Vitrification of F-Area Americium/Curium: Feasibility Study and Preliminary Process Recommendation

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DOE Contract No. DE-AC09-89SR18035

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WSRC-TR-94-0211 Rev. 0

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Summary

Work was performed to identify a process to vitrify the contents of F-canyon Tank 17.1. Tank 17.1 contains the majority of americium (Am) and curium (Cm) contained in the D.O.E Complex. Oak Ridge National Laboratory (ORNL) has made a formal request for this material as fuel for production of Cf^{252} and other transplutonium actinides. The Am and Cm (and associated lanthanide fission products) are currently in nitric acid solution. Transportation of the intensely radioactive Am/Cm in liquid form is not considered possible. As a result, the material will either be solidified and shipped to ORNL or discarded to the Tank Farm. NMPD, therefore, requested DWPT to determine if the Tank 17.1 material could be vitrified, and if it was vitrified could the americium and curium be successfully recovered.

Research was performed to determine if the Tank 17.1 contents could indeed be mixed with glass formers and vitrified. Additional studies identified critical process parameters such as heat loading, melter requirements, off-gas evolution, etc. Discussions with NMPD personnel were initiated to determine existing facilities where this work could be accomplished safely.

A process has been identified which will convert the Am/Cm material into ~300kg of glass. This will be done by initially removing the large quantities of transition metals and aluminum. Oxalic acid will be added to Tank 17.1 and the actinides and lanthanides will be precipitated as oxalates. The remaining aqueous fraction will be removed and the lanthanides and actinides will then be redissolved. The solution will be evaporated and steam stripped down to ~100g/l solids and 1N nitric acid prior to being transferred into a receipt/feed tank in the Multi-Purpose Processing Facility (MPPF - formerly Section 18 of F-Canyon). The Am/Cm solution will then be fed directly into a bushing melter. Glass beads will be added simultaneously alongside the solution and the mixture will be vitrified and cast into stainless steel canisters. The filled glass canisters will be allowed to cool and deconned prior to packaging for transport to ORNL. The melter will be capable of a minimum 1-2kg/hr glass output, so total processing time will be on the order of a few hundred hours.

The glass is a borosilicate composition designed to be selectively attacked by nitric acid. Complete recovery of Am and Cm surrogates (La, and Nd) from the glass has been demonstrated by a nitric acid extraction technique. While the glass can be attacked by concentrated acid, it is approximately 1000 times more durable than the required specification for high-level waste glass. The Am/Cm glass produced will simultaneously act as both a product form for ORNL and also meet the acceptance criteria for high-level waste if all of the Am/Cm glass is not processed by ORNL.

This document details the results of our initial effort. Supporting documents concerning heat generation, off-gas modeling, melter design, and a process engineering flowsheet developed by NMPD are included. A reference list of the initial design basis glass and canister product specifications is given in Table 1.

Table 1. Am/Cm Glass Product and Canister Characteristics (Process Bases)

Glass Type	Borosilicate
Tank 17.1 Component	40wt% (as oxide) of the final glass composition
Total Production	~ 300kg glass@
Specific Gravity	~ 4.5±0.5 kg/L
Power Density	~ 0.135W/ml*
Melt Temperature	≤ 1400°C
Aqueous Durability	1000X more resistant than specification for HLW glass
Recoverability	100% recovery of Am & Cm surrogates La & Nd demonstrated with concentrated nitric acid extraction
Glass per Canister	1.6 - 1.7L / 7.2 - 7.7kg
# Canisters Projected	~ 45*
Max Centerline Temperature	~ 275°C*
Canister Shape	Right circular cylinder
Canister Material	304L Stainless Steel
Canister Dimensions**	43cm tall / 8.2cm OD (17.25" / 3.25")
Canister Wall Thickness	0.64cm bottom / 0.32cm sidewall (0.25" / 0.125")

@ This value assumes that the oxalate precipitation is performed, i.e. no transition metals, alkali, or aluminum in the feed stream.

* The power density, number of canisters projected, and glass centerline temperature was calculated assuming forty weight percent Tank 17.1 oxides. A glass density of 5.5kg/L was used to be conservative. Additional melts with up to 18 weight percent uranium oxide indicate 4.5kg/L is a more realistic value. This is reflected in the 275°C centerline temperature compared with the value shown in SRT-GTC-94-00027 (Appendix 2).

** The canisters are small enough to fit inside a sleeve used by ORNL for Mark 42 fuel rod sections. An additional overpack canister will also fit inside the ORNL sleeve.

Introduction

F-Canyon (221-F) will eventually undergo Decontamination and Decommissioning (D&D) [1]. Prior to the D&D process, however, a considerable quantity of transuranic elements must be dispositioned. One such source of transuranic material is the Am/Cm solution stored in Tank 17.1 [1,2]. Kilogram quantities of extremely rare Am and Cm isotopes are stored in Tank 17.1, representing the majority of Am and Cm within the D.O.E. Complex [1-4]. Currently SRS has no mission which require Am or Cm isotopes [5]. As a result, the material is effectively a waste at SRS [4]. The primary usage of Am and Cm is as fuel for production of Cf^{252} and other transplutonium isotopes [3]. ORNL, which has the transplutonium production and Isotope Sales missions, has made a formal request to obtain the Am/Cm [3]. Shipment of the Am/Cm to ORNL will therefore allow SRS to successfully convert a "waste" into a product.

It is not considered feasible to ship the Am/Cm to ORNL as a nitric acid solution [6]. If the material cannot be shipped to ORNL, the most likely disposition is discard to the Tank Farm [1,4,7,8]. As part of a program to determine if viable alternatives to Tank Farm discard exist, Nuclear Materials Processing Division (NMPD) tasked Defense Waste Processing Technology (DWPT) to determine if it was possible to vitrify the Am/Cm for shipment to ORNL [1,2]. It was also considered necessary to provide an Am/Cm recovery process that could be performed in the existing ORNL "hot" cells. A corresponding task was issued to Chemical Technology to provide an oxide process for Am/Cm solidification [1,2]. Simultaneously, F-Area Labs and SRTC Analytical Development Section were provided samples from Tank 17.1 to fully characterize the material [1,2].

Work was performed to develop a vitrification process and identify the necessary equipment and facilities. The effort was coordinated between SRTC Analytical Development Section, (ADS), NMPD and DWPF Waste Acceptance personnel. The following sections of this document describe the experiments performed, the type of melter selected, and the required canyon/MPPF operations needed to vitrify the Am/Cm material. The final section discusses recovery of the Am/Cm material from the glass..

Processing Experiments

This section is divided into two parts: An analysis of the composition of Tank 17.1 from a glass processing perspective and feed specification optimization modeling studies are described in Feed Preparation. Initial studies performed to determine processing ranges and relative glass durability are discussed in Glass Processing.

Feed Preparation

An aggressive characterization program was performed on the Tank 17.1 samples by F-Area Analytical Labs and ADS [9,10]. Tank 17.1 contains 15,000L of 4N nitric acid solution [9]. The americium and curium inventories were determined to be approximately 10.1 kilograms (9.7kg Am^{241}) and 2.7 kilograms (2.5kg Cm^{244}), respectively [9]. The majority of dissolved solids are lanthanides, transition metals (predominately iron), and aluminum [9,10]. In total, there are over 270kg of solids (on an oxide basis) in Tank 17.1 [9,10]. The principal Tank 17.1 components are listed in Table 2 [9,10].

Table 2. Tank 17.1 Dissolved Solid Components (listed as oxides) [from 9,10].

<u>Component</u>	<u>Oxide Mass (kg)</u>
Fe	107 (as Fe ₂ O ₃)
Al	31
Nd	30
Ce	14
Pr	14
Na	14
La	12
U	11 (as UO ₂)
Sm	7.1
Cr	6.3
Mn	4.7 (as MnO)
Ni	4.4
Gd	3.6
Pu	2.7
Eu	1.4
Am	10.1
Cm	2.7

In order to ship ORNL the smallest volume of glass with the highest concentration of Am and Cm, it was decided to separate the lanthanides and actinides from the transition metals. Oxalate precipitation processes have been previously developed and demonstrated to separate actinides from aluminum and other metal waste streams [11-14]. Chemical Technology Section (CTS) demonstrated that an oxalate precipitation was indeed feasible for the Tank 17.1 solution [15]. Effectively all of the lanthanide and actinides were precipitated as oxalates [15]. The soluble transition metal, alkali, and aluminum ions remained in solution [15]. Only traces of iron and nickel were detected in the precipitate [15]. By separating the lanthanides and actinides from the other constituents, the total mass of Tank 17.1 oxides will be reduced from 270 to approximately 115kg. Further, the separation results in an expected Am/Cm glass composition closer to the original commercial composition chosen as a basis for this project [16,17].

After removal of the soluble species to Tank 17.2 for waste disposal, the lanthanide and actinides will be re-dissolved in nitric acid (Mn²⁺ will be added to complex the oxalate) [14]. Calculations performed using the Chemical Process Evaluation System (CPES) to simulate the vitrification indicate that the relatively concentrated nitric acid solutions with low solids content will generate considerable NO_x during the vitrification program [18]. Additional simulations performed using higher solids content and lower nitric concentrations were performed to target lower NO_x generating feed specifications. CPES calculations indicate that a 1N HNO₃ solution with approximately 60-100g/L dissolved solids will be an adequate feed-stock for glass production rates of 1-2kg/hr. A detailed description of the CPES simulation experiments is given in Appendix 1.

In order to reduce the amount of potential NO_x, the solution can be evaporated and then steam stripped in Tank 17.3E [19,20]. Tank 17.3E is also the delivery vessel from Section 17 into MPPF, so it will be used to perform both feed processing and transfer functions [19].

Glass Processing

Over fifty glasses representing two distinct glass forming systems (borosilicate and iron-phosphate) were fabricated to determine the best glass for Am/Cm vitrification. A commercial borosilicate composition was selected as the best candidate [17]. The commercial glass was designed to incorporate up to fifty-five weight percent lanthanide oxides (32.5% Nd_2O_3 , 18.3% La_2O_3 , & 3.2% Pr_2O_3) [16]. The iron-phosphate glass had a much lower lanthanide solubility (less than ten weight percent) and would have resulted in a considerably higher volume of Am/Cm glass being produced [21].

Glass Processing - Solubility Specifications

A series of glasses were melted to determine the solubility of the highly concentrated Tank 17.1 lanthanides Nd, La, and Ce in the borosilicate glass. Neodymium and lanthanum are representative of the other tri-valent lanthanides and are also non-radioactive surrogates for Am and Cm [16,22]. Neodymium and lanthanum were shown to be soluble in the glass up to 55wt% as oxides. Also, any combination of Nd_2O_3 and La_2O_3 up to 55wt% is soluble in the glass. Cerium oxide solubility was also determined. Cerium is a major component in the Tank 17.1 solution and is also an excellent non-radioactive surrogate for plutonium [23]. Up to ~18wt% cerium oxide (added as either Ce^{+3} [cerous nitrate] or Ce^{+4} [ceric oxide] in the batch) with total lanthanide fixed at 55wt% made acceptable glass. Cerium oxide concentrations greater than ~18wt% resulted in insoluble CeO_2 precipitating from the melt.

Glasses doped with UO_2 or ThO_2 behaved in a manner similar to glasses containing CeO_2 . Up to 9 mole percent UO_2 or ThO_2 (another surrogate for PuO_2) is soluble in the borosilicate glass. Beyond that level, insoluble UO_2 or ThO_2 precipitates formed. Likewise, a solid solution of $\text{U}_x\text{Ce}_{(1-x)}\text{O}_2$ or $\text{Th}_x\text{Ce}_{(1-x)}\text{O}_2$ precipitated if the glass composition contained a combination of ThO_2 and CeO_2 or UO_2 and CeO_2 greater than 9 mole percent. These data indicate that a limiting factor for solubility of Tank 17.1 oxides in glass will be the sum of UO_2 , PuO_2 and CeO_2 . This sum will need to be controlled to less than 9 mole percent (roughly 16wt%). The other solubility limit appears to be a total lanthanide and actinide content equivalent to the 55wt% demonstrated for Nd_2O_3 and La_2O_3 . The initial target composition easily meets both criteria. Table 3 shows the initial Am/Cm glass target composition and the range of components evaluated.

Glass Processing - Thermal Loading Specifications

Studies were performed to determine the heat generation and maximum internal temperature expected for the target Am/Cm glass. P/THERMAL, a multi-purpose heat transfer code, was utilized to predict the temperatures resulting from the radioactive decay of Tank 17.1 material when solidified as a glass [24]. These calculations were used to determine the maximum canister size possible for the target glass while not exceeding 400°C internal glass temperature. 400°C was chosen as a reference point as this is an acceptance criteria for high-level waste [25]. The 400°C value is based on the glass transition temperature of the less refractory high-level waste glass compositions and is definitely conservative for the Am/Cm glass. The P/THERMAL simulations predict that a glass log 7.5cm diameter by 37.5cm tall of the target composition will have a maximum temperature of approximately 315°C. As the glass density used in the simulations (5.5g/cc) has since been shown to be conservative, the actual maximum temperatures will be lower still [21]. The P/THERMAL simulations are described in detail in Appendix 2.

Table 3. Am/Cm Target Glass Composition (in oxide weight percent).

Component	Target Wt%	Range Tested (Wt%)
<i>SiO₂</i> *	29.8	21 - 30
<i>B₂O₃</i>	6.6	5 - 8
<i>BaO</i>	2.4	2 - 2.5
<i>Al₂O₃</i>	6.6	13 - 3
<i>PbO</i>	13.9	8 - 14
MnO	0.2	
PuO ₂	1.0	**
UO ₂	3.8	0 - 18
CeO ₂	5.1	0 - 18
Am ₂ O ₃	3.9	***
Cm ₂ O ₃	1.0	***
Pr ₂ O ₃	5.0	***
Sm ₂ O ₃	2.6	***
Gd ₂ O ₃	1.2	***
Eu ₂ O ₃	0.5	***
La ₂ O ₃ #	5.8	0 - 55
Nd ₂ O ₃	10.6	0 - 55

* Glass additives italicized.

** CeO₂ and ThO₂ used as surrogates.

*** Nd₂O₃ and La₂O₃ tested as surrogates.

A small fraction of the lanthanum will come from the frit.

Glass Processing - Glass Durability

Durability tests were performed on several simulated Am/Cm glasses to ensure that the glasses would meet the durability specification for high-level waste. The Product Consistency Test (PCT), the specified test for high-level waste glass, was performed on a composition with over 50 weight percent lanthanide oxides [26]. The composition of this glass is shown in Table 4.

The PCT experimental results indicate this glass is extremely durable. The leachate solutions contained ~60ppb B. The glass used as a standard for high-level waste glass (SRS- EA) releases ~580ppm B under PCT conditions [27]. After correcting for the boron content in the glass compositions, the simulated Am/Cm glass is approximately 4000 times more durable than SRS-EA glass.

A second test was performed on 4 glasses designed to be less durable. These glasses had approximately twice the boron content (the most soluble component of the Am/Cm glasses) and half of the alumina content of the glass shown in Table 4. Due to the relative boron oxide and alumina content, these glasses should represent "worst case" compositions for relative durability. These compositions are shown in Table 5.

The PCT experimental results indicate these glasses are also considerably more durable than SRS-EA glass. The leachate solutions contained ~500ppb B. After correcting for the boron content in these glasses, they are approximately 1000X more durable than SRS-EA glass. These results indicate there will be no problem meeting the high-level waste glass durability specification. A detailed comparison between the Am/Cm glass and other pertinent high-level waste criteria is given in Reference 28.

Table 4. Analyzed Composition of a Simulated Am/Cm Glass (in oxide weight percent). Average of 2 Analyses.

<u>Component</u>	<u>Weight %</u>
La ₂ O ₃	17.4
Nd ₂ O ₃	31.3
CeO ₂	3.5
SiO ₂	21.5
B ₂ O ₃	4.4
BaO	1.8
Al ₂ O ₃	12.9
PbO	7.2

Table 5. As-Batched Composition of 4 Glasses with Varying Actinide Content (in oxide weight percent).

<u>Component</u>	<u>Glass #2</u>	<u>Glass #5</u>	<u>Glass #6</u>	<u>Glass #8</u>
La ₂ O ₃	19.8	8.3	3.6	1.2
Nd ₂ O ₃	33.5	34.3	34.6	34.8
ThO ₂	1.9	11.6	15.6	17.6
SiO ₂	23.5	24.0	24.3	24.4
B ₂ O ₃	7.6	7.8	7.9	7.9
BaO	2.1	2.1	2.1	2.1
Al ₂ O ₃	3.5	3.5	3.6	3.6
PbO	8.1	8.3	8.4	8.4

Process Equipment - Melter

Vitrification of the Tank 17.1 material will differ from the DWPF process for several reasons. They include:

- 1) small amount of glass to be processed (300kg),
- 2) intense heat generated by alpha decay,
- 3) concentrated nitric acid feed stream,
- 4) isotopic value of the Am and Cm.

As the 7.5cm diameter glass log will heat itself to $\sim 300^{\circ}\text{C}$, a small melter is essential, lest the glass melt itself (and potentially the melter). Likewise, any glass that remains in the melter will constitute both high-level waste and lost product. The nitric acid feed will generate highly corrosive, acidic, off-gas so that a fairly involved offgas system will be required. Another major difference is that this program will be performed in a facility not designed for vitrification. The equipment must be designed to fit on canyon process racks, which also limits the size. Each of the above factors, therefore, favor a small melter which can be easily drained.

Based on the above criteria, a bushing melter was chosen as the best type of melter available. Bushings are commonly used in the commercial glass fiber industry to produce up to 50kg per hour [29]. Bushings are essentially flat rectangular platinum-rhodium (Pt/Rh) alloy box shaped melters [29]. They are resistance heated by passing electric current through the Pt/Rh alloy. The Pt/Rh, in turn, heats the glass by conduction. The molten glass is emptied from the bushing via a bottom drain. The flow can be controlled by raising or lowering the melt viscosity by adjusting the melt temperature.

Bushings have several advantages over other common melters in terms of process and chemical "robustness":

- 1) Bushings can be cooled to room temperature and then restarted,
- 2) They are chemically inert due to the Pt/Rh alloy,
- 3) The normal commercial bushings have a geometrically favorable "slab tank" design. This is unique to among common production vitrification units.

The bushing design, power supply diagram and approximate materials cost are shown in Appendix 3.

Process Operations - F-Canyon and MPPF

The preceding sections and their Appendices described the preliminary process (and limits) for vitrifying Tank 17.1 material. This section outlines the various operations that will be performed in Section 17 of F-Canyon and the MPPF. An attached flowsheet provides a schematic representation of the Am/Cm vitrification effort within 221-F and MPPF.

1) Oxalate Precipitation

Oxalic acid will be added to Tank 17.1 to precipitate the actinides and lanthanides. Soluble components will be removed by transferring the solution to Tank 17.2. This waste solution will be then prepared and released to the Tank Farm.

Location: Tank's 17.1 and 17.2, Section 17 F-Canyon

2) Actinide/Lanthanide Re-dissolution

The precipitates will be redissolved in nitric acid. A previously developed flowsheet utilized Mn^{2+} to complex the oxalate. Other oxalate flowsheets have been developed and will be evaluated more thoroughly.

Location: Tank 17.1, Section 17 F-Canyon

3) Evaporation / Steam Stripping

Solution from Tank 17.1 will be transferred to Tank 17.3E. The solution will be evaporated and steam stripped to approximately 100g/L solids content and 1N nitric acid. The concentrated solution will be sampled for solids and acid analyses and then transferred to a Feed Tank in the MPPF.

Location: Tank 17.E, Section 17 F-Canyon
Rack 4 or 5, MPPF

4) Melter Feed / Vitrification

Samples will be taken for weight percent solids and acid analyses. The solution will then be gravity fed into the bushing melter. The glass frit will be added as a separate stream. The frit will be in the form of small marbles (0.3-0.5cm diameter) to prevent pluggages. The marbles and Tank 17.1 material will have a residence time of 6-10 hours to ensure complete fusion.

Location: Rack 4 or 5, MPPF

5) Canister Fill / Changeout

The melter will produce approximately 1-2kg glass per hour. The 1.6L canisters will be filled in approximately 5 hours. The canisters will be on a turntable; removal of the canister can best be accomplished by a combination of crane and manipulator functions.

Location: MPPF

6) Canister Closure

This was not specifically addressed by this program. However, a small version of the DWPF canister welder will be recommended.

Location: MPPF

7) Canister Decontamination

This was not specifically addressed by this program. Equipment Engineering Department personnel have suggested a "bagless port" decontamination technique [30].

Location: To be determined.

8) Process Off-gas Treatment

Process off-gas will be drawn from the melter and treated in a manner similar to DWPF off-gas. Much of the generated NO_x can be removed by a knock-out pot (quencher). Other effluent will be sent through the MPPF HEPA filter and mixed with other "hot" canyon off-gas.

Location: MPPF, F-Canyon

Am/Cm Recovery from Glass

The studies were performed to 1) determine dissolution techniques for Tank 17.1 glass analysis to assist process development and process control and 2) develop an Am/Cm recovery technique for ORNL.

The simulated Am/Cm glass composition shown in Table 4 (equivalent to over 50 weight percent Tank 17.1 material) was successfully dissolved by a variation of a method developed for DWPF. The glass was fused with Na_2O_2 and the mixture was dissolved in HNO_3 . The cation constituents of the glass were then determined by Inductively Coupled Plasma - Emission Spectroscopy.

The shielded cells at ORNL are stainless steel lined, so aqua regia or nitric/hydrofluoric combinations are not desirable. Americium and curium are soluble in concentrated nitric acid. Likewise, nitric acid is an effective solvent for all of the glass bead components except for silica. Accordingly, a nitric acid extraction technique was performed on several samples of the simulated Tank 17.1 glass composition shown in Table 4 to demonstrate a potential method for ORNL to recover the Am/Cm from the glass. The four steps to the extraction technique follow:

- 1) 0.25g crushed glass and 5ml concentrated (16M) HNO_3 were combined in a centrifuge tube,
- 2) Duplicate samples were heated in a microwave oven to 115°C ,
- 3) The samples were held at temperature for times ranging between 15 minutes and 4 hours and then allowed to cool to ambient,
- 4) The nitric acid solution was sampled by pipette and the dissolved glass component concentrations were measured by ICP-ES.

The analytical ICP-ES results from the nitric acid extraction were compared with the results of the Na_2O_2 dissolution to determine the effectiveness of the extraction technique. Americium and curium are represented by neodymium and lanthanum. Other Tank 17.1 actinides plutonium and uranium are represented by cerium.

The analytical results indicate that Nd, La, and Ce are all successfully recovered from the glass by the nitric acid extraction. After fifteen minutes, approximately 60% of the lanthanide in the glass has been extracted. The lanthanide concentrations in solution steadily increase with time. After 2 hours, effectively all of the lanthanide is in solution. Figure 1 is a plot of the fraction of each element extracted as a function of reaction time.

The silicon response is also shown in Figure 1. Approximately 40% of the silicon in the glass is in solution after 15 minutes. Unlike the lanthanides, however, the silicon content steadily decreases with increasing reaction time. As the glass is attacked, the soluble silica is initially concentrated well above the equilibrium value. This is referred to as supersaturation [31]. As reaction time increases, the silica begins to precipitate and approach equilibrium concentrations in solution. After 4 hours, almost no silicon remains in solution. Instead, a fine white precipitate, probably chalcedony, is observed in the reaction vessel. These data demonstrate the slow reaction kinetics common to silicates [32]. This behavior is consistent with that displayed by samples pulled from Tank 17.1 [33]. Initially, the samples were clear with no visible solids. After sitting for several days, however, fine, white silicate precipitates were observed [33].

Figure 1. Efficiency of Nitric Acid Extraction Technique for Recovery of Am and Cm from Simulated Tank 17.1 Glass. Tank 17.1 Components La and Nd Represent Am and Cm. Cerium Represents the Actinides Pu and U.

Besides silica, all of the glass frit components are soluble in nitric acid. The extraction of glass components Pb, Al, and Ba is displayed in Figure 2. All three elements have a response identical to that of La. Likewise, the extraction of boron from the glass closely mirrors that of the lanthanides. This is displayed in Figure 3. These data indicate that the entire glass is being attacked by the nitric acid extraction technique.

Based on the extraction test results, it appears that the Am and Cm can be successfully extracted from the glass by ORNL using the existing stainless steel lined "hot" cells. It is concluded that longer (several hour) reaction times be used to both 1) optimize the effectiveness of the extraction and 2) precipitate the silica from the nitric acid. Additional studies will be performed to determine if the nitric acid technique can be used on monolithic pieces of glass, which would remove the need for ORNL to crush the glass prior to the extraction.

Figure 2. Efficiency of Nitric Acid Extraction Technique on Dissolution of Glass Bead Components Al, Ba, and Pb from Simulated Tank 17.1 Glass. This Demonstrates the Completeness of the Nitric Acid Attack.

Figure 3. Efficiency of Nitric Acid Extraction Technique on the Glass Bead Component Boron. This Demonstrates the Completeness of the Nitric Acid Attack on the Glass Matrix.

Conclusions

Process evaluation and research and development work was performed to determine if the Tank 17.1 Am/Cm solution could be vitrified rather than discarded as a waste to the Tank Farm. These investigation can be summarized as follows:

- 1) A commercial glass composition that can accept over 40 weight percent Tank 17.1 oxides was identified. The high solubility of Tank 17.1 oxides in the glass reduces the total amount of Am/Cm glass product to ~300 kilograms.
- 2) Heat transfer calculations performed on this glass composition indicate it will have a maximum centerline temperature 275°C. This is well under the softening temperature of the Am/Cm glass.
- 3) A commercial "bushing" melter can be readily adapted to process the glass bead and nitric acid feed streams into the Am/Cm glass product. Bushings are constructed of chemically durable platinum alloys and have a "slab tank" geometry. These features make the bushing design highly favorable for processing nitric acid and/or fissile material.
- 4) Chemical process modeling indicates that a 1-2 kilogram per hour glass production rate is attainable without exceeding any NO_x generation criteria. The modeling work identifies 50 to 100g/l dissolved solids in the nitric acid feed to be optimal.
- 5) All feed processing and glass production can be performed in Section 17 of F-Canyon and the Multi-Purpose Processing Facility (MPPF). The canyon processes (oxalate precipitation and steam stripping) have been previously outlined for F-Canyon operations. MPPF is the only existing facility with both the required radiation shielding and the manipulator access desired for the vitrification process.
- 6) A nitric acid extraction technique developed to extract Am and Cm from the glass. Complete recovery of Am and Cm surrogates (Nd and La) was demonstrated.

It is concluded that vitrification is a viable disposition option for the Tank 17.1 Am/Cm solution. The nitric acid solution containing the Am/Cm (and associated lanthanides and actinides) can be mixed with glass beads and melted into a highly durable borosilicate glass for shipment to ORNL. The Am/Cm can then be recovered from the glass by a chemical extraction process compatible with stainless steel lined hot cells.

Future Work

Additional studies are underway to determine the effects of Cm²⁴⁴ radiolysis on ten year old glasses stored in SRTC. Off-gas and bushing melter development work is also underway. Procurement of a bushing at SRTC is expected by the end of FY '94. Ion-exchange actinide/lanthanide separations studies performed by Chemical Technology Section will also be evaluated.

Acknowledgments

The authors gratefully appreciate work from the following people: Beverly Burch, Harry Jackson, Miriam Cooley and Loretta Farrow of ADS, S. J. Hensel and J. W. Jerrel of Scientific Computations, and N. E. Bibler, Deddie Marsh, C. J. Crawford, D. C. Beam, Andy Foreman, and Mike Lee of DWPT. Ray Kroger and Jim Lengel of NMPD were especially helpful in providing operations knowledge and blueprints on the MPPF.

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Appendix 1

Preliminary Flowsheet Calculations for Am/Cm Vitrification Project (U)

**WESTINGHOUSE SAVANNAH RIVER COMPANY
SAVANNAH RIVER TECHNOLOGY CENTER**

SRTC-PMC-94-0035

Keywords: Actinides, Vitrification,
Flowsheet Calculations,
NO_x Emission

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May 10, 1994

MEMORANDUM

To: W. G. Ramsey, 773-A

From: A. S. Choi, 704-1T *ASC*

**PRELIMINARY FLOWSHEET CALCULATIONS FOR
AM/CM VITRIFICATION PROJECT (U)**

SUMMARY

This report describes the preliminary results of the overall mass balance analysis for the proposed vitrification of actinides currently stored in the Tank 17.1 in F-Canyon. The primary focus of this scoping analysis was to define the operating envelopes for the key process variables that will not require too large a melter or an off-gas treatment system to fit into the existing racks in the Multi-Purpose Processing Facility (MPPF).

Calculations showed that if the maximum acceptable off-gas flow rate is limited to less than 45 kg/hr based on the dimensions of the MPPF racks, the solids loading of about 60 g/liter of feed appears to be the minimum for glass production rates of less than 2 kg/hr. At a given solids loading and a glass production rate, the NO_x emission increases, as the free acidity in the feed is increased. The maximum NO_x emission during the proposed vitrification is estimated to be about 9 kg/hr at an acidity of 5 molar HNO₃ and the minimum solids loading of 60 g/liter. Based on the inventory data for the Tank 17.1, the total glass production is estimated to be 275 kg at 42 wt% waste oxides loading.

INTRODUCTION

It is estimated that the Tank 17.1 contains 4,000 gallons of highly radioactive liquid at about 7 wt% total solids and less than 0.2 wt% insoluble solids. As shown in Table 1, the analytical data for the samples taken from the Tank 17.1 show that the solution contains 25 kg of actinides and 74.2 kg of lanthanides in a highly acidic medium at 4.3 molar HNO_3 .^{1,2} The chemical composition of the tank constituents shown in Table 2 was constructed by combining all the minor anions with the major cations first, mainly Fe and Al, before the nitrate salts were formed from the remaining cations.

During the pretreatment step, all the actinides and lanthanides will be separated from the solution by the oxalic acid precipitation followed by steam stripping. The precipitates of actinides and lanthanides are then redissolved in nitric acid and fed to the melter along with the glass forming frit. The glass thus formed is poured into canisters and stored safely until they are recovered for a later use. The purpose of this scoping study was two-fold; (1) to develop a mass balance framework for the vitrification step, and (2) to perform a sensitivity analysis to determine the effects of the major operating parameters on the melter and off-gas treatment system design.

DISCUSSION

Based on the feed pretreatment step described above and the low insoluble solids content in the Tank 17.1, the melter feed will contain only the salts of actinides and lanthanides in a nitric acid solution, as shown in the three right-hand-side blocks of Table 2. So, there are two major feed characteristics that could impact the design of the melter and off-gas system; solids loading and acidity. The solids loading together with the glass production rate will impact the design of the off-gas treatment system, whereas the acidity of the feed will directly impact the NO_x emission.

Waste Oxides Loading in Glass

The total volume of glass produced and thus the number of canisters required are directly determined by the waste oxides loading in glass. Throughout this study, the waste oxides loading was fixed at 42 wt% based on the results of an earlier study.³ At this waste loading, the total glass production is estimated to be 274 kg or 0.055 m^3 based on the theoretical composition of waste oxides in glass shown in Table 3.

Solids Loading in Feed

Here, the solids loading in the feed is defined as the sum of all species except H_2O and free acid. As shown in Table 4, the solids loading in the feed was varied from 1 to 100 g/liter at two different glass production rates of 1 and 2 kg/hr during this study. As expected, Cases 1 through 8 show that the off-gas flow rate increases linearly as the solids loading decreases at a constant glass production rate. At a

given solids loading, doubling the glass production rate will reduce the off-gas flow by exactly 50 %. So, if the maximum acceptable off-gas flow rate is limited to less than 45 kg/hr based on the dimensions of each MPPF rack, the off-gas flow will exceed this maximum, when the solids loading falls below 60 g/liter at a glass production rate of 2 kg/hr or less.

This means that at a solids loading of 60 g/liter, the thermal capacity of the melter should be large enough to sustain a melt rate of 2 kg/hr and also to continuously vaporize and superheat nearly 45 kg/hr of H₂O. When the glass production rate is reduced to 1 kg/hr, the solids loading can be reduced to as low as 40 g/liter without exceeding the maximum off-gas flow rate.

Acidity of Feed

At given solids loading and glass production rate, the change in the free acidity, i.e., molar concentration of HNO₃ in the feed, practically has no impact on the off-gas flow rate. However, in Cases 7 and 16, a five-fold increase in acidity from 1 to 5 molar HNO₃ resulted in a 3.5 times increase in the NO_x emission from 1.4 to 4.9 kg/hr at a solids loading of 5 g/liter and a glass production rate of 1 kg/hr. When the solids loading was increased from 10 to 100 g/liter at 1 molar HNO₃, the NO_x emission was reduced by a factor of 5.3 regardless of the glass production rate. As expected, doubling the glass production rate doubled the NO_x emission. The maximum NO_x emission during the proposed actinides vitrification is estimated to be about 9 kg/hr at 5 molar HNO₃, when the solids loading is at its minimum, as shown in Case 24.

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Table 1. Tank 17.1 Analytical Data from F-Area / SRTC Labs

Total Liquid Volume in Tank 17.1 = 1.514E+04 liters

Ionic Distributions

	Conc (M)	Equiv (M)
Anions		
NO2	1.800E-02	1.800E-02
NO3	4.820E+00	4.820E+00
free OH	0.000E+00	0.000E+00
SO4	1.000E-01	2.000E-01
C2O4	1.400E-02	2.800E-02
Al(OH)4	0.000E+00	0.000E+00
F	1.100E-02	1.100E-02
Cl	2.800E-02	2.800E-02
CO3 *	-5.185E-03	-1.037E-02
PO4	0.013	0.039
Total	4.998E+00	5.134E+00
Cations		
	g/liter	moles/liter
Fe	4.950E+00	8.863E-02
Al	1.070E+00	3.866E-02
Na	6.720E-01	2.923E-02
Cr	2.860E-01	5.600E-03
Mn	2.370E-01	4.314E-03
Ni	2.280E-01	3.883E-03
K	2.150E-01	5.499E-03
Zr	4.000E-03	4.385E-05
B	1.020E-02	9.436E-04
Ca	8.600E-02	1.647E-03
Si	1.800E-02	6.409E-04
Zn	2.100E-02	3.212E-04
Free Acids		4.315E+00
Other Acids		1.150E-01
La/Ac Equivalents		2.331E-01
Total Cation	7.672E+00	
Total Equivalent Cation =		5.134E+00

* CO3 concentration adjusted to balance charge.

Lanthanides

	AW	g/liter	gmole/liter	g (total)
La	138.9	7.000E-01	5.040E-03	1.060E+04
Ce	140.12	8.000E-01	5.709E-03	1.212E+04
Pr	140.91	8.000E-01	5.677E-03	1.212E+04
Nd	144.24	1.700E+00	1.179E-02	2.575E+04
Sm	150.4	4.000E-01	2.660E-03	6.058E+03
Eu	151.96	8.000E-02	5.265E-04	1.212E+03
Gd	157.2	2.000E-01	1.272E-03	3.028E+03
Tb	158.9254	3.000E-02	1.888E-04	4.543E+02
Dy	162.5	3.000E-02	1.846E-04	4.543E+02
Ho	164.93	3.000E-02	1.819E-04	4.543E+02
Er	167.26	3.000E-02	1.794E-04	4.543E+02
Tm	168.9342	4.000E-02	2.388E-04	6.058E+02
Yb	173.04	3.000E-02	1.734E-04	4.543E+02
Lu	174.94	3.000E-02	1.716E-04	4.543E+02
Total		4.800E+00	3.399E-02	7.421E+04

Actinides

	AW	g (total)
Am-241	241.05884	4.580E+02
Am-242m	242.059	2.030E+00
Am-243	243.06139	9.700E+03
Cm-244	244.06277	2.560E+03
Cm-245	245.06551	9.780E+01
Cm-246	246.06725	8.520E+01
Cm-247	247.07038	1.370E+00
Np-237	237.04819	2.590E+01
Np-239	239.0529	1.570E-02
Pu-238	238.04958	1.450E+02
Pu-239	239.05218	6.220E+01
Pu-240	240.05383	2.160E+03
Pu-241	241.0566	1.450E+00
Pu-242	242.05877	1.240E+02
U-234	234.05098	1.930E+01
U-235	235.04394	1.880E+02
U-236	236.04558	3.500E+00
U-238	238.05082	9.420E+03
Total		2.505E+04

Eu-154	153.9224	3.560E+01
Ce-137	136.9068	2.080E+01

Table 2. Assumed Salt Compositions in Tank 17.1

Others				
	MW	g (total)	gmole	Conc (M)
Fe ₂ (SO ₄) ₃	399.88	2.026E+05	5.067E+02	3.346E-02
Fe(NO ₃) ₃	241.85	2.354E+04	9.732E+01	6.426E-03
Fe(NO ₂) ₃	193.65	1.756E+04	9.060E+01	5.983E-03
FeCl ₃	162.21	2.286E+04	1.409E+02	9.306E-03
Al(NO ₃) ₃	212.98	7.418E+04	3.483E+02	2.300E-02
AlPO ₄	121.86	2.417E+04	1.982E+02	1.309E-02
AlF ₃	45.98	2.485E+03	5.405E+01	3.569E-03
NaNO ₃	84.99	1.580E+03	1.859E+01	1.228E-03
NaNO ₂	68.99	0.000E+00	0.000E+00	0.000E+00
Na ₂ C ₂ O ₄	133.98	2.841E+04	2.120E+02	1.400E-02
Cr(NO ₃) ₃	237.998	1.983E+04	8.330E+01	5.500E-03
Mn(NO ₃) ₄	302.84	1.979E+04	6.533E+01	4.314E-03
Ni(NO ₃) ₂	182.71	1.075E+04	5.882E+01	3.883E-03
KNO ₃	101.1	8.419E+03	8.328E+01	5.488E-03
Zr(NO ₃) ₃	277.22	1.841E+02	6.641E-01	4.385E-05
Bi(NO ₃) ₃	196.81	2.812E+03	1.429E+01	9.436E-04
Ca(NO ₃) ₂	164.08	4.092E+03	2.494E+01	1.647E-03
Si(NO ₃) ₄	90.0855	8.744E+02	9.706E+00	6.409E-04
Zn(NO ₃) ₂	189.38	9.212E+02	4.865E+00	3.212E-04
Total 1		4.651E+05	2.012E+03	1.328E-01
HNO ₃	63	4.117E+06	6.535E+04	4.316E+00
Other Acids	240	4.180E+06	1.742E+03	1.150E-01
Total 2		4.535E+06	6.709E+04	4.430E+00

Salt Balance			
Ac/La Salts	3.184E+05	6.191E+02	4.088E-02
Total Salts excluding HNO ₃	1.202E+06	4.373E+03	2.887E-01
NO ₃ in La/Ac =		3.531E+03	2.332E-01
NO ₃ in other salts including HNO ₃ =		6.830E+04	4.576E+00
Total NO ₃ =		7.283E+04	4.809E+00
H ₂ O	18.02	1.236E+07	

Lanthanides		
	MW	g (total)
La(NO ₃) ₆	510.9	3.899E+04
Ce(NO ₃) ₆	512.12	4.428E+04
Pr(NO ₃) ₆	512.91	4.410E+04
Nd(NO ₃) ₆	516.24	9.215E+04
Sm(NO ₃) ₆	522.4	2.104E+04
Eu(NO ₃) ₆	523.96	4.178E+03
Gd(NO ₃) ₆	529.2	1.020E+04
Tb(NO ₃) ₆	530.9254	1.518E+03
Dy(NO ₃) ₆	534.5	1.494E+03
Ho(NO ₃) ₆	536.93	1.479E+03
Er(NO ₃) ₆	539.26	1.465E+03
Tm(NO ₃) ₆	540.9342	1.940E+03
Yb(NO ₃) ₆	545.04	1.431E+03
Lu(NO ₃) ₆	546.94	1.420E+03
Total 3		2.657E+05

Actinides		
	MW	g (total)
Am(NO ₃) ₆	613.05684	1.165E+03
Am(NO ₃) ₆	614.059	5.150E+00
Am(NO ₃) ₆	615.06139	2.455E+04
Cm(NO ₃) ₆	616.06277	6.462E+03
Cm(NO ₃) ₆	617.06551	2.463E+02
Cm(NO ₃) ₆	618.06725	2.140E+02
Cm(NO ₃) ₆	619.07038	3.433E+00
Np(NO ₃) ₄	485.04819	5.300E+01
Np(NO ₃) ₄	487.0529	3.199E-02
Pu(NO ₃) ₄	488.004958	2.961E+02
Pu(NO ₃) ₄	487.05218	1.267E+02
Pu(NO ₃) ₄	488.05383	4.391E+03
Pu(NO ₃) ₄	489.0566	2.942E+00
Pu(NO ₃) ₄	490.05977	2.510E+02
UO ₂ (NO ₃) ₂	390.05098	3.216E+01
UO ₂ (NO ₃) ₂	391.04394	3.128E+02
UO ₂ (NO ₃) ₂	392.04558	5.813E+00
UO ₂ (NO ₃) ₂	394.05082	1.559E+04
Total 4		6.371E+04

Eu(NO ₃) ₆	525.9224	1.216E+02
CeNO ₃	198.9068	3.022E+01
Total 5		1.519E+02

Table 3. Waste Oxides Compositions in Glass

Lanthanides

Oxides	MW	g in Glass	gmolcs
La2O3	325.8	1.243E+04	3.816E+01
Ce2O3	328.24	1.419E+04	4.323E+01
Pr2O3	329.82	1.418E+04	4.289E+01
Nd2O3	336.48	3.003E+04	8.925E+01
Sm2O3	348.8	7.025E+03	2.014E+01
Eu2O3	351.92	1.403E+03	3.987E+00
Gd2O3	362.4	3.491E+03	9.634E+00
Tb2O3	365.8508	5.230E+02	1.428E+00
Dy2O3	373	5.215E+02	1.398E+00
Ho2O3	377.86	5.205E+02	1.377E+00
Er2O3	382.52	5.195E+02	1.356E+00
Tm2O3	385.8684	6.919E+02	1.793E+00
Yb2O3	394.08	5.174E+02	1.313E+00
Lu2O3	397.88	5.167E+02	1.298E+00
Total		8.656E+04	2.574E+02

Actinides

Oxides	MW	g in glass	gmolcs
Am2O3 (241)	530.11368	5.036E+02	9.500E-01
Am2O3 (242)	532.118	2.231E+00	4.193E-03
Am2O3 (243)	534.12278	1.066E+04	1.995E+01
Cm2O3 (244)	536.12554	2.812E+03	5.245E+00
Cm2O3 (245)	538.13102	1.074E+02	1.995E-01
Cm2O3 (246)	540.1345	9.351E+01	1.731E-01
Cm2O3 (247)	542.14076	1.603E+00	2.772E-03
NpO2 (237)	289.04819	2.940E+01	1.093E-01
NpO2 (239)	271.0629	1.780E-02	6.568E-05
PuO2 (238)	270.004958	1.645E+02	6.092E-01
PuO2 (239)	271.05218	7.053E+01	2.602E-01
PuO2 (240)	272.05383	2.448E+03	8.998E+00
PuO2 (241)	273.0566	1.642E+00	6.015E-03
PuO2 (242)	274.05877	1.404E+02	5.123E-01
U3O8 (234)	830.15294	2.282E+01	2.749E-02
U3O8 (235)	833.13182	2.221E+02	2.686E-01
U3O8 (236)	836.13674	4.133E+00	4.943E-03
U3O8 (238)	842.16248	1.111E+04	1.319E+01
Total		2.839E+04	5.051E+01

Eu2O3 (154)	355.8448	4.115E+01	1.156E-01
Cs2O (137)	289.8136	2.202E+01	7.596E-02
Oxides in Glass =		1.160E+05	
O Atoms in Oxides =		1.570E+04	9.810E+02
wt fraction O in Oxides =		1.365E-01	

Table 4. Results of Am/Cm Vitrification Flowsheet Case Study

	Case 1	Case 2	Case 3	Case 4	Case 5	Case 6	Case 7	Case 8	Case 9	Case 10	Case 11	Case 12
Basest:												
Feed solids content (g/liter) =	2	10	50	100	2	10	50	100	50	50	50	50
HNO ₃ conc in feed (molar) =	1	1	1	1	1	1	1	1	2	3	4	5
Glass production rate (kg/hr) =	2	2	2	2	1	1	1	1	2	2	2	2
wt% Oxide in Glass =	42	42	42	42	42	42	42	42	42	42	42	42
Calculated Results:												
Assumed feed density (g/ml) =	1.0288182	1.0344699	1.0618794	1.0943724	1.0288182	1.0344699	1.0618794	1.094372	1.0897905	1.11644321	1.1419938	1.1665688
Total slurry feed (kg) =	164377.76	33056.15	6786.402262	3497.0313	164377.76	33056.15	6786.4023	3497.031	6964.7803	7135.11603	7298.4081	7455.4641
Total slurry feed (liters) =	159773.38	31954.675	6390.935035	3195.4875	159773.38	31954.675	6390.935	3195.468	6390.935	6390.93504	6390.935	6390.935
HNO ₃ in feed (kg) =	10065.723	2013.1445	402.6289073	201.31445	10065.723	2013.1445	402.62891	201.3145	805.25781	1207.88672	1610.5156	2013.1445
H ₂ O in feed (kg) =	153992.49	30723.458	6064.226003	2976.1701	153992.49	30723.458	6064.2266	2978.17	5839.9757	5607.68255	5368.3457	5122.7728
Calculated feed density (g/ml) =	1.0288182	1.0344699	1.061879421	1.0943724	1.0288182	1.0344699	1.0618794	1.094372	1.0897905	1.11644321	1.1419938	1.1665688
Solids content in feed (wt%) =	0.1943978	0.9666787	4.708632638	9.1378676	0.1943978	0.9666787	4.7086326	9.137868	4.5880378	4.47850814	4.3783076	4.2860746
Ac/La content in feed (g/liter) =	0.6303339	3.1516696	15.7583478	31.516698	0.6303339	3.1516698	15.758348	31.5167	15.758348	15.7583478	15.758348	15.758348
Total glass produced (kg) =	273.84806	273.84806	273.8480693	273.84806	273.84806	273.84806	273.84806	273.8481	273.84806	273.848059	273.84806	273.84806
Total melter run time (hr) =	136.92403	136.92403	136.9240297	136.92403	136.92403	136.92403	136.92403	136.92403	136.92403	136.92403	136.92403	136.92403
Feed rate (kg/hr) =	1200.6034	241.41864	49.56326715	25.539839	600.25168	120.70882	24.781834	12.76997	50.966019	52.1100354	63.30261	54.44964
Feed rate (liter/hr) =	1166.8761	233.37522	46.67504347	23.337522	583.43904	116.68761	23.337522	11.66876	46.675043	46.6750435	46.675043	46.675043
% calcination of Ac/La slurry =	35.993539	35.993539	35.99353906	35.993539	35.993539	35.993539	35.993539	35.99354	35.993539	35.9935391	35.993539	35.993539
Off-gas: Total (kg/hr) =	1199.6634	240.67864	48.72326715	24.698939	599.83168	120.26982	24.361634	12.34887	50.026019	51.2700354	52.46261	53.60964
H ₂ O (kg/hr) =	1135.17	226.48596	44.70952838	21.848184	567.58488	113.24298	22.354765	10.9731	43.492298	42.2163264	40.888915	39.51596
NO _x (kg/hr) =	45.320852	9.847819	2.753212345	1.8683865	22.660426	4.9239095	1.3766062	0.933193	4.526864	6.30051565	8.0741673	9.847819
O ₂ (kg/hr) =	19.183561	4.2475472	1.360344434	0.8859441	9.5917808	2.1237736	0.6301722	0.443472	2.0071461	2.75394582	3.5007465	4.2475472

Note that the assumed feed density should be set such that it matches the calculated feed density.

Table 4. Results of Am/Cm Vitrification Flowsheet Case Study (Continued)

	Case 13	Case 14	Case 15	Case 16	Case 17	Case 18	Case 19	Case 20	Case 21	Case 22	Case 23	Case 24
Basic:												
Feed solids content (g/kg) =	60	50	50	50	50	50	50	50	50	50	50	50
HNO ₃ conc in feed (molar) =	2	3	4	5	1	2	3	4	5	1	3	5
Glass production rate (kg/hr) =	1	1	1	1	1.5	1.5	1.5	1.5	1.5	2	2	2
wt% Oxide in Glass =	42	42	42	42	42	42	42	42	42	42	42	42
Calculated Results:												
Assumed feed density (g/ml) =	1.0897905	1.1164432	1.141993789	1.1665686	1.0618794	1.0897905	1.1164432	1.141994	1.1665686	1.08852607	1.1225094	1.1721839
Total slurry feed (kg) =	6964.7803	7135.116	7298.408117	7455.4641	6786.4023	6964.7803	7135.116	7298.408	7455.4641	5690.73391	5978.2373	6242.7928
Total slurry feed (liters) =	6390.935	6390.935	6390.935036	6390.935	6390.935	6390.935	6390.935	6390.935	6390.935	5325.7792	5325.7792	5325.7792
HNO ₃ in feed (kg) =	1207.8867	1610.516629	2013.1445	402.62891	805.25781	1207.8867	1610.516	2013.1445	335.524089	1006.5723	1677.6204	1677.6204
H ₂ O in feed (kg) =	5007.9767	5607.6825	5385.346736	5122.7728	4064.2266	5839.8757	5607.6825	5368.348	5122.7728	5035.66307	4652.1182	4245.6254
Calculated feed density (g/ml) =	1.0897905	1.1164432	1.141993789	1.1665686	1.0618794	1.0897905	1.1164432	1.141994	1.1665686	1.08852607	1.1225094	1.172184
Solids content in feed (wt%) =	4.5880378	4.4785092	4.378307525	4.2860746	4.7086326	4.5880378	4.4785081	4.378307	4.2860746	5.61621162	5.3451668	5.1186508
Ac/La content in feed (g/liter) =	15.758348	15.758348	15.7583478	15.758348	15.758348	15.758348	15.758348	15.75835	15.758348	18.9100174	18.910017	18.910017
Total glass produced (kg) =	273.84806	273.84806	273.8480593	273.84806	273.84806	273.84806	273.84806	273.8481	273.84806	273.848059	273.84806	273.84806
Total melter run time (hr) =	273.84806	273.84806	273.8480593	273.84806	182.56537	182.56537	182.56537	182.5654	182.56537	136.92403	136.92403	136.92403
Feed rate (kg/hr) =	25.433009	26.055017	26.65130487	27.22482	37.17245	38.148514	39.082527	39.97696	40.83723	41.5612508	43.66098	45.593112
Feed rate (liter/hr) =	23.337522	23.337522	23.33752173	23.337522	35.006283	35.006283	35.006283	35.00628	35.006283	38.8958696	38.89587	38.89587
% calcination of Ac/La slurry =	35.993539	35.993539	35.99353906	35.993539	35.993539	35.993539	35.993539	35.99354	35.993539	35.993539	35.993539	35.993539
Off-gas:												
Total (kg/hr) =	25.013009	25.835017	26.23130487	26.80482	36.54245	37.519514	38.452527	39.34696	40.20723	40.7212506	42.82098	44.753112
H ₂ O (kg/hr) =	21.746146	21.106153	20.44445759	19.75788	33.532147	32.618222	31.662245	30.65669	29.63697	37.1275105	35.027263	32.75942
NO _x (kg/hr) =	2.263432	3.1502578	4.03708395	4.8239095	2.0649093	3.395148	4.7253867	6.055825	7.3858642	2.45780374	5.4136898	8.3697769
O ₂ (kg/hr) =	1.0035720	1.3769729	1.76037326	2.1237738	0.9452583	1.5053588	2.0654594	2.62556	3.1856604	1.13587765	2.3805455	3.6252133

Note that the assumed feed density should be set such that it matches the calculated feed density.

Appendix 2

Temperatures for Tank 17.1 Wasteforms: Part III

WESTINGHOUSE SAVANNAH RIVER COMPANY
SAVANNAH RIVER TECHNOLOGY CENTER

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SRT-GTC-94-00027

March 25, 1994

TO: W. G. Ramsey, 773-A

FROM: B. J. Hardy, 773-41A
J. W. Jerrell 773-42A
S. J. Hensel, 773-42A

CC: M. J. Plodinec, 773-A
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TEMPERATURES FOR TANK 17.1 WASTEFORMS: PART III

INTRODUCTION

As requested by W. G. Ramsey, maximum glass temperatures were calculated for smaller wasteforms than in Parts I and II and for an extended range of oxide loadings.^{1,2} As in Parts I and II, the general purpose heat transfer code P/THERMAL was used for the glass temperature calculations. It must be noted that P/THERMAL has not been qualified according to the standards set in the E-7 Manual. Hence the results of this analysis can be used for scoping purposes only.

The authors understand that this task is described in an existing task plan.

DESCRIPTION OF ANALYSIS

The steady state centerline temperatures were conservatively calculated for glass wasteforms produced in isotopic containment experiments with material from Tank 17.1.

Wasteform Dimensions and Properties

The dimensions and material properties of the wasteforms are as follows:

1. The wasteforms are right circular cylinders having an overall length of 14.76 in. and 2.95 in. diameter. The wasteforms are composed of glass contained in a 304L stainless steel sheath. The sheath is 1/8 in. thick at the sides and 1/4 in. thick at each end (see Figure 1). In the previous analyses the wasteforms were

conservatively assumed to be infinitely long.^{1,2} However, in this analysis the actual wastefrom dimensions were used.

2. The thermal conductivity of the glass and waste was assumed to be that of pure Pyrex glass (obtained from the P/THERMAL data file) and the specific gravity of the glass and waste was assumed to be 5.5.³ The thermal conductivity and density of the steel sheath was that of 304L stainless steel.

Thermal radiation was neglected in the heat transfer calculations. Natural convection to stagnant air from a vertical free standing cylinder was assumed as a boundary condition for the wastefrom. The convective heat transfer coefficients used in the analysis were as follows:

$$\begin{aligned} \text{side of canister} \quad h &= 0.19 \Delta T^{1/3} \frac{\text{Btu}}{\text{ft}^2 \text{ } ^\circ\text{F hr}} \\ \text{top of canister} \quad h &= 0.22 \Delta T^{1/3} \frac{\text{Btu}}{\text{ft}^2 \text{ } ^\circ\text{F hr}} \\ \text{bottom of canister} \quad h &= 0.12 \left(\frac{\Delta T}{D} \right)^{1/4} \frac{\text{Btu}}{\text{ft}^2 \text{ } ^\circ\text{F hr}} \end{aligned}$$

where $\Delta T = (T_{\text{surface}} - T_{\infty})$
D = Canister diameter

The ambient temperature (T_{∞}) was assumed to be 100°F in these calculations.

Power Density for the Waste Glass

It was assumed that the heat source in the wastefrom was uniformly distributed in the glass, due to isotopic decay and deposited locally at the site of the decay. Spontaneous fission was neglected. The nominal total decay energy was calculated from the specific activity, the decay energy and the concentration of the isotopes in the wastefrom. The power density for a particular isotope in the wastefrom was calculated as follows:

Let

$$\frac{\rho_{wo}}{\rho_t} = f$$

1

where: ρ_{wo} = Density of the oxide in the waste glass

ρ_t = Total density (glass and oxide) in the waste glass = $\rho_{wo} + \rho_{wg}$

ρ_{wg} = Density of pure glass in the waste glass

f = Mass fraction of oxide in the waste glass

Now,

$$\Pi_i = \rho_{wi} \alpha_i \bar{E}_i = \frac{\rho_{wi}}{\rho_t} \rho_t \alpha_i \bar{E}_i = \frac{\rho_{wi}}{\rho_{wo}} \frac{\rho_{wo}}{\rho_t} \rho_t \alpha_i \bar{E}_i = \frac{\rho_i}{\rho_o} \frac{\rho_{wo}}{\rho_t} \rho_t \alpha_i \bar{E}_i \quad 2$$

where Π_i = Power density of isotope in the waste glass

ρ_{wi} = Density of isotope i in the waste glass

ρ_i = Density of isotope i in the oxide

ρ_o = Density of the oxide

α_i = Specific activity of isotope i

$\bar{E}_i$ = Average energy per disintegration for isotope i

From Eq. (1) and the assumption that $\rho_t = \rho_g$ = (density of pure glass) Eq. (2) becomes

$$\Pi_i = \frac{\rho_i}{\rho_o} f \rho_g \alpha_i \bar{E}_i \quad 3$$

where: ρ_i = Density of isotope i in the oxide

Note that in Eq. (3) $\frac{\rho_i}{\rho_o}$ is the mass fraction of isotope i in the oxide and $f \left(= \frac{\rho_{wo}}{\rho_t} \right)$ is the mass fraction of oxide in the waste glass.

The total power density in the waste glass is given by

$$(\text{Total Power density}) = \sum_i \Pi_i \quad 4$$

Assumed Isotopics

The specific activities were the more conservative of those from References 3 and 4, the data for the oxide concentrations were obtained from Reference 3, and the decay energies were obtained from Reference 4. For conversion electrons the largest value of energy was used to calculate the energy deposition rate. All data and isotopic calculations are contained in References 5 and 6.

It was assumed that the oxides of Fe, Al, Mn, Ni and Cr were removed from the Tank 17.1 oxide before processing into waste glass. This is Case 2 of the isotopics in Reference 2. The power densities were calculated for 5, 10, 25, 30 40 and 50 weight percents of the total oxide in the waste glass. From Eq. 3 it can be seen that the power density is directly proportional to the mass fraction of the oxide in the glass. Table 1 gives the power densities for the six weight percents described above. The individual contributions of each of the isotopes to the total waste glass power density, for this oxide, are given in Table 2 for 5 weight percent oxide loading.

RESULTS AND CONCLUSIONS

The results of the calculations are given in Table 3. The maximum temperatures occurred at the centerline, and at an axial position located 7.125 in. from the bottom of the wasteform.

It should be noted that the calculations were for free-standing wasteforms. The maximum temperatures would be expected to be higher for wasteforms grouped closely together or contained in a shipping cask or other enclosure.

REFERENCES

1. B. J. Hardy, S. J. Hensel. *Temperatures for Tank 17.1 Wasteforms: Part I*. Westinghouse Savannah River Company Document, SRT-GTC-94-023 (1994).
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3. Personal Communications with W. G. Ramsey (1994).
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5. B. J. Hardy. *Laboratory Notebook*. Westinghouse Savannah River Company Document, WSRC-NB-93-143, ppg 46-54 (1993).
6. S. J. Hensel. *Laboratory Notebook*. Westinghouse Savannah River Company Document, WSRC-NB-93-179 (1993).

Figure 1. Schematic of outer surface of wasteform.

TABLE 1
Power Densities in (W/ml) for Waste Glass

Oxide Weight Percent	Power Density (W/ml)
5	1.69×10^{-2}
10	3.38×10^{-2}
25	8.46×10^{-2}
30	1.02×10^{-1}
40	1.35×10^{-1}
50	1.69×10^{-1}

TABLE 2
Spreadsheet Calculation for Case 2 Power Density
for 5 Weight Percent Oxide in the Glass

Oxide has Fe/Al/Mn/Ni/Cr Removed

wt. fr. oxide	Waste Glass dens	Tot. Mass Oxide
0.05	5.50 g/cm ³	1.24E+05 g

ALPHA DECAY

Isotope	Specific Activity (dpm/g)	Mass of Isotope (g)	Mass Fract of Isot. in Oxide	Energy & Intensity (MeV)	Avg. Energy (MeV)	Power Density (W/ml)
Am-241	7.60E+12	4.58E+02	3.69E-03	5.49(85%) 5.44(13%)	5.37E+00	1.11E-04
Am-243	3.33E+11	9.70E+03	7.82E-02	5.28(87%) 5.23(11.5%)	5.20E+00	9.93E-05
Cm-244	1.82E+14	2.56E+03	2.06E-02	5.81(77%) 5.77(23%)	5.80E+00	1.60E-02
Cm-245	2.79E+11	9.78E+01	7.89E-04	5.36(80%) 5.31(7%)	4.66E+00	7.53E-07
Cm-246	5.10E+09	8.52E+01	6.87E-04	5.39(81%) 5.34(19%)	5.38E+00	1.38E-08
Cs-137	1.93E+14	2.08E+01	1.68E-04			0.00E+00
Eu-154	6.61E+14	3.56E+01	2.87E-04			0.00E+00
Np-237	1.56E+09	2.59E+01	2.09E-04	4.78(75%) 4.65(24%)	4.70E+00	1.12E-09
Np-239	5.16E+17	1.57E-02	1.27E-07			0.00E+00
Pu-238	3.77E+13	1.45E+02	1.17E-03	5.50(72%) 5.46(28%)	5.49E+00	1.78E-04
Pu-239	1.13E+11	6.22E+01	5.02E-04	5.16(88%) 5.11(11%)	5.10E+00	2.12E-07
Pu-240	4.16E+11	2.16E+03	1.74E-02	5.19(76%) 5.12(24%)	5.17E+00	2.75E-05
Pu-241	2.05E+14	1.45E+00	1.17E-05	4.90(0.0019%) 4.85(0.0003%)	9.46E-04	1.67E-09
Pu-242	7.06E+09	1.24E+02	1.00E-03	4.9(76%) 4.86(24%)	4.89E+00	2.54E-08

BETA DECAY

Isotope	Specific Activity (dpm/g)	Mass of Isotope (g)	Mass Fract of Isot. in Oxide	Energy & Intensity (MeV)	Avg. Energy (MeV)	Power Density (W/ml)
Am-241	7.60E+12	4.58E+02	3.69E-03			0.00E+00
Am-243	3.33E+11	9.70E+03	7.82E-02			0.00E+00
Cm-244	1.82E+14	2.56E+03	2.06E-02			0.00E+00
Cm-245	2.79E+11	9.78E+01	7.89E-04			0.00E+00
Cm-246	5.10E+09	8.52E+01	6.87E-04			0.00E+00
Cs-137	1.93E+14	2.08E+01	1.68E-04	1.176(7%) 0.314(93%)	3.70E-01	8.80E-06
Eu-154	6.61E+14	3.56E+01	2.87E-04	1.85(10%) 0.87(90%)	9.70E-01	1.35E-04
Np-237	1.56E+09	2.59E+01	2.09E-04			0.00E+00
Np-239	5.16E+17	1.57E-02	1.27E-07	0.713(11%) 0.437(89%)	4.70E-01	2.25E-05
Pu-238	3.77E+13	1.45E+02	1.17E-03			0.00E+00
Pu-239	1.13E+11	6.22E+01	5.02E-04			0.00E+00
Pu-240	4.16E+11	2.16E+03	1.74E-02			0.00E+00
Pu-241	2.05E+14	1.45E+00	1.17E-05	0.021(100%)	2.10E-02	3.70E-08
Pu-242	7.06E+09	1.24E+02	1.00E-03			0.00E+00

CONVERSION ELECTRONS

Isotope	Specific Activity (dpm/g)	Mass of Isotope (g)	Mass Fract of Isot. in Oxide	Energy & Intensity (MeV)	Avg. Energy (MeV)	Power Density (W/ml)
Am-241	7.60E+12	4.58E+02	3.69E-03	0.022 0.038 0.054	5.40E-02	1.11E-06
Am-243	3.33E+11	9.70E+03	7.82E-02	0.011 0.027 0.052 0.069	6.90E-02	1.32E-06
Cm-244	1.82E+14	2.56E+03	2.06E-02	0.022 0.038	3.80E-02	1.05E-04
Cm-245	2.79E+11	9.78E+01	7.89E-04			0.00E+00
Cm-246	5.10E+09	8.52E+01	6.87E-04			0.00E+00
Ce-137	1.93E+14	2.08E+01	1.68E-04	0.624 0.656	6.56E-01	1.56E-05
Eu-154	6.61E+14	3.56E+01	2.87E-04	0.073 0.115 0.122	1.22E-01	1.70E-05
Np-237	1.56E+09	2.59E+01	2.09E-04	0.009 0.024 0.036 0.051 0.067 0.082	8.20E-02	1.96E-11
Np-239	5.16E+17	1.57E-02	1.27E-07	0.04 0.048 0.088 0.106 0.156	1.56E-01	7.48E-06
Pu-238	3.77E+13	1.45E+02	1.17E-03	0.024 0.039	3.90E-02	1.26E-06
Pu-239	1.13E+11	6.22E+01	5.02E-04	0.008 0.019 0.033 0.047	4.70E-02	1.96E-09
Pu-240	4.16E+11	2.16E+03	1.74E-02	0.026 0.04	4.00E-02	2.13E-07
Pu-241	2.05E+14	1.45E+00	1.17E-05			0.00E+00
Pu-242	7.06E+09	1.24E+02	1.00E-03			0.00E+00

GAMMA DECAY

Isotope	Specific Activity (dpm/g)	Mass of Isotope (g)	Mass Fract of Isot. in Oxide	Energy & Intensity (MeV)	Avg. Energy (MeV)	Power Density (W/ml)
Am-241	7.60E+12	4.58E+02	3.69E-03	0.06(36%) 0.101(0.04%)	2.00E-02	4.12E-07
Am-243	3.33E+11	9.70E+03	7.82E-02	0.44(4%) 0.075(50%)	6.00E-02	1.15E-06
Cm-244	1.82E+14	2.56E+03	2.06E-02	0.043(0.02%) 0.10(0.0015%)	1.01E-05	2.79E-08
Cm-245	2.79E+11	9.78E+01	7.89E-04	0.13(5%) 0.173(14%)	3.07E-02	4.96E-09
Cm-246	5.10E+09	8.52E+01	6.87E-04			0.00E+00
Ce-137	1.93E+14	2.08E+01	1.68E-04	0.662(85%)	5.63E-01	1.34E-05
Eu-154	6.61E+14	3.56E+01	2.87E-04	0.123(38%) 0.248(7%) 0.593(6%) 0.724(21%) 0.759(5%) 0.876(12%) 1.00(31%) 1.278(37%)	1.18E+00	1.64E-04 0.00E+00 0.00E+00
Np-237	1.56E+09	2.59E+01	2.09E-04	0.030(14%) 0.086(14%) 0.145(1%) 0.278(14%)	1.77E-02	4.23E-12 0.00E+00
Np-239	5.16E+17	1.57E-02	1.27E-07	0.106(23%) 0.209(4%) 0.228(12%) 0.278(14%)	9.90E-02	4.75E-06 0.00E+00
Pu-238	3.77E+13	1.45E+02	1.17E-03			0.00E+00
Pu-239	1.13E+11	6.22E+01	5.02E-04			0.00E+00
Pu-240	4.16E+11	2.16E+03	1.74E-02			0.00E+00
Pu-241	2.05E+14	1.45E+00	1.17E-05			0.00E+00
Pu-242	7.06E+09	1.24E+02	1.00E-03			0.00E+00

Total Power Density* (W/ml)

1.69E-02

TABLE 3
Maximum Glass Temperatures (°C)

Oxide Weight Percent	Maximum Temperature (°C)
5	93.3
10	132.8
25	231.1
30	261.1
40	314.4
50	366.1

Appendix 3

Bushing Melter Schematics and Cost Estimates

FRONT VIEW

Feed Inlet

Off-Gas Outlet

Melt Line

10", 25.4 cm

7.5" 19 cm

Thermocouple

12" 30 cm

1.5" 2.0 cm

0.5" 1.25 cm

1/2" Tube
I.D. = 0.45"

8.0" 20 cm

Ray Schumacher
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SIMPLIFIED SCHEMATIC BUSHING MELTER SYSTEM

Ray Schumacher
WSRC 4/27/94

Bushing SS

Glass Volume Weight etc.		CGS System		Density=5.0	
Glass Melt:	Length-cm	Width-cm	Depth-cm	Volume-cc	Weight-kg
	25	6	20	3000	15
Temperature:	1350				
Feed:	Beads	Sol'n	Total-kg/hr		
	1.2	0.8	2		3413BTU/kwh
		Vol. Sol'n-liter	Heat Vap.cal g	BTUs	KW
		8	4,944,000	19619	5.75
		Glass	Glass Fusion		
		2	675000	2679	0.78
Platinum Metal Required:		Width	Height	Sq. In.	Weight gr.
.030"	Front:	10	12.5	125	1317.50
.030"	Back:				1317.50
.030"	End:	2.36	12	28.32	298.49
.030"	End:				298.49
0.125"	Top Ear:	1.5	9	13.5	594.00
0.125"	Top Ear:				
0.125"	Bottom Ear:	1.5	3	4.5	198.00
0.125"	Bottom Ear:				
.030"	Bottom:	2.36	10	23.6	248.74
.020"	Top:	2.36	10	23.6	165.84
	Misc.				300.00
0.030"	Screen Top	4	10	40	421.60
	Total				5160.17
	Tr. Oz.				165.92
	Cost:	\$600/TrOz			\$99,553
	(EST.)				

Bushing SS

		Units
Glass Volume:	3000	cc
Glass Weight:	15	kg
Feed Rate:	2	kg/hr
Power to Dry:	5.75	kwh
Power to Melt:	0.78	kwh
Est. Heat Loss	2	kwh
Total Weight	5160.17	grams
	165.92	Tr.Oz.
Est. Cost	\$99,553	

Record Indexing — Receipt

Required with all records submitted to D&RA
Print or type in black ink.

Transmittal No.

Part A — Transmittal			
			Date 5-23-94
To: W. G. Ramsey	Location 773-A	Dept DWPT	Phone 5-8175
Items in Enclosure are required for all records.			
Keywords and other information are also required and shall be supplied as necessary to insure accurate and timely record retrieval.			
Part B — Indexing Information (Completed by Originator)			
Document ID WSRC-TR-94-0211	Sheet	Revision 0	Document Type Report
Alternate ID	Sheet	Revision	
Title Vitrification of F-Area Americium/Curium: Feasibility Study and Preliminary Process Recommendation			Classification Unclassified
Author/Originator W G RAMSEY			Location Sav. River LAB
Security Classification Unclassified			
Document Date (YYMMDD) 94-04-29			No. of Pages 44
Keywords Am/Cm, Tank 17.1, Vitrification, Bushing			
Design/Engineering Document Descriptors			
Supersedes	Sheet	Revision	Supr-DTD
Reference Document	Sheet	Revision	Supr-DTD
Equipment	Material		
System	Purchase Order Number		
Building	Elevation	Area	
Project Number	Project File Number		
Design Function	Category	Design Area	
Retention Period Permanent	Retention Period N1-434-89-8-7.5-4(1)	Retention Period N1-434-89-8-7.5-4(1)	Retention Period 1004 + 960 4169
Part C — For D&RA Use Only			
Record Location			
Transmitted By			Date
Received By			Date
Verified By			Date

Distribution Part 1 - Original, filed with record copy, Part 2 - D&RA use only, Part 3 - Returned to Sender, Part 4 - Retained by user, if needed.