

DELIVERABLE REPORT



Thermal treatment for radioactive waste minimisation and hazard reduction

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Theramin Project Partners

Andra	Agence nationale pour la gestion des déchets radioactifs – 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
ORANO	Orano - France
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
AWE	The Atomic Weapons Establishment – UK
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
INL	Idaho National Laboratory – USA
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

DIVA	Advanced Instrumented Vitrification Process (<i>Dispositif Instrumenté de Vitrification Avancé</i>)
EC	European Commission
HEPA	High Efficiency Particulate Air
IER	Ion Exchange Resins
IRIS	Solid Incineration Research Facility (<i>Installation de Recherche en Incinération des Solides</i>)
THERAMIN	Thermal treatment for radioactive waste minimization and hazard reduction
R&D	Research and Development
SHIVA	Hybrid System for Advanced Incineration Vitrification (<i>Système Hybride d'Incinération Vitrification Avancé</i>)
TRL	Technology Readiness Level
WP	Work Package
wt. %	Weight percent
XRF	X-Ray Fluorescence



1. SHIVA technology for the incineration-vitrification of organic and inorganic ion-exchange media

1.1 Executive summary

The SHIVA process is well suited to treat organic and mineral waste with high alpha contamination management capabilities. It allows, in a single reactor, waste incineration by plasma burner and ash vitrification in a cold wall direct glass induction melting system.

The SHIVA trial shows the success of the processing of a waste stream containing a mixture of mineral and organic ion exchange media. The load rate reaches a promising 38 wt.% and the wasteform appears homogeneous on the millimeter scale.

Waste composition	<ul style="list-style-type: none">➤ 45 wt.%: zeolites (chabazite)➤ 44 wt.%: diatoms➤ 5.5 wt.%: strong acid ion exchange resin➤ 5.5 wt.%: strong base ion exchange resin
Waste load	38 wt.% <ul style="list-style-type: none">➤ waste: 25 kg➤ alumino-borosilicate glass frit: 40 kg
Trial duration	24 h (3 × 8 h feeding campaigns)
Wasteform	Homogeneous glass

1.2 Introduction

As part of the THERAMIN project, studies were carried out on the treatment and conditioning of mixed waste containing inorganic and organic ion exchange media. The trial described in this section is based on the SHIVA incineration–vitrification process combining heating by transferred arc plasma and direct induction. The waste incineration is carried out above the plasma torches and a molten glass bath heated by the induction generator.

This trial aims to test the current configuration of the SHIVA process for the thermal treatment of mixed waste containing ion exchange resins.

1.3 Description of the technology to be deployed

The SHIVA process (Figure 1) uses cold wall direct glass induction melting and plasma burner technologies. It is therefore an incineration–vitrification process well suited for organic wastes.

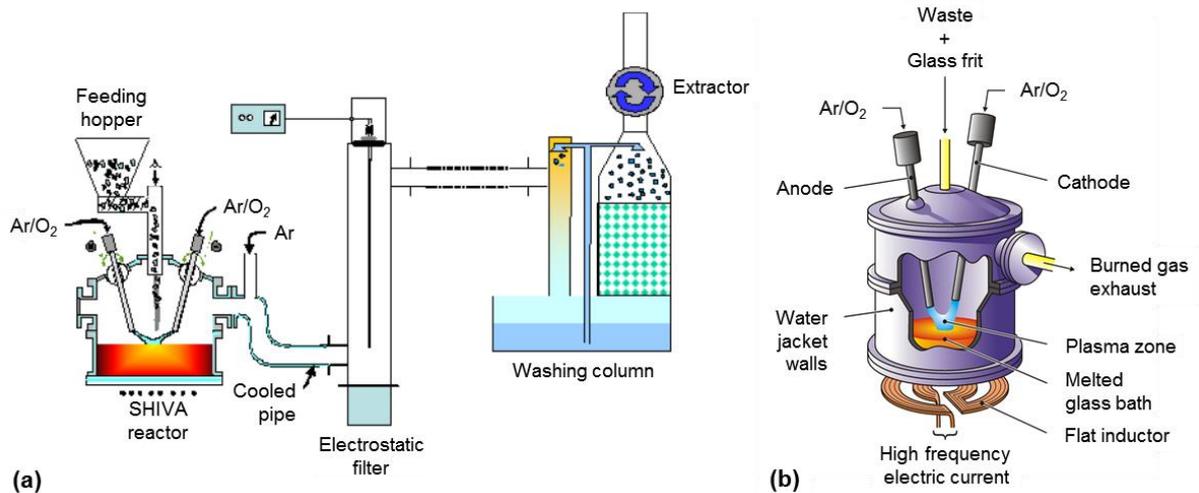


Figure 1: (a) Simplified diagram of the SHIVA process and (b) artist's view of the reactor.

SHIVA consists of a water-cooled, stainless steel cylindrical reactor, equipped with a flat coil at the bottom and a transferred arc plasma system in the reactor chamber (Figure 2). The SHIVA bottom structure is built to be transparent to the electromagnetic field such that the glass is directly heated by the field while the cylindrical shell is not. The plasma torches were developed to minimize their maintenance requirements, in particular graphitic consumable electrodes automatically fed. As oxygen is used and no secondary chamber is present, the gas treatment is simple and small. It consists of an electrostatic tubular filter and a gas scrubber. The dust in the filter is recovered in a bottom ashtray for recycling. The glass is drained from the cold crucible.

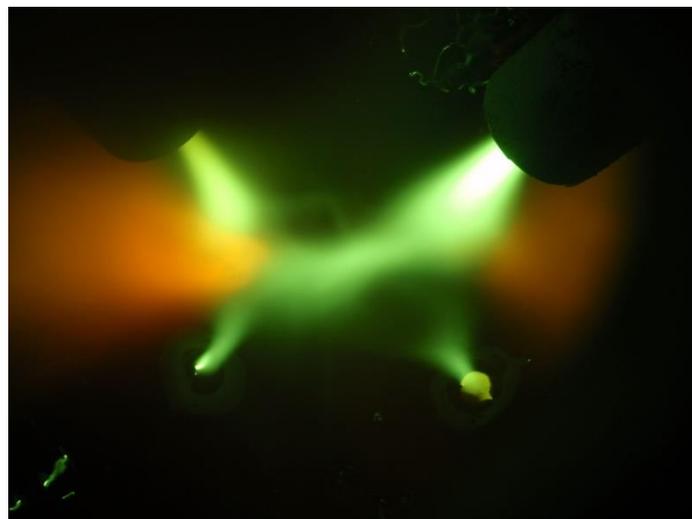


Figure 2: Transferred arc plasma system.

SHIVA was designed to treat organic waste, potentially with high chloride or sulphur content, with high alpha contamination management capabilities. The waste can be in solid or liquid form. SHIVA is not compatible with metallic waste or a separate metallic phase during treatment.



The technology is specifically designed to operate in a hot cell for high level or intermediate level waste. The small size and internal cold walls of the vessel were chosen for high alpha contamination waste capabilities. The end product is a glass.

The SHIVA process is at TRL 5-6 as a full-scale inactive pilot which has been tested by CEA since 1998 for various wastes.

1.4 Feeds

The waste selected for the trial was a 25 kg mixture of inorganic and organic ion exchange media composed of zeolites, diatoms, strong acid IER, and strong base IER (Table 1). The zeolites came from crushed natural zeolite rock composed of 60 ± 5 wt.% chabazite, having a high specific surface $> 100 \text{ m}^2\cdot\text{g}^{-1}$ and a cation exchange capacity of $2 \text{ meq}\cdot\text{g}^{-1}$ (SOMEZ, 2014). Diatomite is a high porosity material used, once cleaned and reduced in powder, as a filtration and clarification media (LAFFORT, 2017). The IER were styrene divinylbenzene copolymer with a strong acid cation exchange site $-\text{SO}_3^-\text{H}^+$ for cation exchange ($\geq 1.90 \text{ eq}\cdot\text{L}^{-1} \text{ H}^+$ form) and a strong base anion exchange site ($-\text{N}(\text{CH}_3)_3^+\text{OH}^-$) for anion exchange ($\geq 1.20 \text{ eq}\cdot\text{L}^{-1} \text{ OH}^-$ form) (LENNTECH, 2018a, LENNTECH, 2018b). The IER contained 50 wt.% of water.

Waste	Fraction in the mixture (wt.%)	Quantity processed (kg)	Supplier and reference
Zeolites (including 60 ± 5 % chabazite)	45	11.3	SOMEZ Siliz [®] 14, ZN-024-14
Diatoms	44	11.0	LAFFORT Diatomyl P0
Strong anion IER	5.5	1.4 of wet product	LENNTECH Amberlite [™] IRN77
Strong base IER	5.5	1.4 of wet product	LENNTECH Amberlite [™] IRN78

Table 1: Waste feeds for SHIVA trial.

Vitrification was allowed by the addition of 40 kg of a glass frit whose composition is given in Table 2. It should be noted that in a first approach, the vitrification was carried out with a known and available glass frit. Its composition could be optimized for this specific waste as a result of a formulation study.

Inputs were therefore composed of 38.4 % of waste and 61.5 % of glass frit. It should be noted that ion exchange media were not exchanged with solutions containing radionuclide surrogates prior to the trial: IRE were therefore completely burned. The theoretical composition of the glassy end product (Table 2) can be calculated from the theoretical compositions of glass frit, zeolites, and diatoms (SOMEZ, 2014, LAFFORT, 2017), and the amounts of these materials introduced into the process.



	Glass frit	Zeolites	Diatoms	IER	Waste glass
Al ₂ O ₃	5.2	17.0	3.0	Completely burned	7.2
B ₂ O ₃	14.7	-	-		9.8
CaO	4.1	5.5	0.5		3.8
CoO	0.7	-	-		0.5
Fe ₂ O ₃	4.0	2.5	1.6		3.4
K ₂ O	-	4.5	0.2		0.9
Li ₂ O	2.0	-	-		1.3
MgO	-	1.3	0.3		0.3
Na ₂ O	10.0	-	2.3		7.1
Nd ₂ O ₃	7.3	-	-		4.9
NiO	0.5	-	-		0.3
SiO ₂	46.6	51.5	91.1		57.2
TiO ₂	-	-	0.5		0.1
ZnO	2.5	-	-		1.7
ZrO ₂	2.4	-	-		1.6

Table 2: Theoretical compositions (expressed in oxide weight percent) of glass frit, zeolites SOMEZ Siliz[®]14, diatoms LAFFORT Diatomyl P0, and calculated composition of the glassy end product. The 7 main oxides (> 93 wt.%) of the end product are written in bold.

1.5 Trial information

1.5.1 Heating systems

To ensure complete mineralization of the waste and guarantee its incorporation into the vitreous matrix, the two modes of heating — induction and plasma torches — were used simultaneously during the waste feeding campaigns.

- The induction heating system was started at the beginning of the trial and was maintained throughout the trial duration to maintain a molten glass bath at the bottom of the SHIVA reactor. The induction was started using a titanium starter ring and the generator power was incrementally increased from 40 kW up to 90 kW.
- The sleeves and electrodes of the torches were fed with argon during the start-up phases and with a mixture of argon and oxygen ($\approx 10 \text{ L}\cdot\text{min}^{-1}$) during the waste



introduction periods. The electrodes were pushed a few millimeters an hour to compensate for their wear.

1.5.2 Feeding

The waste feed was made by the upper part of the reactor using a feeding hopper (Figure 1 and Figure 3) and a worm screw with a feed average of $1 \text{ kg}\cdot\text{h}^{-1}$ during the three 8-hour feed campaigns. This sequencing was used to allow a daytime operation for the waste incineration.

Before the start of the trial, the reactor was pre-filled with 40 kg of glass frit.

At this stage of the demonstration, no maximum were sought in terms of processing capacity and waste load in the vitreous matrix. These parameters would be higher in the case of a process on an industrial scale.



Figure 3: Waste introduction system.

1.5.3 Generated effluents

The incineration of 2.8 kg of IER containing 50 % of moisture involved additions of soda lye to keep the solution in the washing column at a neutral pH to avoid corrosion issues. This neutralization causes the release of 1.3 L of chlorinated effluents.

With a maximum quantity of 2.8 kg of organic matter incinerated, the production of gaseous effluents will be 2.8 m^3 .

1.5.4 Produced wasteform

The end product of the process is a glass which is macroscopically (millimeter scale: visual inspection) homogeneous (Figure 4). This wasteform was characterized in the framework of the THERAMIN WP4 (refer to section 3). In this context, the wasteform is broken into pieces and a sample is selected by quarter splits.



Figure 4: Waste glass sample from the SHIVA trial.

1.6 SHIVA trial: Concluding remarks

The SHIVA trial conducted in the framework of the THERAMIN project showed the success of the process for the thermal treatment of a mixture of organic and mineral waste composed of zeolites, diatoms and ion exchange resins. The waste load of 38 wt.% is high and could probably be increased in the future. Indeed, during this feasibility trial, it was not sought to maximize the waste load and the processing capacity. The waste product is a aluminoborosilicate glass, macroscopically homogeneous, whose long term behavior could be characterized according to proven methodologies (AFNOR Normalisation, 2008).



2. In-Can Melting technology for the vitrification of ash

2.1 Executive summary

The In-Can Melter is a metallic crucible heated in a refractory furnace using electrical resistors allowing in-container vitrification.

The In-Can Melter trial was designed to vitrify inactive ash coming from the incineration of technological waste. Thus, this trial aims to demonstrate the feasibility of the confinement in a vitreous matrix of by-products of existing incineration processes. The ash is pelletized to allow its introduction into the can without dust emissions and then is incorporated in a 50 wt.% waste loading confinement matrix. The full-scale trial was preceded by laboratory-scale and bench scale tests.

Waste	<ul style="list-style-type: none">➤ Ash from incineration of technological waste (e.g., cotton, polyvinyl chloride, latex, neoprene, polyethylene)
Waste load	49 wt.% <ul style="list-style-type: none">➤ waste: 26 kg of ash➤ sodium-borosilicate glass frit: 26 kg➤ bentonite (ash pellet binder): ≈ 1kg
Trial duration	15 h <ul style="list-style-type: none">➤ Melting of the pre-loaded charge: 8 h➤ Feeding of the glass pellets and glass frit: 1 h➤ Soaking of the glass: 2 h➤ Recycling of the dust scrubber into the can: 2h➤ Soaking of the glass: 2h➤ Free cooling of the can
Wasteform	Homogeneous crystallized glass

2.2 Introduction

Studies were carried out on the treatment and conditioning of ash coming from the incineration of technological surrogate waste. The trial described in this section is based on the CEA In-Can Melting process consisting in a metallic crucible melter heated in a simple refractory furnace using electrical resistors. Prior to the full-scale trial (≈ 50 kg), laboratory-scale (≈ 10 g), and bench scale (≈ 1 kg) tests were conducted to select optimized operating conditions.

2.3 Description of the technology to be deployed

The In-Can Melter is a metallic crucible heated in a refractory furnace (Figure 3). The can is renewed after each filling.

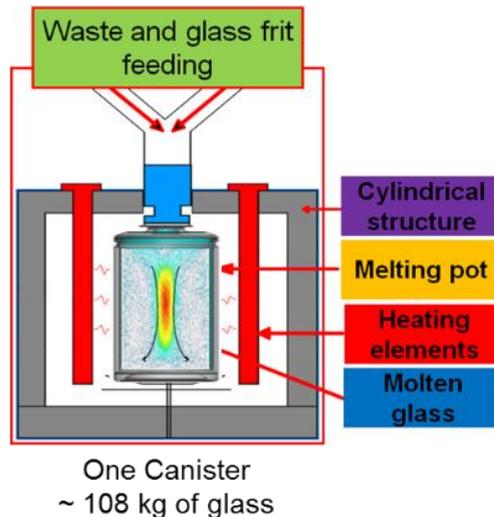


Figure 5: Simplified diagram of the In-Can Melter.

The process can support either liquid or solid waste feeds. With the current gas treatment process, it can only tolerate small amounts of organics. It can also accept a small fraction of metal in the waste. The design ensures that the process can operate remotely for high-activity waste. The design can also be adapted for dealing with plutonium containing material in gloveboxes. The end product can be glass, glass ceramic or simply a high-density waste product.

The technology is at TRL 7 and a full-scale pilot has undergone inactive commissioning in France with inactive simulants of alpha effluent. The technology is currently being qualified for solid high level waste.

2.4 Feeds

2.4.1 Ash production

The waste selected for this vitrification trial is ash from multiple incineration tests of surrogate technological waste (polyvinyl chloride, latex, neoprene, polyethylene, cotton...) produced by the CEA IRIS process. IRIS is a research facility for the incineration of solids developed to treat organic waste from glove boxes in the nuclear industry, contaminated with alpha bearing actinides and containing high quantities of chlorine (Figure 6). The robustness and efficiency of this process is based on a decoupling of (i) the step of elimination of corrosive materials such as chlorine and (ii) the step of combustion of the organic waste. Figure 6 shows that the organic waste first goes into a pyrolysis step at a temperature of 500 °C to remove the most corrosive gaseous compounds and then in a calciner (900 °C) fed with oxygen to complete the combustion while concentrating the contamination in the mineral ash. The relatively long residence time in furnaces with low gas flow rates makes it possible to produce carbon-free ash concentrating almost all of the initial activity. The gas treatment system — consisting of a postcombustion chamber followed by electrostatic filtration — ensures excellent purification.

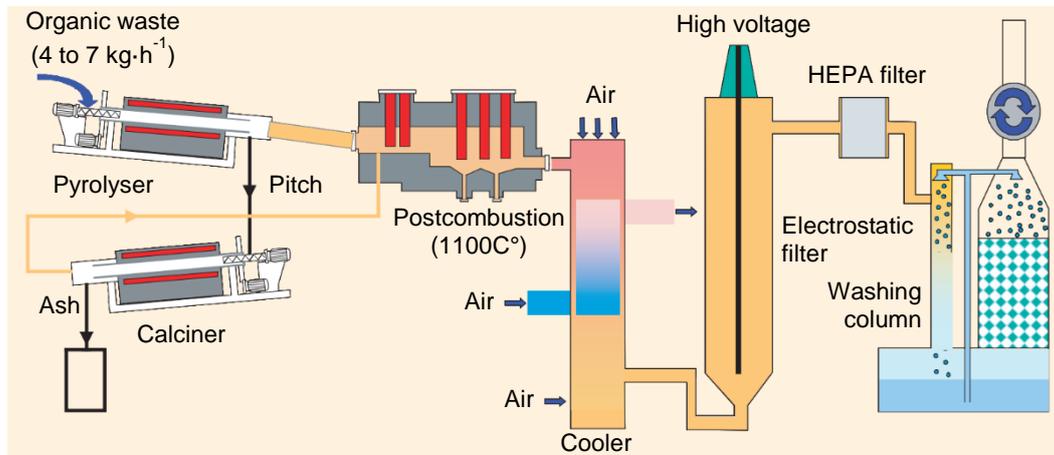


Figure 6: IRIS process for the treatment of organic waste.

2.4.2 Feeds preparation

In order to have a sufficient quantity of ash for the tests at different scales, 230 batches of ≈ 220 g each — coming from various R&D trials with the IRIS process — were homogenized. This homogenization of 50 kg (≈ 250 L) of ash was carried out by mixing for 2 hours in a Morton Mixers ribbon blender (Figure 7) ensuring the chemical homogeneity of the ash and a reduction of the size of the ash clusters. The composition of the final mixture was analyzed by X-ray fluorescence (XRF) spectroscopy (Table 5).

The typical characteristics of this ash are an apparent density of $0.2 \text{ g}\cdot\text{cm}^{-3}$ (increased to $0.6 \text{ g}\cdot\text{cm}^{-3}$ after homogenization), a true density measured by helium pycnometry of $1.9 \text{ g}\cdot\text{cm}^{-3}$ and a high flying behavior. This flying behavior forced us to consider an agglomeration method to avoid (i) clogging of the feeding pipe and (ii) dust carry over when feeding ash into the can.

In order to carry out this temporary densification, pelletizing by a Frogerais rotative press was conducted. It should be noted that this press did not allow to jointly pellet ash and glass frit, the latter being too abrasive. Approximately 60,000 ash pellets (Figure 8.a), representing 9 kg of ash, were therefore produced in the presence of 10 wt.% of bentonite used as a binder ensuring the mechanical cohesion of the pellets. Bentonite was chosen after the laboratory scale tests (refer to section 2.4.3). The pellets production rate is around 27,000 units per hour ($\approx 3 \text{ kg}\cdot\text{h}^{-1}$). Their diameter is 7 mm for a height of 2 to 3 mm (Figure 8.b) and a mass of 0.12 ± 0.04 g (Figure 8.c), representing an average densification ratio of 72%.



Figure 7: (a) Morton Mixers ribbon blender. Ash in the blender (b) before and (c) after homogenization.



Figure 8: (a) Total production of 60,000 pellets, (b) detail view of an ash pellet and (c) mass distribution of a sample of 50 pellets.

2.4.3 Choice of adjuvants and waste loading

These preliminary tests aimed at selecting the best operating conditions in order to obtain an optimized waste load and a good quality end product. The laboratory-scale tests consisted of bringing into contact — at 1100 °C during 2 hours — different amounts of ash and glass frit, in the presence or absence of an adjuvant. These tests are carried out using a few grams of materials. Two types of binder for ash pelletizing were tested: organic — sorbitol $C_6H_{14}O_6$ and fructose $C_6H_{12}O_6$ — and mineral — bentonite, containing mainly montmorillonite $(Na,Ca)_{0.33}(Al,Mg)_2Si_4O_{10}(OH)_2(H_2O)_n$.



In a first approach, the vitrification was carried out with a known and available glass frit composed of 35.5 wt.% SiO₂, 32.4 wt.% B₂O₃, and 32.1 wt.% Na₂O. Its composition could be further optimized for this specific waste, requiring a formulation study, which was not done in the framework of the project.

At the end of the laboratory-scale tests, the crucibles were cut after immobilization in epoxy resin and the products obtained were observed under a binocular magnifier (Figure 9). The success criteria were the followings: (i) having a homogeneous end product and (ii) the absence of foaming during the melting. The best results were obtained for a 50/50 mixture between ash and glass frit with an addition of 10% fructose or bentonite relative to the ash mass. However, bentonite was preferred to avoid possible emission of CO_x gas during combustion.

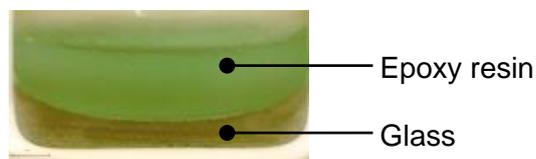


Figure 9: Cut crucible observed with a binocular magnifier showing a 50/50 mixture between ash and glass frit with an addition of 10% fructose and after pretreatment for 2 h at 120 °C and a thermal treatment of 2 h at 1100 °C.



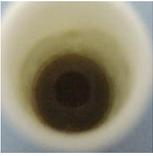
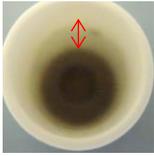
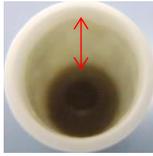
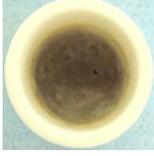
 <p>Ash/glass ratio: 50/50 Glass: yes Expansion: ½ crucible height</p>	 <p>50/50 + 10% fructose Glass: yes Expansion: ½ crucible height</p>	 <p>50/50 + 10% sorbitol Glass: yes Expansion: ¾ crucible height</p>
	 <p>60/40 + 10% fructose Glass: no Expansion: all crucible height</p>	
	 <p>70/30 + 10% fructose Glass: no Expansion: ¾ crucible height</p>	
	 <p>80/20 + 10% fructose Glass: no Expansion: no</p>	
	 <p>100/0 + 10% fructose Glass: no Expansion: no</p>	

Table 3: Comparison of laboratory-scale melting tests at 1100 °C of different ash/glass weight ratios using organic binders. Tests were carried out after a pretreatment of 2h at 120 °C except the one of the left column.

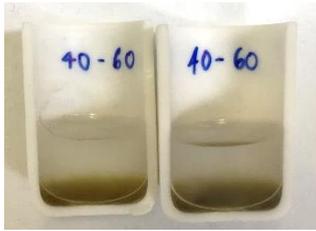
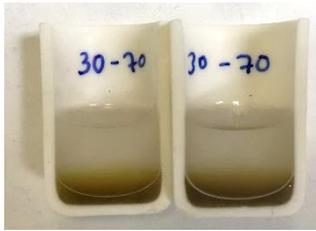
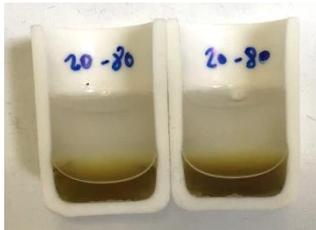
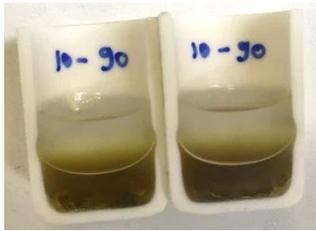

Ash/glass ratio: 50/50 + 10% bentonite

60/40 + 10% bentonite

70/30 + 10% bentonite

80/20 + 10% bentonite

90/10 + 10% bentonite

Table 4: Comparison of laboratory-scale melting tests at 1100 °C of different ash/glass weight ratios using bentonite as a binder.



2.5 Bench scale demonstration

The last step before carrying out the full-scale trial consisted in a bench scale test (Figure 10) using the optimized conditions determined after laboratory-scale tests. This test was carried out in an Inconel 601 crucible of outer diameter 100 mm and a wall thickness of 5 mm, filled with 400 g of ash (not pelletized) and 400 g of glass frit. Both materials were mixed before being poured. The mix is heated at $300\text{ °C}\cdot\text{h}^{-1}$ and maintained at 1100 °C for 8h. This test confirmed the good corrosion behavior of the Inconel 601 and helped to determine the possible quantity to be pre-loaded in the can and the tapped density of the feeds ($\approx 1\text{ g}\cdot\text{cm}^{-3}$). A mass loss of 2.5 wt.% is observed during the test after which a glass-ceramic wasteform is obtained, with a density of 2.6 and a calculated composition given in Table 5.

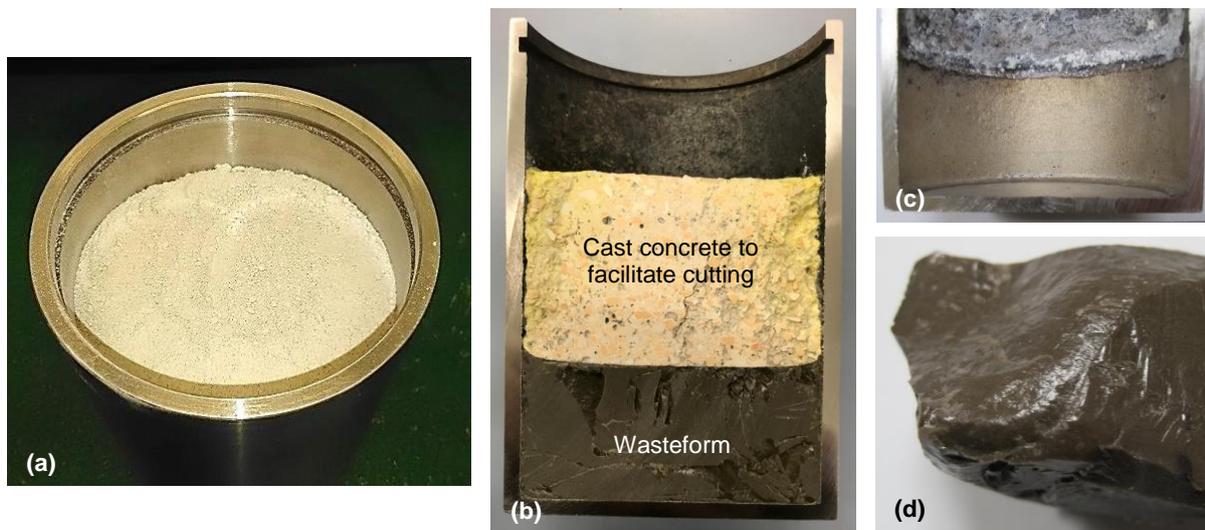


Figure 10: (a) Ash/glass frit mixture poured in the Inconel 601 crucible, (b) cut crucible after the test, (c) internal surface of the crucible after the test, (d) wasteform obtained.



Element	Ash (analyzed, XRF)	Glass frit	Wasteform (calculated)
Al	11.9		6.0
B	0.0	10.0	5.0
Ba	0.7		0.4
Bi	4.0		2.0
Ca	9.9		5.0
Cl	1.4		0.7
Cr	0.1		0.1
Fe	0.5		0.3
K	2.3		1.1
Mg	1.6		0.8
Na	0.6	23.7	12.1
Ni	0.7		0.3
P	1.6		0.8
S	0.3		0.2
Sb	0.2		0.1
Si	7.5	16.6	12.0
Ti	0.5		0.2
Zn	8.1		4.1

Table 5: Compositions (expressed in element weight percent) of the feeds and the wasteform. The main elements are written in bold. Oxygen is supposed to be the balance to 100%.

2.6 Trial information

2.6.1 Feeding and course of the trial

The full-scale trial was carried out using the In-Can mockup DIVA (Figure 11) equipped with a resistive furnace of maximum power 35 kW and a complete gas treatment system and an Inconel 601 can with an outer diameter of 400 mm, a height of 600 mm and a wall thickness of 10 mm.

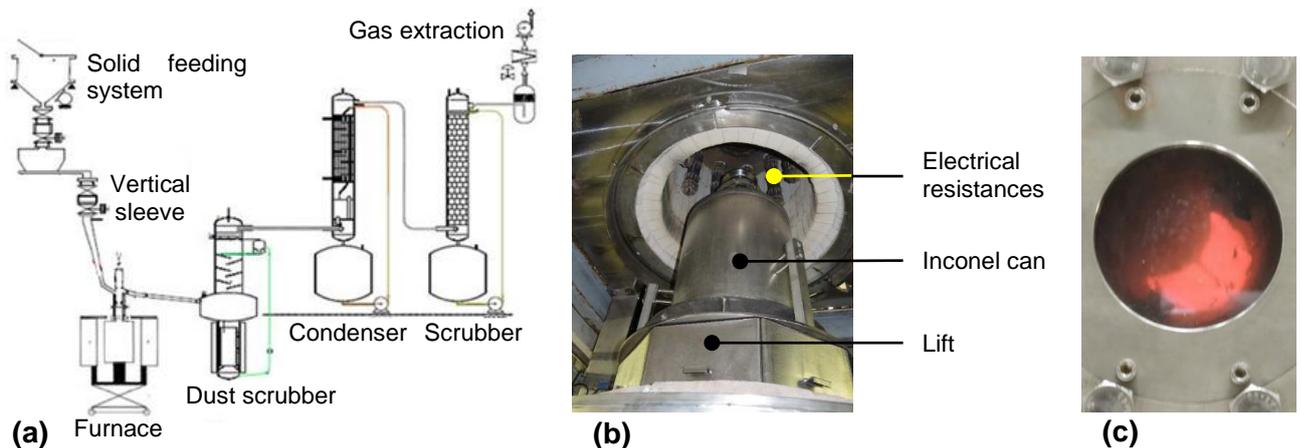


Figure 11 : (a) Diagram of the In-Can mockup DIVA and pictures of (b) the can introduction in the furnace and (c) the molten glass bath seen through the porthole.

The first step of the test was the preloading of the can with 25 kg of glass frit and 17.8 kg of non-pelletized ash. This mixture was heated up to 1100 °C at 300 °C·h⁻¹. Then 9.2 kg of ash pellets were introduced into the can by the solid feeding system at 10 kg·h⁻¹ (a batch of 500 g as shown in Figure 12 every 3 min) followed by 1 kg of glass frit. After that, the mixture whose composition is given in Figure 13 was soaked for 2 h. Two additional hours were added to recycle the dust scrubber deposits followed by a final soaking of 2 h at 1100 °C. Then, the can was cooled down freely (Figure 14). A mass loss of 2.3 wt.% — consistent with the one measured during the bench scale test (2.5 wt.%) — is observed.

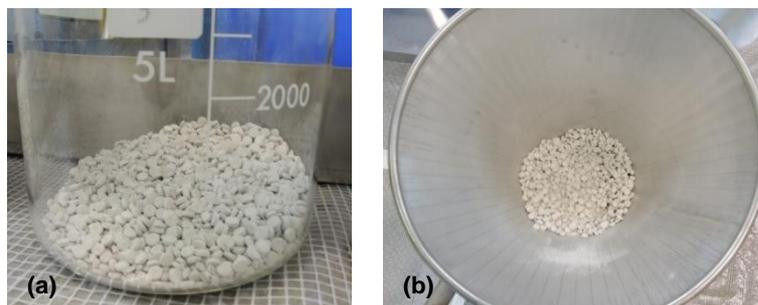


Figure 12: (a) Batch of 500 g of pellets fed every 3 minutes for 1 hour through the (b) feeding funnel.



Figure 13: Distribution of the feeds in the In-Can trial

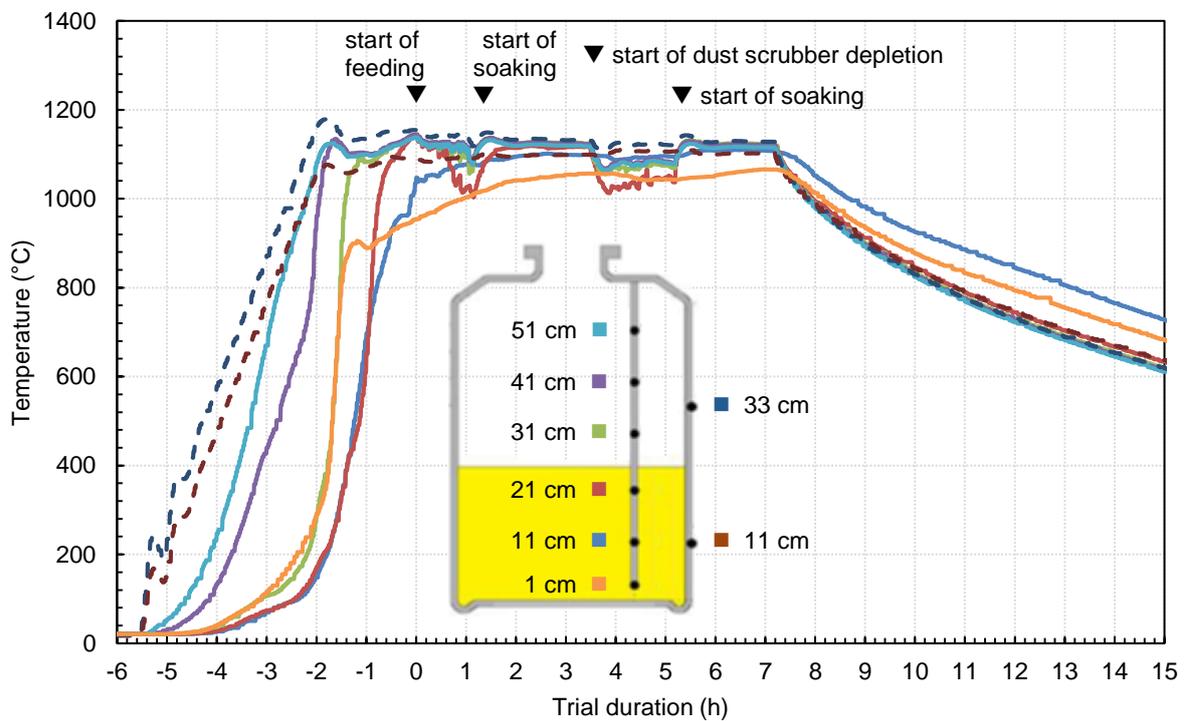


Figure 14: Temperature evolution during the trial measured on walls (top and bottom of the can) and by thermocouples placed at different heights inside the can.

2.6.2 Produced wasteform

Note that to respect the deadlines imposed for the delivery of WP4 reports, it is the material resulting from the bench scale test (refer to section 2.5) that was analyzed in the framework of WP4 (refer to section 3). The end product of the bench scale (Figure 10.d) is a glass ceramic which is macroscopically homogeneous. The coring made in the can from the full-scale trial (Figure 15) shows a wasteform having the same aspect (by visual inspection) as the material from the bench scale test.



Figure 15: Coring of the wastefrom from the In-Can Melter trial.

2.7 In-Can Melter trial: Concluding remarks

The In-Can Melter trial conducted in the framework of the THERAMIN project showed the success of the process for the vitrification of ash coming from the incineration of organic waste from glove boxes in the nuclear industry. A very good waste load rate of 50 wt.% was achieved in this first approach. This trial also made it possible to begin the technical reflection required for the introduction of very powdery solids into the can while avoiding the emission of dust: in this trial, a temporary densification by pelletizing was implemented. The wastefrom is a crystallized glass.



3. Post-trial activities

The wasteforms produced after the SHIVA trial and the bench scale test preceding the In-Can Melter full-scale trial were characterized in the framework of the THERAMIN WP4.

The purpose of the characterizations is to determine the sample:

- degree of homogeneity and verify the absence of free liquid or gas,
- overall chemical composition,
- amorphous or crystalline nature and the structure of the possible crystals.

The analytical techniques that were used are:

- scanning electron microscopy associated with with X-ray energy dispersive microanalysis,
- inductively coupled plasma analysis after dissolution of the solid,
- X-ray diffraction.

Moreover, the chemical durability of the samples against the hydrolysis mechanism will be estimated by a leaching test based on the ASTM Standard Test Method C 1285 – 14 (ASTM International, 2014).

4. References

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