maintain or increase the power level for continued waste processing is required. The GeoMelt process is initiated at a relatively high resistance requiring a higher voltage potential and lower current. As the melt progresses and resistance decreases, lower voltage taps allow increased current to the melt. Each output phase of the Scott-T transformer used in the Central Laboratory has six tap secondary windings. Tap settings are selected automatically by the transformer's digital controller (auto-tap selection) based on the power level (set by the operator) and electrical conditions of the melt.

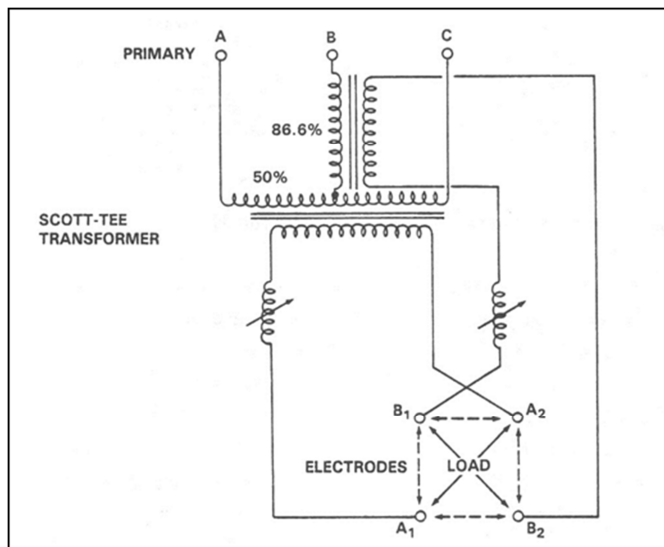


Figure 2: Scott-T- Transformer Diagram For GeoMelt Application

The melter consists of a steel container lined with refractory materials containing the melt. The refractory lining consists of a 200-L cast refractory box (CRB) surrounded by refractory silica sand. The CRB has four internal K-type thermocouples (T-101, T-102, T-103 and T-104) housed within refractory sheaths which penetrate 2 cm into the melt pool. These are mounted vertically on one face of the CRB; T-101 at the top and T-104 at the bottom. The melter hood has an integral feed system which incorporates a dual-isolation valve airlock to add materials to the melter during processing (Feeding While Melting FWM). The melter hood also contains and routes off gas emissions and surplus heat to the downstream off-gas treatment system. The ICV container and associated equipment (e.g., hood, infrared (IR) camera, vertical electrode feeders and electrodes, central feed chute, etc.) are shown in Figure 3.



Figure 3: ICV Container at Sellafield Central Laboratory



Off-gas flow is induced by a blower, maintaining negative pressure throughout the system. Process parameters are monitored at key points to provide hood vacuum control and aid operators in controlling power input to the melter. As melt conditions change, a pre-set vacuum on the ICV container is maintained by a fast acting feedback loop to the variable frequency drive (VFD) on the main off-gas blower. Real-time viewing of the hood interior is provided by an IR camera that records continuous video to a dedicated workstation. The IR camera allows operators to see into the melter by forming an image from infrared radiation rather than visible light. The IR camera is rated to the elevated temperatures experienced in the plenum; it can also see through fumes, gasses, and smoke. The IR camera is not used to monitor temperatures, although it does provide an approximate indication. The plenum temperature is monitored by a dedicated Type K thermocouple as described above.

2.3.Zone 2: Off-gas Pre-Treatment

Off-gas from the ICV melter is routed directly to a sintered metal filter (SMF) that is designed to capture 99.5% of 1 micron or larger particulate. The filter is designed to withstand elevated off-gas temperatures and provides long term use before replacement of the stainless-steel filter media is required. An integrated compressed air cleaning system removes particulates from the filters preventing them from loading up during operations. The particulate is discharged to a particulate bagging system and captured in a sealed plastic bag. The particulate can then be sampled and subsequently recycled into the melter or disposed separately.

2.4.Zone 3: Cooling System

A secondary liquid cooling system is used to cool scrub water from the wet scrubbing system (Zone 4). Water from the scrubbing system passes through the primary side of a plate heat exchanger and transfers heat across the plates to the secondary side where heat is dissipated by a dedicated chiller unit. Active cooling of the scrubber liquor lowers the dew point temperature of the off-gas. This lowers the mass fraction of water in the off-gas to deter condensation forming in the C3 ventilation system.

2.5.Zone 4: Wet Scrubbing

The off-gas from the SMF enters a wet scrubbing system consisting of two venturi scrubbers connected in series, followed by a mist eliminator. Water spray nozzles inside the venturi scrubber quench and cool the incoming off-gas. The spray pattern promotes mixing, condensation, and heat transfer between the scrub liquor and off-gas. The venturi scrubbers cool the off-gas, remove particulate, and remove acid gasses that may have passed through the first stage filtration. Scrub liquor is recirculated by a centrifugal pump through an 1800 L capacity holding tank, and is maintained at optimal pH by automatic addition of sodium hydroxide solution. The off-gasses exiting the second venturi scrubber are then conducted through a mist eliminator that removes entrained water droplets before entering the final conditioning stage.



2.6. Zone 5: Off-gas Final Conditioning

After wet scrubbing, the off-gasses pass through an inline electrical heater to raise its temperature, typically by at least 10 °C. This is to ensure that the off-gasses remain above the dew point temperature and eliminate condensation downstream. After heating, the off-gas enters a HEPA filter to remove remaining particulate, and then enters the primary off-gas blower for discharge to the off-gas stack. A backup HEPA filter and backup off-gas blower are available if needed. The backup blower starts automatically by an independent safety circuit in the event of a loss of vacuum or flow in the system.

2.7. Zone 6: Off-gas Discharge

The off-gas stack is connected to the Central Laboratory C3 Ventilation system where additional HEPA filtration occurs. Emissions are then exhausted to the atmosphere.

2.8. Instrumentation

The entire treatment and off-gas system is instrumented to provide continual monitoring of key process parameters, including temperature, pressure, and flow. This information is fed to a supervisory control and data acquisition (SCADA) system that monitors and records data, and controls key system components. A secondary and independent Safety Integrity Level 2 (a formal requirement of the process safety case) rated system monitors temperature, pressure, and flow at key points. This system will shut off power to the GeoMelt equipment and/or start the backup blower if one of these parameters falls outside predetermined safety limits.

2.9. Melting Configuration

The GeoMelt process can be set-up to treat waste in several configurations. The selected treatment configuration is chosen based on the waste type. For both experiments reported here, a "Top-Down" configuration was selected, primarily due to the high metal content, the low moisture content and presence of volatile radionuclide and surrogate components. In this configuration melting begins near the surface of a batch of waste material in the ICV container and melts downward until the batch of material has been fully treated. As the melt pool propagated the level inside the melter falls, to maintain the joule heating, the electrodes need to be progressively lowered into the melt pool until they have reached their maximum travel. Typical electrode lowering rates are 1 to 1.5 inches/hour.



3. Waste Feeds

This section describes the materials which were treated in each experiment. This includes; the simulate waste, the glass forming materials, additional material feed whilst melting and the surrogate materials used to represent key elements or fission products of interest; paint tins filled with grout to represent the cement likely to be found in sea dump cans and, aluminium and PVC strips to represent metallic and organic materials which have been grouted.



3.1.Experiment TH-01

The materials used in Experiment TH-01 are shown in Table 1.

WASTE MATERIALS	Mass (Kg)
Mild steel tins	3.15
Cement	13.35
Aluminium	2.45
Plastic (PVC)	3.05
GLASS FORMERS	
"Fluxed soil" Quarry soil, Boric acid, Soda carb + VTR frit blend	176.6
Total waste	198.6
TRACERS	
Cs-137 (Active)	25 MBq
SrCO ₃ (Non-active)	(8.42 g)
CeO ₂ (Non-active)	(6.14 g)
Starter path	N/A
VTR FRIT (with starter path)	20
TOTAL FOR PRE STAGE	218.6
FEED WHILE MELTING	Mass (Kg)
VTR frit	50
"Fluxed soil"	10.65
TOTAL MATERIAL	279.25

Table 1:Materials Used in TH-01

The composition of the glass forming materials used to vitrify the waste is shown in Table 3. In the context of this experiment the quarry soil is considered as both a waste and a glass former. The quarry soil used in this test has been included to demonstrate the capability to treat radioactively contaminated soils arising from operations on nuclear



installations. The soil has a high silica content which enables it to be used as the main glass former see Table 2. The soil was “fluxed” with Sodium and Boron to reduce the working temperature of the glass component.

Oxide	Quarry Soil (wt%)
SiO ₂	82.08
Al ₂ O ₃	7.94
CaO	0.39
TiO ₂	0.37
Fe ₂ O ₃	2.99
MgO	0.76
K ₂ O	2.10
Na ₂ O	1.08
P ₂ O ₅	0.10
Mn ₃ O ₄	0.10
LOI	2.09
Total	100

Table 2: Composition of Quarry soil

The nominal waste loading for this experiment is:

Mass of waste	=	137.2kg
Total mass of material	=	279.25kg
Waste loading	=	<u>49%</u>

Glass Former Blend	Mass (Kg)
Dry and Screened Quarry Soil	115.2
Boric Acid	20.2
Sodium Carbonate	23.0
VRT Frit	18.2
TOTAL	176.6

Table 3: Composition of Glass forming materials



3.2. Experiment TH-02

As stated earlier in this report, experiment TH-02 was carried out in two phases, an initial melt which was terminated prematurely (see later) and a subsequent re-melt. The materials used in the first phase of this experiment are shown in Table 4. Corroded Magnesium sludge (CMgS) has been used as a simulant for corroded Magnox sludge.



WASTE MATERIALS	Mass (Kg)
CMgS Sludge (45 % v/v solids)	50.25
Clinoptilolite	83.8
Aluminium	1
Iron	1
PAG Oil	1
GLASS FORMERS	
Silica	8.4
Sodium Carbonate	16.7
Boric Acid	8.4
TOTAL WASTE	170.55
TRACERS	
Cs-137 (Active)	25 MBq
Sr-85 (Active)	16.1 MBq
EuO ₂ (Non-active)	5.8 g
CeO ₂ (added with feed via FWM system)	6.14g
STARTER PATH	
VTR FRIT (with starter path)	20 kg
TOTAL FOR PRE STAGE	190.55
FEED WHILE MELTING	MASS (KG)
VTR FRIT	12
TOTAL MATERIAL	202.55

Table 4: Materials used in TH-02

This experiment had to be restarted due a failure of the joule heating in which electrode contact with the melt was lost part way through the initial melt. The restart was



facilitated by laying a fresh starter path on the surface of the partially melted product. This consisted of an additional 35Kg of VTR frit which was used to reform the starter path; therefore the total material processed in TH02 remelt was 237.55Kg.

[Note: 55 kg for starter path cover frit includes 20 kg originally used to cover starter path for TH-02 2 and a further 35 kg used to lay the starter path for the re-melt].

The nominal waste loading for this experiment is:

Mass of waste = 170.55kg

Total mass of material = 237.55kg

Waste loading = 72%

3.3. Use of Radioisotopes

Two common fission products were identified as nuclides of interest. These are Cs-137 and Sr-90.

Sr-90 is a fission product that is present in ILW; however Sr-85 was used as a replacement in this work to facilitate subsequent analysis of the product and secondary wastes. Sr-85 can be readily measured using gamma spectroscopy, whereas Sr-90 would have required complex analysis to produce quantitative data. For this experimental work the Cs-137 and Sr-85 was added,

3.4. Experiment TH-01

The test material was staged as shown in Figure 4 and Figure 5. The metallic tins were loaded in 3 layers of 12 tins with fluxed quarry soil between the tins and between the individual layers. The Cs-137 tracer solution and non-active tracer powders were added to the middle layer of tins, each tracer was added to a different tin. Once the CRB was packed with the waste materials the ICV unit was deployed under the hood and electrodes lowered into the starter path. Electrical continuity checks showed that the resistivity of the electrode paths were below the operational limit of <50 Ohms.

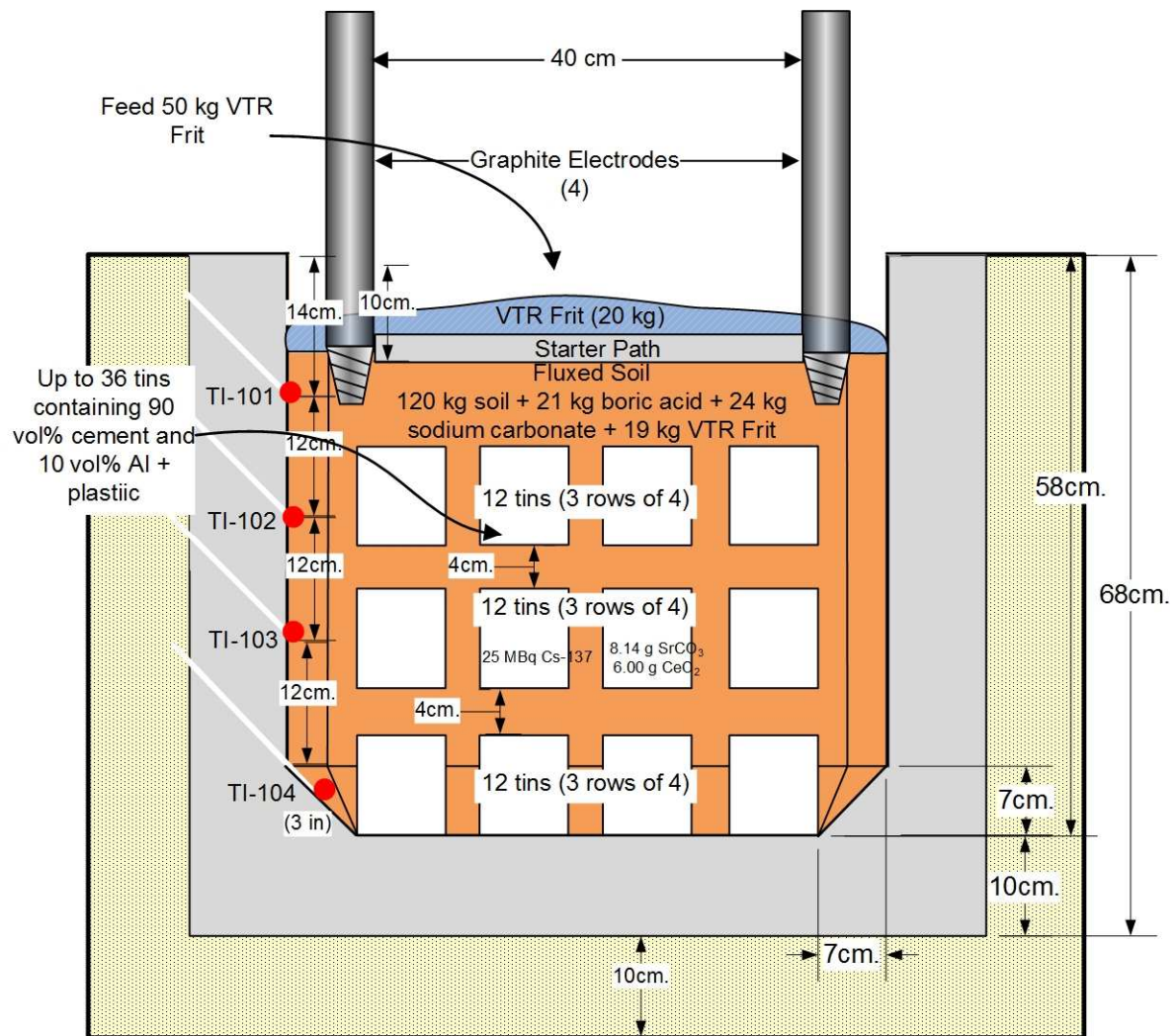




Figure 5: Photographs of TH-01 Staging Operations

The melt was carried out on 13-06-18; the power was initiated at 07:55 and the melt was completed at 22:55 giving a 15 hour melt. In total 279.25 kg of material was processed at a rate of 18.6 kg/hr.

At several points throughout the melt significant chemical activity was observed at the surface, this was consistent with the decomposition of grout and PVC and was observed in phases consistent with the melting of each successive layer of tins. At one point, tins were observed to be floating on the surface of the melt Figure 6. This could have potentially caused a problem of electrical shorting across the electrodes; however addition of cold feeds was used to disperse the paint tins which mitigated this risk. The SMF pressure differential (DP) peaked at ~15 inWG with the blower operation peaking at ~80% of its maximum output. Peak power was 34 kW/phase, this was slightly higher



than predicted but the higher power was required to reach the target temperature of 1350°C. The melter hood temperature was maintained below its operational control point high of 550 °C and the electrical parameters for both phases were well balanced throughout the melt. IR camera images of the melt are shown in Figure 6 and Figure 7. Operator observation, via the IR camera, indicates that adequate convection and movement suggesting good mixing within the melt pool.

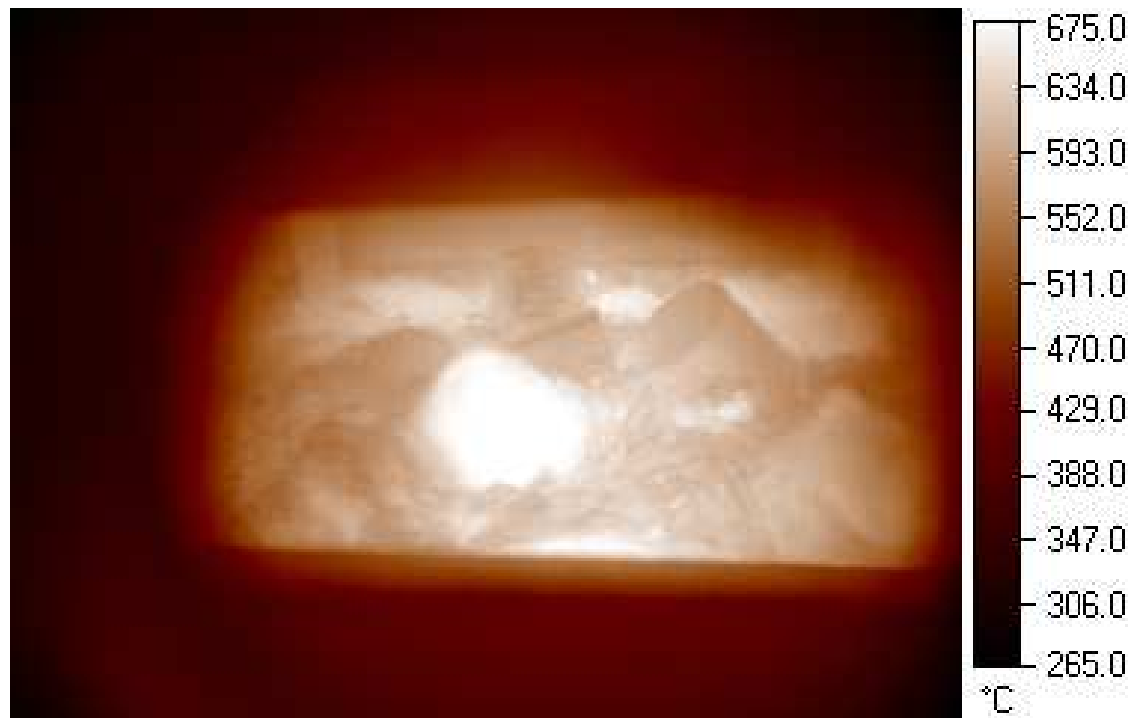


Figure 6: IR Camera Image During Melt TH-01 at ~18:45

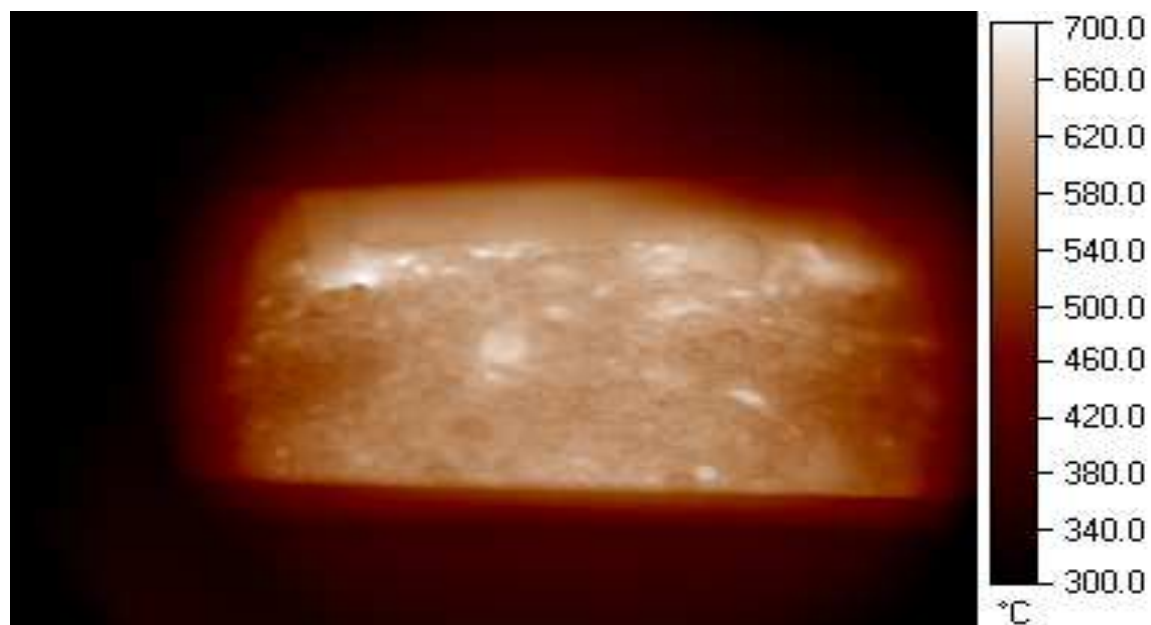


Figure 7:IR Camera Image During Melt TH-01, showing process completion



QR showing video of TH-01

Table 5 is extracted from the experimental log book and describes how the experiment was carried out.

Run Time (hrs)	Action	Observation
07:55 T=0.	Power ON Off-gas and support system started in readiness for melt Back pulsing of the SMF commenced at 1 pulse/sec.	SMF DP was 3.7 in WG
T=3	Ramp up continuing at 4kW per hour per phase, 12kW/phase reached	Upper thermocouple (TI_101) 202 °C and lower (TI_104) 19 °C.
T=4	Electrode lowering commenced at a rate of 1.0" /hr	
T=4 – T=6.25	Ramp up to 25kW continued	Internal thermocouples were (TI_101) 560 °C, (TI_102) 458 °C, (TI_103) 100 °C and (TI_104) 30 °C. The plenum temperature was 315 °C and the electrodes were by 2.5".
T=6.25	First feed (10.65Kg of fluxed quarry soil added	Carried out to maintain cold cap, as there was a molten surface on the melt and there was space available to accept the feed.



Run Time (hrs)	Action	Observation
T=7 – T=9	Power was ramped up from 25kW to 30kW per phase	Rate of 2kW/phase/hr
T=8	Rate of electrode descent was increased from 1.0"/hr to 1.5"/hr	
T=8.5		SMF DP peaked at ~15 in WG at t=8.5 hrs at this time the melter plenum temperature was (TI_118) 442 °C
T=9		Power was at 30 kW/phase and the internal thermocouples were (TI_101) 869 °C, (TI_102) 1091 °C, (TI_103) 1075 °C and (TI_104) 206 °C.
T=9- T=11.75	Frequency of batch feeding increased	Feeding rate was ~12 kg/hr
T= 11.25 – T=13.5	Power was steadily ramped up from 30 kW/phase to 34 kW/phase in 1 kW increments.	This increase was required to hit the target temperatures.
T=13.5		Electrodes fully deployed
T-13.5		Internal thermocouples were (TI_101) 967 °C, (TI_102) 1265 °C, (TI_103) 1257 °C and (TI_104) 1209 °C
T=13.5 – T=15		Power was maintained at 34 kW/phase and the temperature of the melt increased to hit the target temperatures. The final internal thermocouple readings were (TI_101) 981 °C, (TI_102) 1348 °C, (TI_103) 1367 °C and (TI_104) 1317 °C.
T= 15	Power off	Melt completed

Table 5: Experimental Log Book Extract for TH-01



The following charts present operational data from Experiment TH-01.

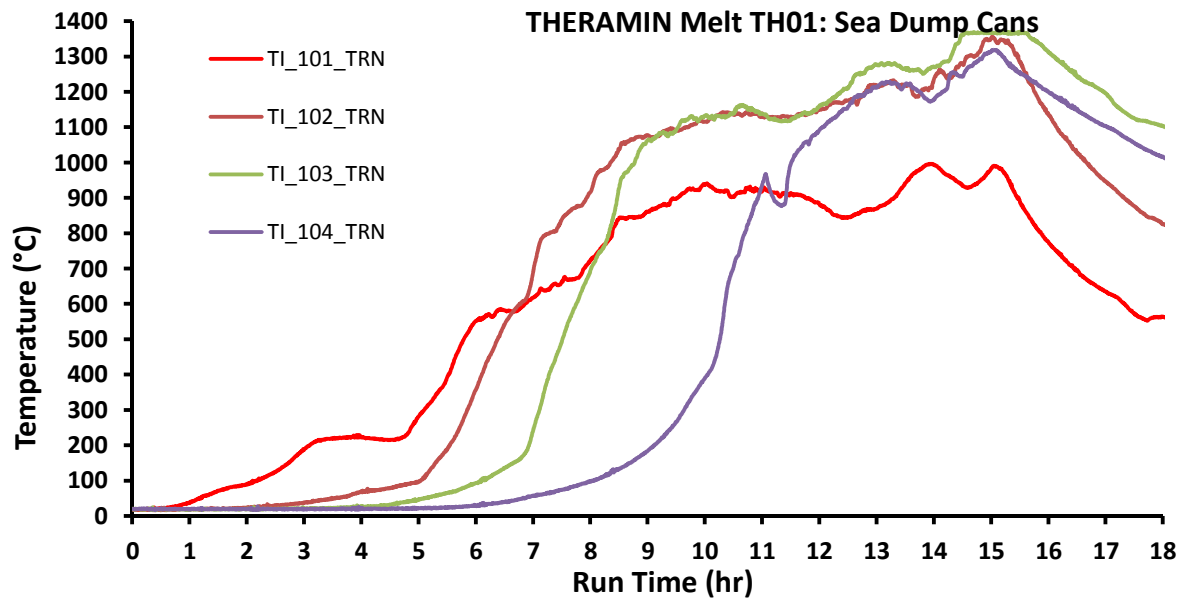


Figure 8: Internal Thermocouple Trend

Figure 8 indicates the internal temperature profiles measured during melt TH-01. Figure 4 indicates the positions of the 4 internal thermocouples. At T=15 hours three of the internal thermocouples reached the desired temperature of 1350°C, therefore the melt was deemed to be complete and the power was switched off. The uppermost thermocouple TI-101 was above the surface of the melt pool due to the volume reduction of the waste during the melt cycle. Since this thermocouple was not in the bulk glass it indicated a relatively low temperature i.e. 1000°C.

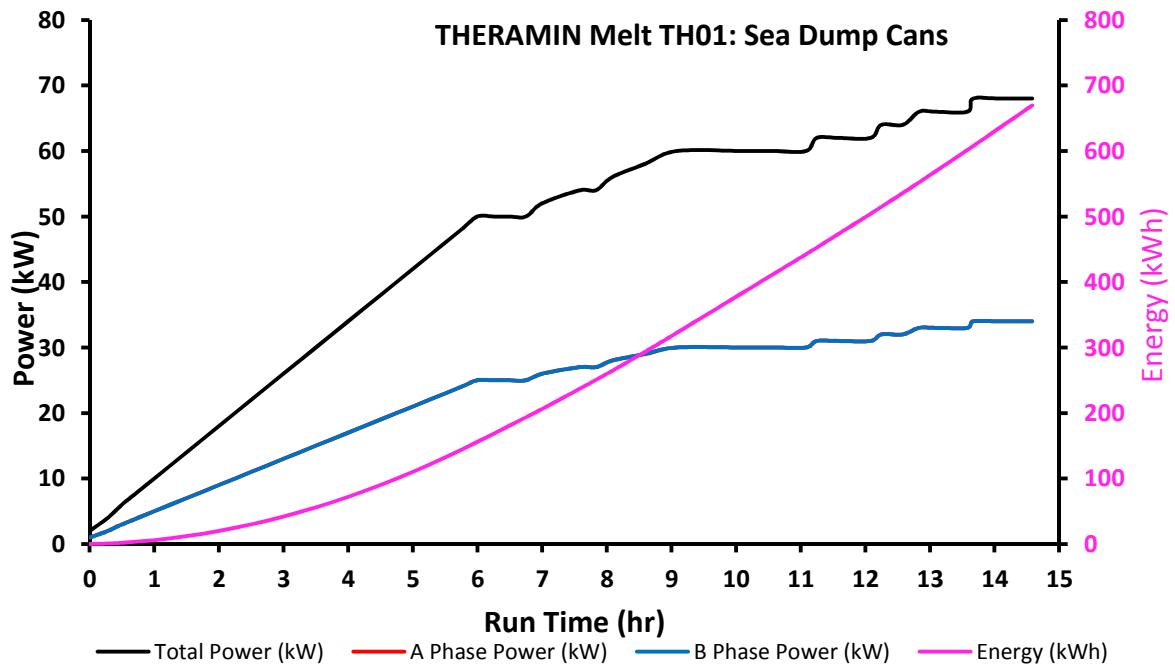


Figure 9: Applied Power

Figure 9 shows the steady power ramp up between hours T=0 and T=6, thereafter operators manually applied power to achieve the target temperatures shown in Figure 8. Application of power at the rate shown in Figure 9 is consistent with melts which require a target temperature of 1350°C. The power applied to each phase remained exactly the same throughout the experiment, thus the A phase and B phase power overlap in Figure 9.

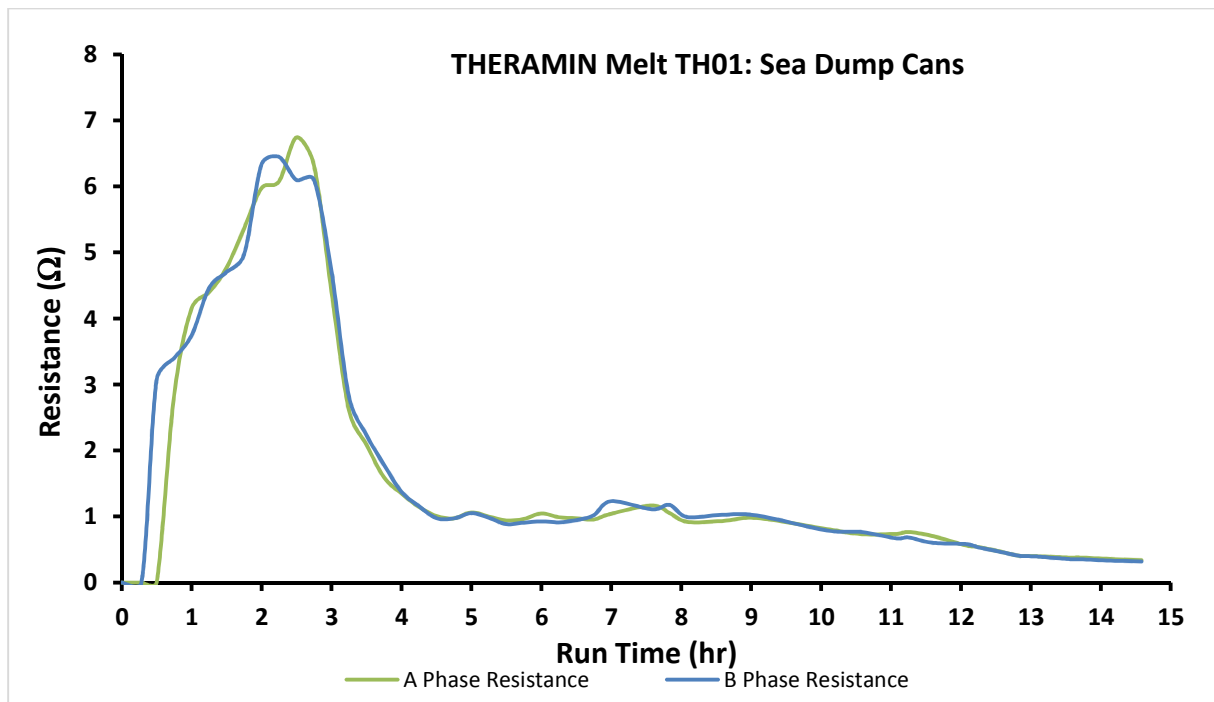


Figure 10: Melt Resistivity

The resistivity of the melt decreases during processing because as the melt pool grows the conductive capacity of the melt pool increases; this is normal behaviour for the majority of melts undertaken with this equipment.

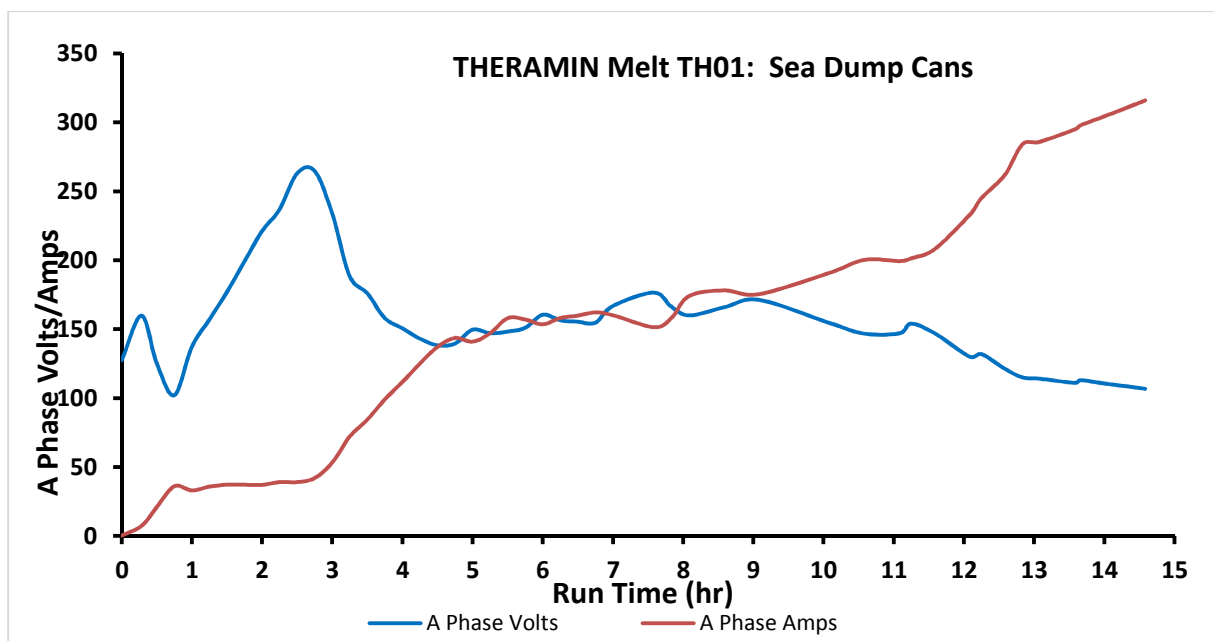


Figure 11: Phase A Electrical Parameters

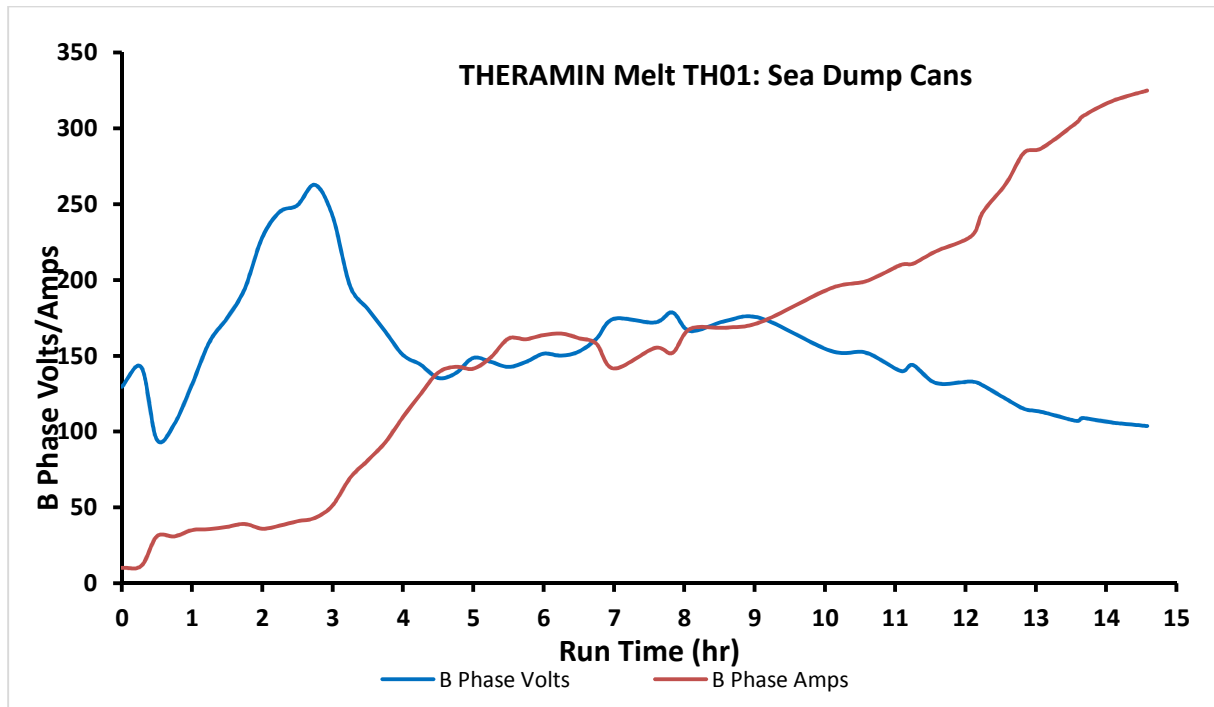


Figure 12: Phase B Electrical Parameters

Figure 11 and Figure 12 show electrical potential (volts) and current (amps) experienced by the melt over the course of the experiment. These plots are typical; as the melt pool volume increase so does the conductive capacity of the melt this is due to the increase of conductive material between the electrode pairs. The melt transformer is controlled to specific power set points so as current increases the voltage decreases (amps x volts = watts).

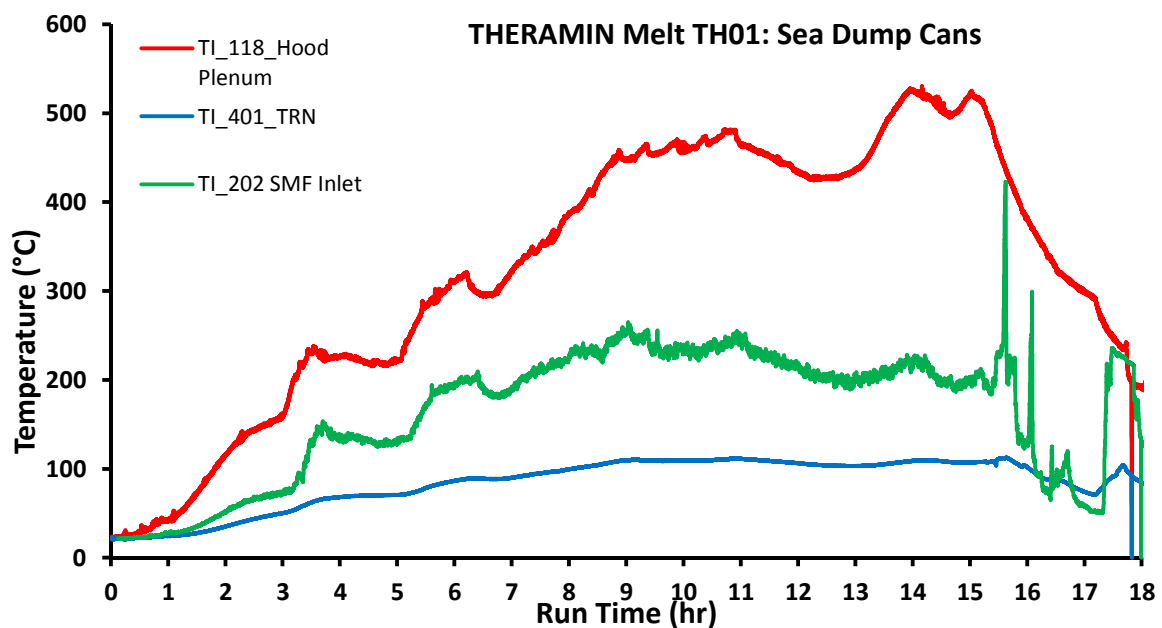


Figure 13: Melter Hood and Off-Gas Temperature



Thermocouple T1-118 indicates the hood plenum temperature throughout the melt, it can be seen that the temperature has been maintained below its operational control limit of 550°C, (Figure 13). This was managed by a combination of; controlling the power applied to the melter and the deployment of feed batches to produce a cold cap on the melt surface. The inlet temperature to the SMF (TI-202) was observed to be around 200°C, the temperature of the gases exiting the SMF (TI-401) was observed to be around 50°C this temperature drop is consistent with all melts carried out on the GeoMelt facility. (Figure 13). The temperature drop in the SMF is due to a cooling effect from the large void space and the cold compressed air used to back pulse the system.

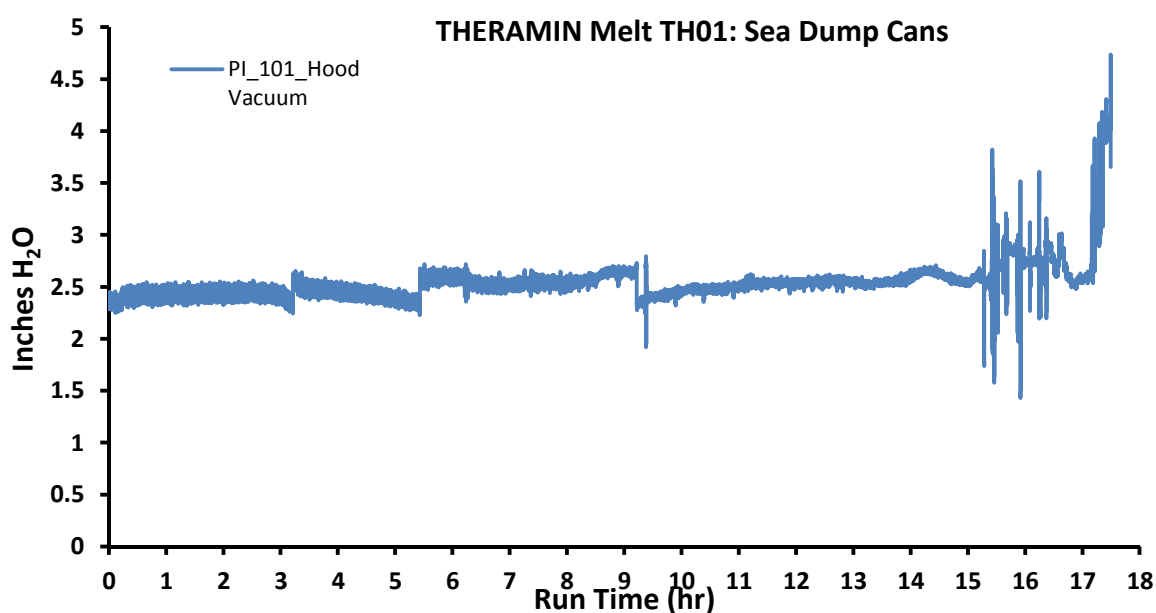


Figure 14: Melter Hood Vacuum

Throughout the melt the hood vacuum was maintained adequately at around -2.5 inWG as seen in Figure 14. The blower output required to maintain the hood vacuum was within its normal operating range of 60-80%. At T=15 hours, the off gas control was put into manual mode to assist with plant shutdown.

3.5 Experiment TH-02

The simulated waste materials were staged as shown in Figure 15 and Figure 16. A blend of sludge and clinoptilolite was loaded in layers with a mixture of steel, aluminium, PAG oil and the tracers. The active (Cs-137, Sr-85) and the EuO₂ tracers were added to

the middle of the refractory box, the CeO_2 tracer was blended into a feed batch for addition later on in the melt

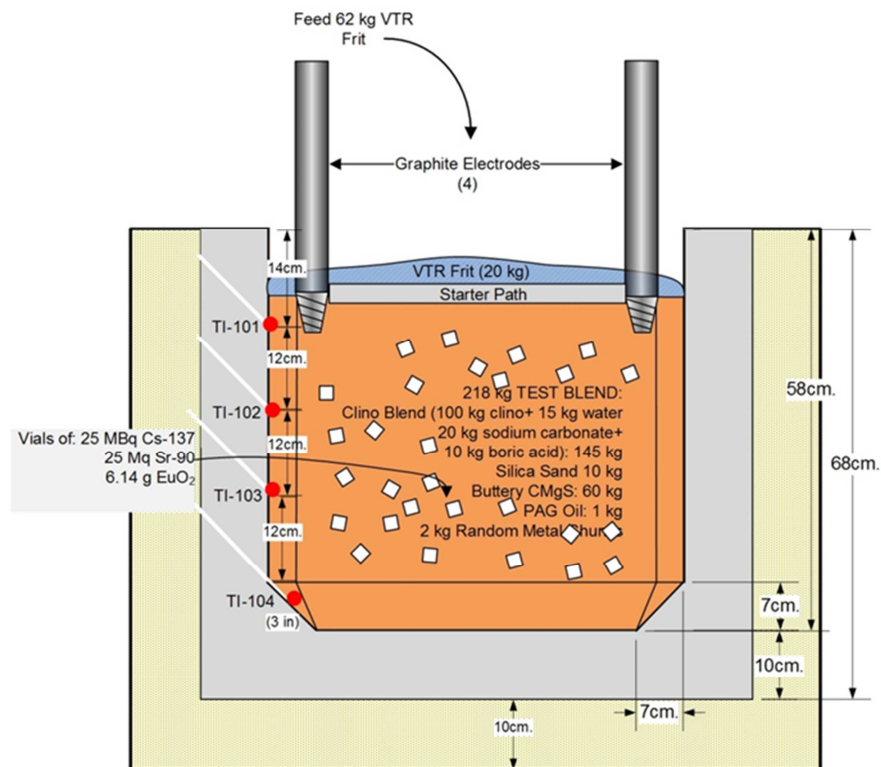


Figure 15: Packing Arrangements for Experiment TH-02



Figure 16: Photographs Showing Staging Waste Simulant into CRB for TH-02

Once the CRB was loaded, the ICV was deployed under the hood and electrodes lowered into the starter path, continuity checks showed that the resistivity of the electrode paths were all <50 Ohms.

The melt was carried out on 11-07-18; the power was initiated at 07:45 and the melt was terminated prematurely ~5 hours later due to a fault condition. The materials were incompletely reacted (see later).

This was an incomplete melt due to an imbalance in the transformer voltage experienced four hours into the melt. Operators lowered the electrodes in response to the imbalance but shortly after the transformer indicated an output fault for the A phase electrodes due to the voltage being too high. Operators switched off this phase and ran on B phase alone. At the time, the blower output changed, and it could be seen from the plant instrumentation that the blower was behaving erratically; this lasted for a couple of minutes before settling down. Shortly after the A phase fault, the B phase output also went into fault, again the voltage was too high. In an attempt to salvage the melt the electrodes were lowered, however both phases remained in fault and the melt was terminated. Due to the premature termination of this melt no additional feed batches were made. It was believed that the observed issue with the blower control was coincidental to the issue with the transformer voltage.



Observation of the IR camera footage post melt clearly showed that both electrode pairs were not deployed into the melt surface. This in effect created a break in the electrical continuity. The transformer automated response to the break in the circuit was to drive the voltage higher to deliver a current and therefore power, the rapid increase in the voltage created a transformer fault. The root cause of this problem was found to be traced back to the point at which the electrodes were lowered into the waste during melt preparations. The waste material presented a relatively hard surface which limited the extent to which the electrodes were immersed within the solid waste. Normally the electrodes penetrate the waste to a depth to 5 to 7 cm; in this instance the penetration was much less. Operators did not take this into account when commencing the electrode lowering sequence. Because of the low initial penetration depth, electrode lowering should have commenced sooner to keep the electrodes immersed as the glass melted. This did not happen, resulting in electrodes not being immersed in the melt pool.

The following table (Table 6) is extracted from the experimental log book and describes how the experiment was carried out.

<u>Run Time (hrs)</u>	<u>Action</u>	<u>Observation</u>
07:45, T=0.	Power ON Off-gas and support system started in readiness for melt Back pulsing of the SMF commenced at 1 pulse/sec.	SMF DP was 3.0 inWG at melt start-up
T=3	Power ramped at 4kW/phase/hr reaching 12kW/phase.	Internal thermocouples were (TI_101) 130 °C, (TI_102) 52 °C, (TI_103) 24 °C and (TI_104) 22 °C.
T=4	Electrode lowering commenced with an initial lowering of 0.5".	
T=4 – T=4.5	B phase electrodes lowered 1"	An imbalance in the transformer voltage was noticed, this was corrected by lowering B phase electrodes to 1.0".
T=4.5	A phase switched off	The transformer showed output fault for A phase (voltage too high), kW was zeroed



<u>Run Time (hrs)</u>	<u>Action</u>	<u>Observation</u>
		but transformer remained in fault for A phase output. A phase switched off. At this point B phase was running at 18 kW. At this time the blower noise changed, it was seen on the SCADA that the blower was behaving erratically for a couple of minutes before settling down.
T=5	Decision to carry on with single phase	Peak temperature for the melt (TI_101) 220 °C, (TI_102) 107 °C, (TI_103) 65 °C and (TI_104) 227 °C.
	A phase electrodes lowered to 1.0"	Immediately after lowering A phase electrodes to 1.0" (A phase output was still off) B phase went into fault on the transformer (voltage too high). At this point all electrodes were 1.0" down.
	A phase electrodes raised back up to 0.5"	Both transformer outputs remained in fault.
T=5.25	Power OFF	No power to the melt
T=6	Attempts to lower electrodes.	Upon cooling, it was visible on the IR camera that the electrodes were not in the melt, thus an attempt was made to lower the electrodes however the surface was too hard and the electrodes could not penetrate through.
T=6	Experiment terminated	

Table 6: Experimental log book Excerpts for TH02

The melt cooled in situ between the 11th and the 16th June. The ICV and product were removed from plant on the 16th June. The partially melted product and off-gas were not sampled at this time. It was clear at this point that the electrodes were above the melt surface confirming the reason for the fault condition encountered 4.5 hours into processing. Once the cause of the premature termination of the experiment had been investigated and understood, the decision was taken to re-start the melt to complete the experiment. An assumption was made that due to the short processing time and the relatively low temperatures attained, little or no carry over of volatile or particulate

material from the melter would have occurred and therefore the original starting materials would be present in a largely unreacted form.

This was confirmed by visual observation of the product. Only the upper surface of the waste materials had melted to form a thin crust above the remaining unreacted material.



Figure 17: Partially Vitrified Product From Initial Melt TH-02.

The waste materials within the CRB were prepared for re-melting by compacting the uneven surface and adding additional glass frit and starter path, see Figure 18 and Figure 19.



Figure 18: Compacting the Melt Surface in Preparation for a New Starter Path



Figure 19: New Starter Path in Place.

The CRB was loaded into the ICV and the melter deployed under the hood. The electrodes were lowered into the starter path; continuity checks showed that the resistivity of the electrodes were all <50 Ohms. The electrodes were not all initially deployed 3" into starter path, as was the case in the original TH-02 melt. In this case the operators were aware of the need to commence lowering electrodes earlier in the melting cycle to avoid recurrence of the same fault condition.

The re-melt was carried out on 19-09-18; the power was initiated at 08:00 and the melt was completed at 23:00 giving a 15 hour melt. In total 237.55 kg of material was processed at a rate of 15.8 kg/hr.

The following table (Table 7) is extracted from the experimental log book and describes how the experiment was carried out.



Run Time (hrs)	Action	Observation
08:00 T=0.	Power ON Off-gas and support system started in readiness for melt Back pulsing of the SMF commenced at 1 pulse/sec.	SMF DP was 3.0 in WG
T=2	Attempt to lower electrodes	No movement
T=4		Power reached 17 kW/phase following ramp up at 4 kW/phase/hr, internal thermocouples were (TI_101) 823 °C, (TI_102) 204 °C, (TI_103) 56 °C and (TI_118) 198 °C. All electrodes lowered to initial starting point on 3".
T=5.5		Power ramp up reached 23 kW/phase. At this point the internal thermocouple temperatures were (TI_101) 783 °C, (TI_102) 415 °C, (TI_103) 112 °C and (TI_118) 305 °C. The SMF DP was 4.0 in WG.
T=6- T=9		Power was held at 25 kW/phase as delay with bringing the FWM system on line meant there had been no feeding. The plenum temperature was at ~500 °C at T=8.
T= 8	Feeding commenced	6kg of VTR frit was fed at T=8, a further 6 kg of frit was fed at T=9 at this point the plenum temperature had dropped to 450 °C. CeO ₂ tracer was fed with first batch of feed.
T=9		Power at 25 kW/phase, thermocouples (TI_101) 875 °C, (TI_102) 980 °C, (TI_103) 733 °C, (TI_104) 101 °C and (TI_118) 450 °C. The SMF DP was 6.0 inWG.
T=5 – T=10	Electrode lowering commenced from the	



Run Time (hrs)	Action	Observation
	initial starting depth of 3". Electrodes were lowered at 1.5"/hr and reached 8.5" at t=10	
T=9 -T=10		Power was ramped up from 25kW/phase to 28 kW/phase as TI-102 had levelled off and the plenum temperature was low enough to allow for increased power.
T=10		Power at 28 kW/phase, it was noticed that the red and yellow electrodes would not be able to travel the full 12" as the grippers had slipped down the electrodes at the beginning of the melt when the team attempted to lower the electrodes. Red electrode was released to raise the gripper and it dropped to 11".
T=10.25		Output A (Green/Blue) went into fault on the transformer. It was not immediately apparent as to why the output was in fault. The melt continued on Output B only (Red/Yellow). As there was only one phase operating the decision was taken to stop feeding. 12 kg was the total amount fed.
T=10.5 and T=14		Power on Output B was raised from 28 kW to 34 KW.
t=10.5 and t=12.5		Yellow electrode was lowered to 11" to match the red electrode. Both electrodes reached 11.5" at t=14.5 hours.
T=15		Internal thermocouples had reached the target temperatures with TI_101 and TI_102 no longer in the melt. (TI_103) 1260 °C, (TI_104) 1157 °C and (TI_118) 326 °C.



Run Time (hrs)	Action	Observation
T= 15	Power off	Melt completed

Table 7: Experimental log book Excerpts for TH02 re-Melt

During the initial phase of the melt it was noticed that the usual "X" pattern of the melting starter path was not visible on the melt surface as observed via the IR camera, only the box appeared. The starter path must not have made contact diagonally between the electrodes. The initial continuity looked good but the conductive path must have been made around the edges of the box not diagonally. This did not prevent the melt from starting and propagating.

At T=10.5 hours into the melt one of the joule heating phases went into fault. It was not immediately obvious at the time what had happened. The operational decision at this point was to continue processing using the remaining operational phase. Only post melt was the cause of the fault determined. On retrieval of the product it could be seen that the lowest segment of one of the electrodes had fallen away from the segment above it, breaking the electrical circuit. This was also clearly visible on play back of the IR camera footage, Figure 20. Due to the loss of one heating phase, no further feeding was undertaken, this was to minimise the challenge on the melter. The power on the remaining phase was increased to 34 kW to compensate for the absence of the second phase. The most probable cause of the detachment of the electrode segment is that that the segments were not screwed together robustly at the electrode installation phase of the melt cycle. If this was the case this would be deemed to be an operator error.

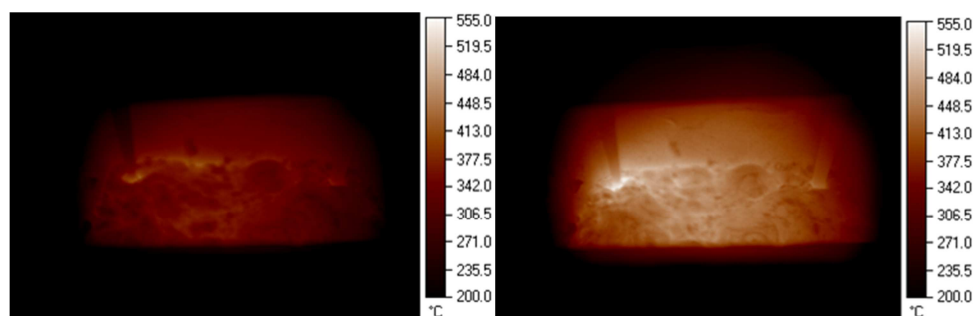


Figure 20: IR Camera Images Captured During the Melt Cycle.



QR Showing video of TH-02



The following charts present operational data from Experiment TH-02 Re-melt

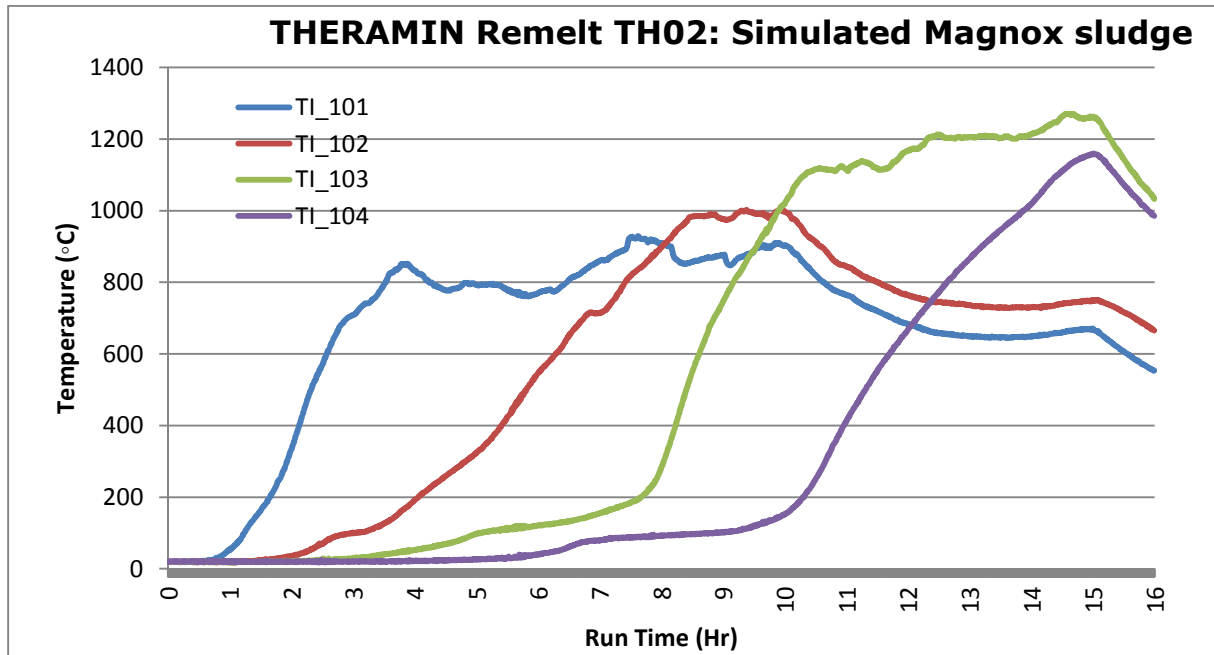


Figure 21: Internal Thermocouple Trend

At T=15 hours two of the internal thermocouples reached the desired temperature of 1150°C, therefore the melt was deemed to be complete and the power was switched off. The upper 2 thermocouples; TI-101 and TI-102 were both above the melt pool due to the volume reduction of the waste during the melt cycle and were reading the temperature in the ullage space above the melt pool.

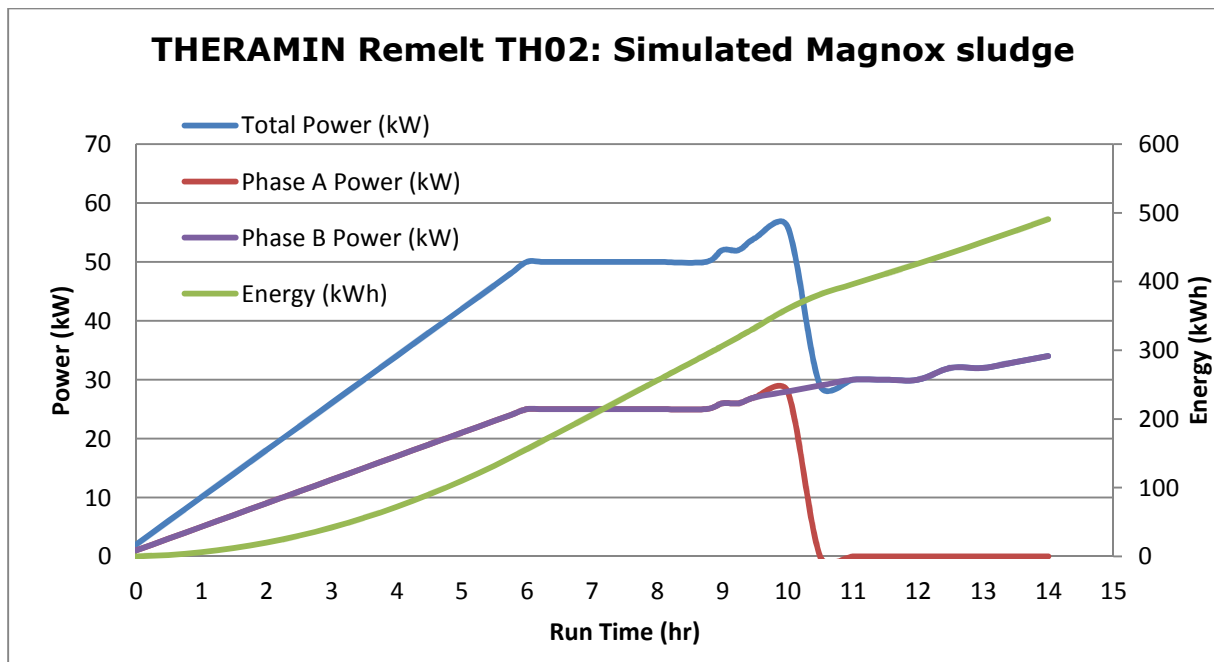


Figure 22: Applied Power

With reference to the applied power graph (Figure 22) the failure of the A phase at T=10.5 can be clearly seen; at this point a total power of 56Kw was being applied. Melting continued using the B phase alone. A maximum of 34 Kw total was applied via the B phase. Due to the relatively low target temperature required to process this waste material, this power level was sufficient to reach the required temperatures and therefore complete the melt cycle.

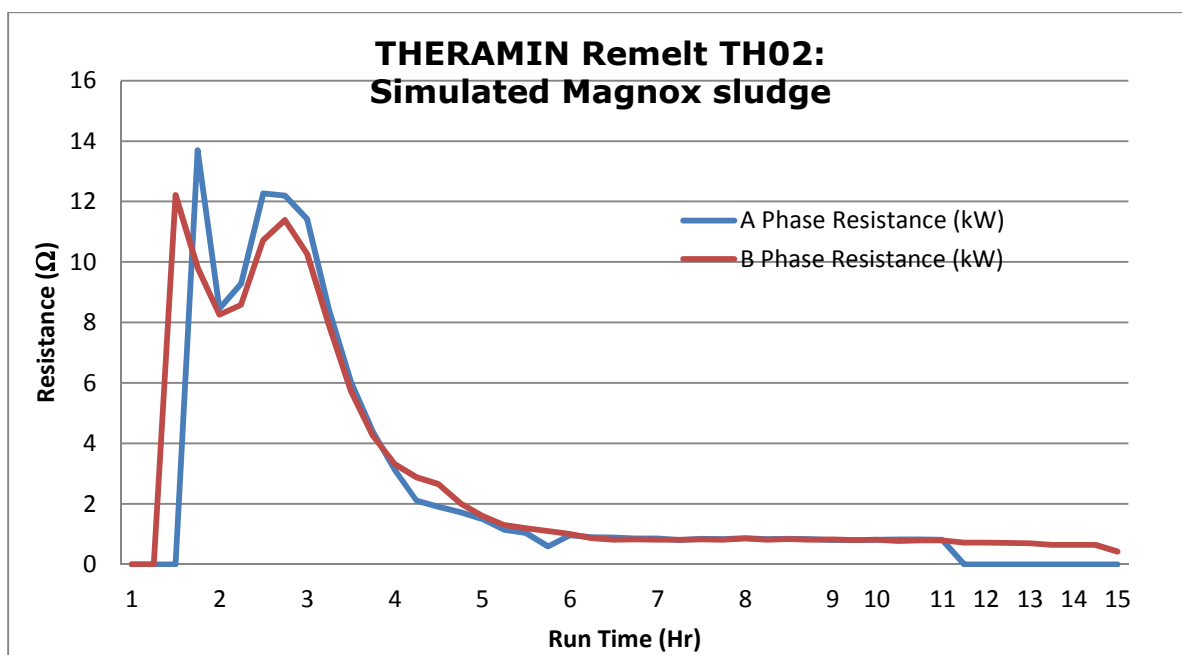


Figure 23: Melt Resistivity



As observed in TH01, the resistivity of the melt decreases during processing because as the melt pool grows the conductive capacity of the melt pool increases; this is normal behaviour for the majority of melts undertaken with this equipment.

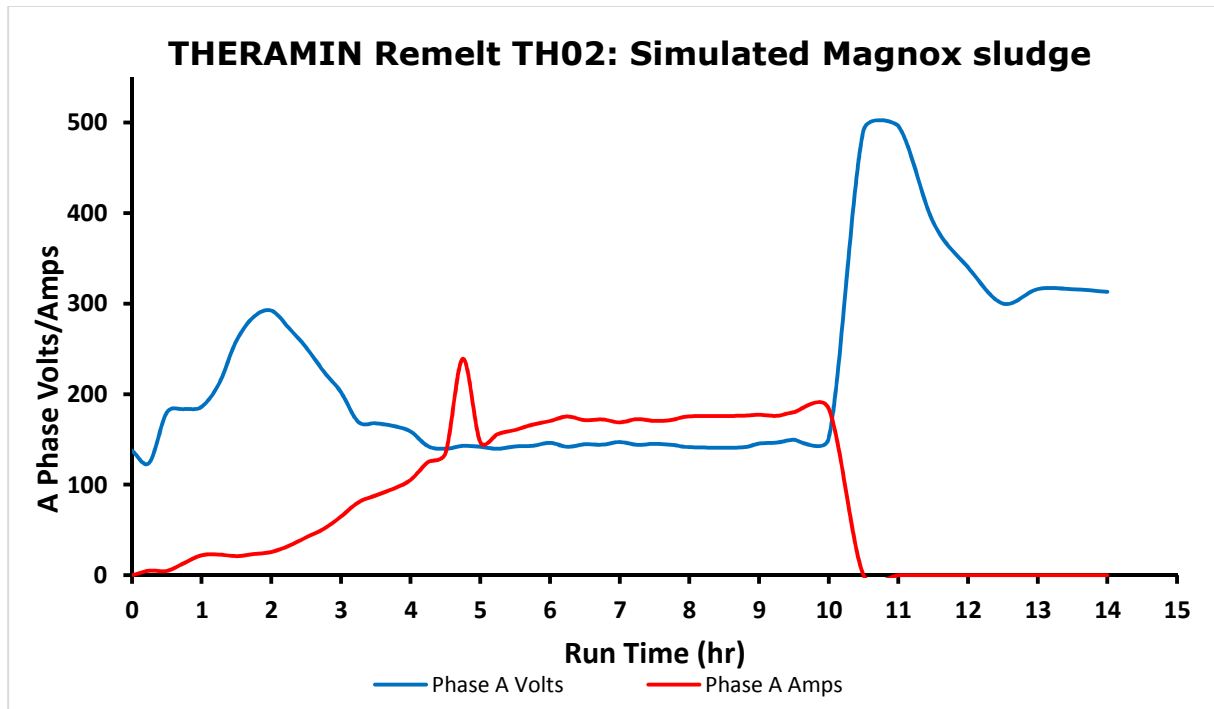


Figure 24: Phase A Electrical Parameters

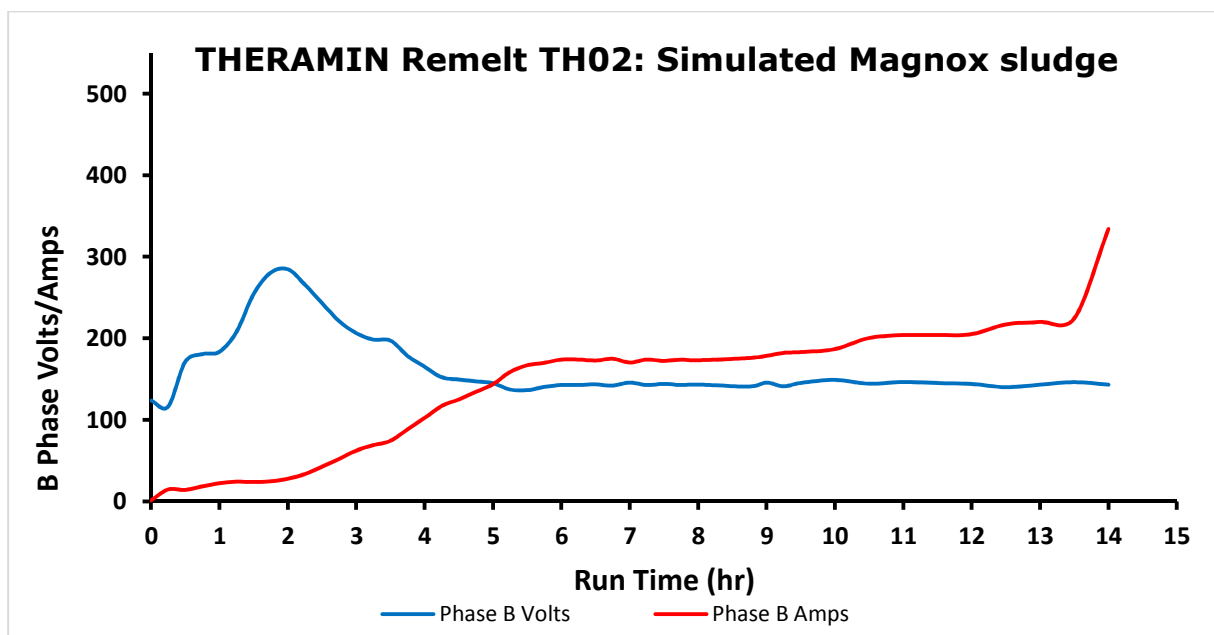


Figure 25: Phase B Electrical Parameters



Figure 24 and Figure 25 electrical potential (volts) and current (amps) experienced by the melt over the course of the experiment. As the melt grows so does the conductive capacity of the melt; this is due to the increase of conductive material between the electrode pairs. The melt transformer is controlled to specific power set points so as current increases the voltage decreases (amps x volts = watts). In the A phase graph it can be seen as the electrode becomes detached, thereby breaking the circuit, the current goes to zero and the voltage goes to its maximum as a consequence of the transformer controller response attempting to apply power.

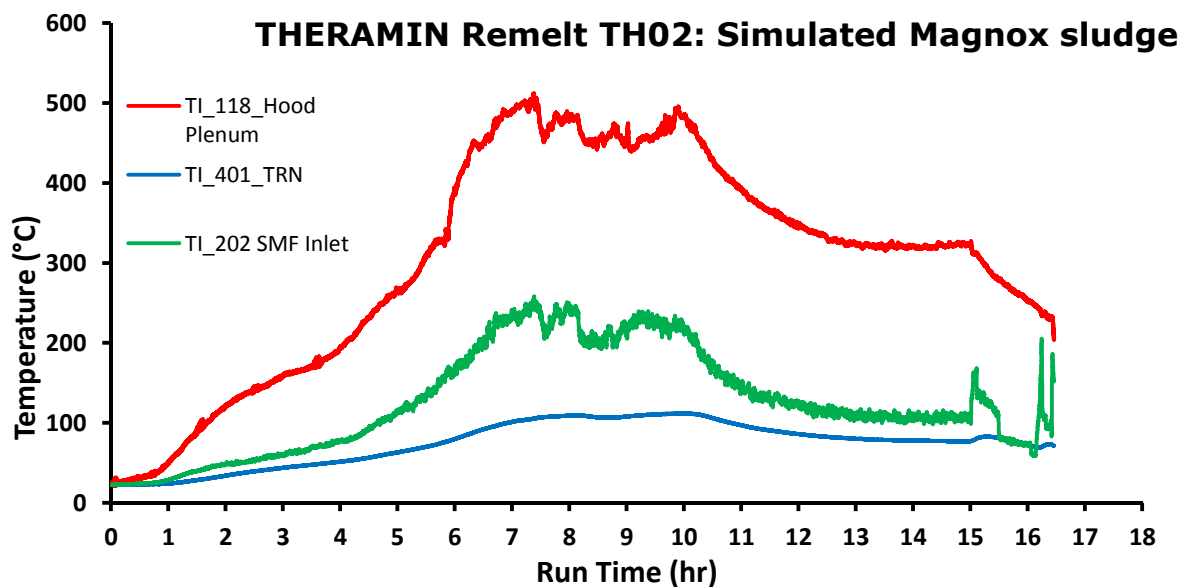


Figure 26: Melter Hood and Off-Gas temperature

T1-118 indicates the hood plenum temperature throughout the melt, it can be seen that the temperature has been maintained within its operational control limit of 550°C. This was achieved by managing the power applied to the melter, however it can be noted that the target temperature of 1150°C did not present a significant challenge on management of the off gas temperature.

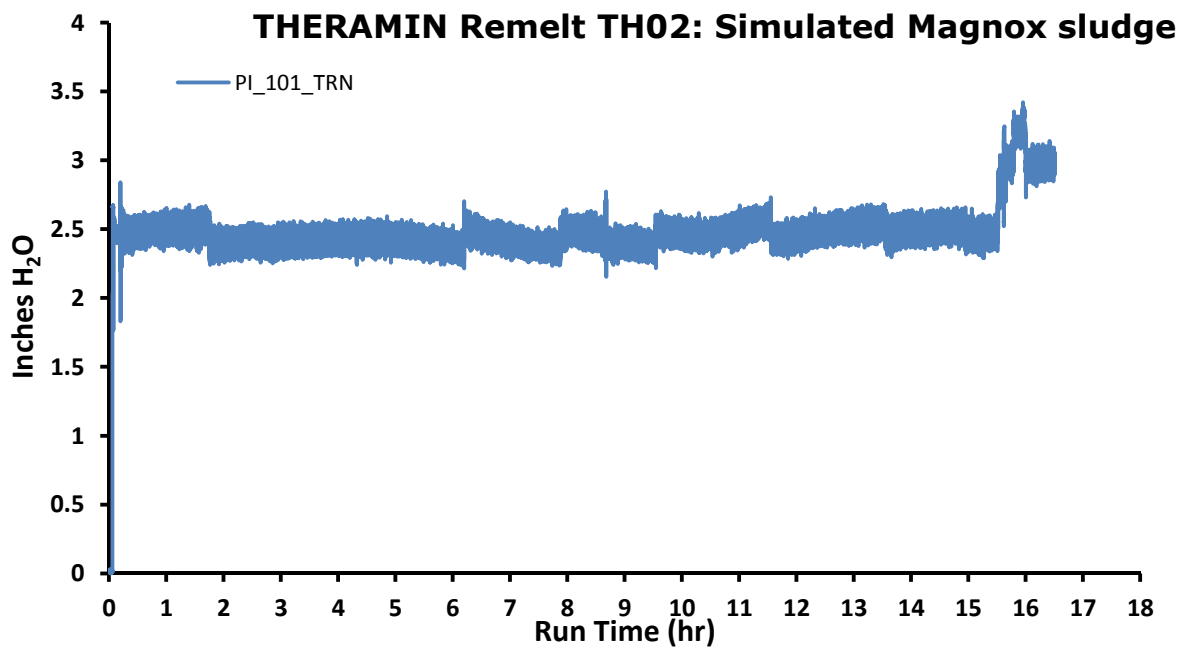


Figure 27: Melter Hood Vacuum

It can be seen that the Melter vacuum was adequately controlled to its set point throughout the melting cycle. There were no observed problems with operation or control of the off gas system and pressure control system.

4. Post-Trial Activities

4.1.TH-01

The melt was cooled in situ between the 14th and the 18th June, when sufficiently cool to handle the ICV and product was removed from plant. On removal of the product, the off-gas pipe connecting to the melter hood was inspected for any obstructions but none were found. On 18th June solids were removed from the SMF filter, in total 1.085 Kg of material was recovered. In addition a sample of the scrub tank liquor was taken. A 200 ml sample was taken from a bulk tank volume of 2000 litres. SMF solids and scrub tank liquor were submitted for radiochemical and chemical analysis.

On 20th June the product monolith was presented for sampling. The bulk product appeared to have a relatively flat glassy surface, looked broadly homogenous and visually appeared to be a satisfactory product. The Joule heating electrodes had thinned significantly; this is normal and is observed in virtually all melts carried out with this equipment. The mass of the glass monolith within the CRB was measured to be 236Kg.

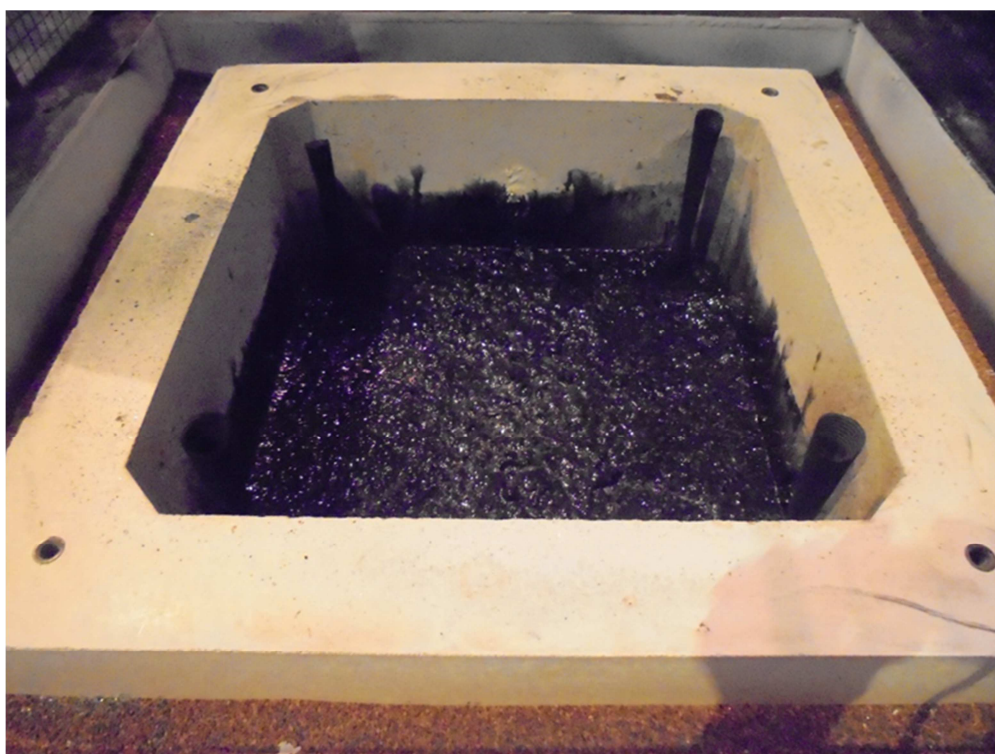


Figure 28: TH-01 Product.

Product Glass sampling is undertaken by core drilling into the monolith using proprietary core drilling equipment (see Figure 29). Product material was recovered from three positions within the monolith to provide samples for subsequent analysis and provide radiochemical and chemical data to facilitate material and activity balances for the process and to indicate the degree of homogeneity within the glass product. The sample



points are shown in Figure 30, Figure 31 and Figure 32. The mass of sample material removed is shown in Table 8.



Figure 29: Product Core drilling Equipment



Figure 30: Position of Sample Point 1



Figure 31: Position of Sample Point 2

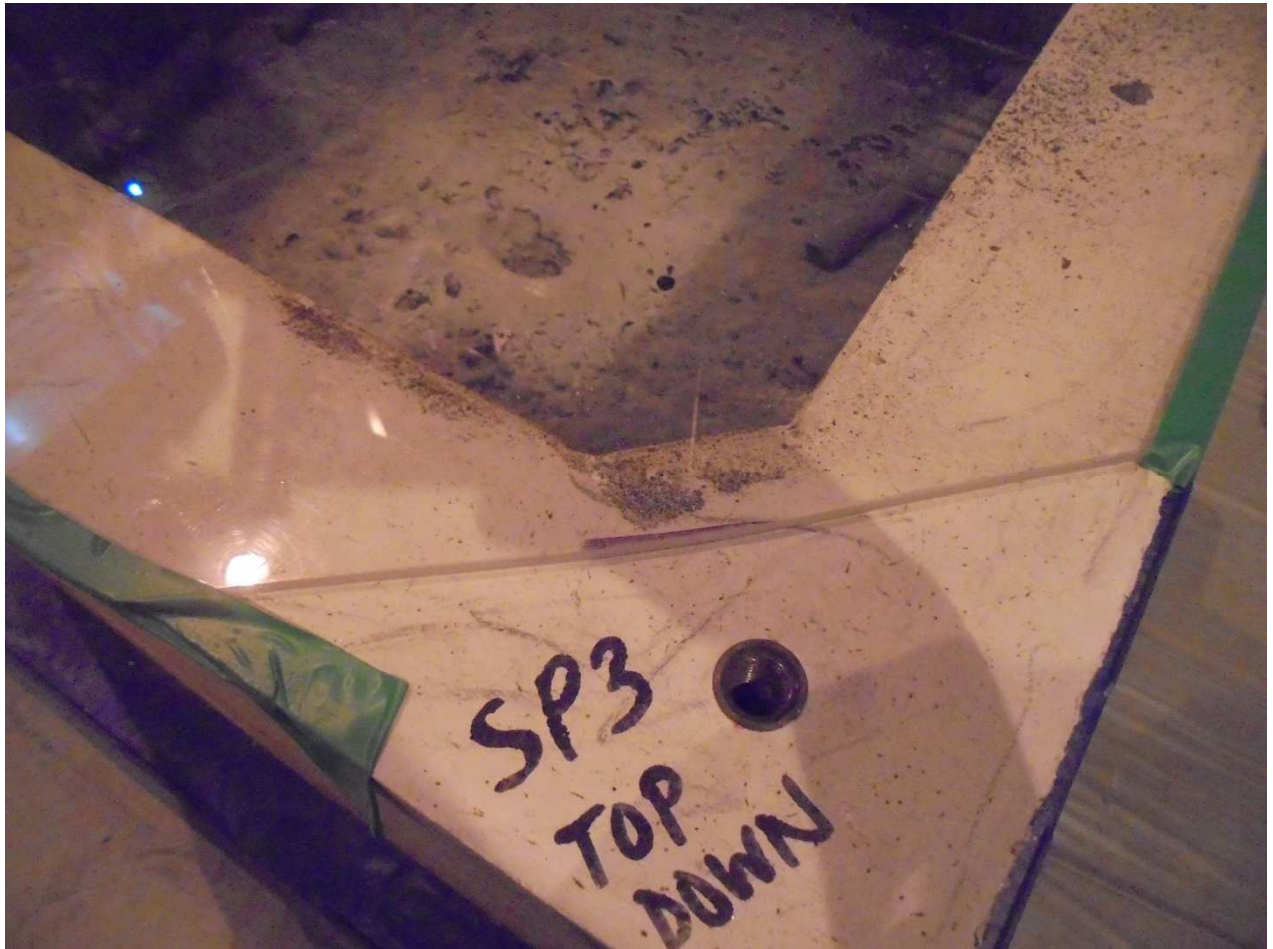


Figure 32: Position of Sample Point 3



Sample Point	Bag Weight (g)	Total Weight (g)	Glass Sample Weight (g)
SP-1.1 Side elevation 0 to 100mm in from side	16.0	162	146
SP-2.1 Side elevation 0 to 100mm in from side	16.0	60	44
SP-2.2 Side elevation 100 to 200mm in from side	16.0	82	66
SP-2.3 Side elevation 200 to 300mm in from side	16.0	50	34
SP-3.1 Top elevation 0 to 100mm in from glass surface	16.0	48	32

Table 8: Details of TH-01 Product Glass Samples

During core drilling the following observations were made:

- Sample point SP1; the drill bit burned out against a hard object at a depth of approximately 100mm into the monolith. This was probably due to contact with a hard metal surface arising from the tins. This indicates that the metallic tins did not fully melt and become fully incorporated into the glass matrix, but have been encapsulated by glass.
- Sample point SP 2; all three target sampling depths were achieved; notable voids were detected at some points indicating presence of bubbles within the monolith.

4.2.TH-02 Re-Melt

The melt was cooled in situ between the 20th September and the 8th October when sufficiently cool to handle, the ICV and product was removed from plant. On 8th October, solids were removed from the SMF filter, in total 0.16 Kg of material was recovered. This amount of material can be considered normal for this process. In addition a sample of the scrub tank liquor was taken. A 200 ml sample was taken from a bulk tank volume of 2000 litres. SMF solids and scrub tank liquor were submitted for radiochemical and chemical analysis.

On 9th October the product monolith was presented for sampling, the bulk product appeared to have a glassy surface and appeared to be a satisfactory product. As in TH-01, the Joule heating electrodes had thinned significantly. The mass of the glass monolith within the CRB was measured to be 197Kg. The relatively low product mass resulted in a product container which approximately half full, this was due to the inability to feed more than 12 kg during the melt cycle due to the failure of one of the heating phases as described earlier.



Figure 33: TH-02 Re-Melted Product.

Samples were recovered from two positions within the monolith, one from the glass surface another from a side elevation. The sampling points are shown in Figure 34 and Figure 35. As in melt TH-01, samples were taken to facilitate analysis and provide radiochemical and chemical data to determine material and activity balances for the process and to indicate the degree of homogeneity



Figure 34: Sample point SP 2 From Side Elevation



Figure 35: Sample point SP 1 From Top Elevation

Details of the glass samples taken from the TH-02 re-Melt product are shown in Table 9.



Sample Point	Bag Weight (g)	Total Weight (g)	Glass Sample Weight (g)
SP-1.1 top elevation 0 to 100mm in from top surface	16.0	39.37	23.37
SP-1.2 top elevation 100 to 200mm in from top surface	16.0	48.62	32.62
SP-1.3 top elevation 200 to 300mm in from top surface	16.0	70.56	54.56
SP-2.1 Side elevation 0 to 100mm in from side	16.0	147.1	131.1
SP-2.2 Side elevation 100 to 200mm in from side	16.0	142.41	126.41
SP-2.2 Side elevation 200 to 300mm in from side	16.0	157.14	141.14

Table 9: Details of TH-02 Re-Melt Product Glass Samples

There were no operational issues associated with taking the glass samples.

Results of the Radiochemical and chemical analysis for both experiments will be presented in a separate report under Work Package 4 of this project.



5. Conclusions

Thermal treatment of 279Kg of representative cementitious waste stream (TH-01) with a pre-treatment waste loading of 49% has been successfully demonstrated using the GeoMelt ICV system. Macroscopic observation of the product indicated that a glassy monolith with broad homogeneity has been produced. Visual inspection of the product would suggest it would be disposable against all key disposability criteria. Observations made during product sampling indicate that at least some of the original metallic objects present in the simulated waste remained on completion of processing. All plant operating parameters observed during this melt were within expected norms.

Similarly thermal treatment of 238Kg of a sludge stream consisting of clinoptilolite, sand and Magnox sludge (TH-02) with a pre-treatment waste loading of 72% has also been successfully demonstrated. The product manufactured from this experiment also had a glassy appearance which appeared to be homogenous. Operational difficulties experienced during the first attempt to melt this material were a consequence of operator error and not a consequence of the thermal treatment process. The failure of the first attempt presented an opportunity to demonstrate a cold re-start of the GeoMelt process. During the re-melt, a further plant abnormality was observed which necessitated completion of the melt on a single phase. This abnormality was due to an operator error and was also not a consequence of the thermal treatment process.

Both experiments have provided successful demonstrations of co-processing; utilising one potential waste material to form the primary glass former for other waste components.