

**Key Words:** Tank 48H  
Tetraphenylborate  
Cesium

## **Downstream Impacts of Tank 48H In-tank and Out-of-tank Processing Alternatives**

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## **Approvals**

## Summary

This document discusses a number of possible impacts that an in-tank or out-of-tank process may have on downstream processing facilities. This analysis was requested as part of a NETL funded task to develop processes to destroy tetraphenylborate using Fenton Chemistry (metal catalyst plus hydrogen peroxide). A meeting was held on October 14, 2003, which included experts representing the various downstream processing facilities at SRS. A summary of the meeting is attached (Attachment 1). The results of this meeting were used to develop this report.

Two processes being evaluated were funded by a grant from DOE's National Energy Technology Center. The first is an in-tank process, where the tetraphenylborate (TPB) is destroyed in Tank 48H by decreasing the pH, increasing the temperature and adding a catalyst and hydrogen peroxide (as required). After the TPB is destroyed, sodium hydroxide and sodium nitrite will be added to the tank to return the tank to conditions that minimize corrosion. The resulting slurry will be stored in a HLW tank, will likely be concentrated in the HLW evaporators and later will be fed to the Salt Waste Processing Facility. The second process is an out-of-tank Fenton process. This process will produce two streams, a high cesium stream that will be fed to DWPF and a low cesium feed that will be returned to a HLW tank with the DWPF recycle. The recycle stream may be evaporated in the HLW evaporators, and will later be fed to the Saltstone Facility or the Actinide Removal Process.

An additional two processes being evaluated were funded by WSRC's Closure Business Unit. Both are in-tank processes. In the first, thermal hydrolysis, the TPB is destroyed in Tank 48H by decreasing the pH and increasing the temperature. In the second process, thermal hydrolysis, the TPB is destroyed in Tank 48H by decreasing the pH, adding a catalyst, and increasing the temperature. After the TPB is destroyed, sodium hydroxide and sodium nitrite will be added to the tank to return the tank to conditions that minimize corrosion. The resulting slurry will be stored in a HLW tank, will likely be concentrated in the HLW evaporators and later will be fed to the Salt Waste Processing Facility.

This evaluation was designed to identify possible downstream impacts that may limit the productivity or quality of existing and proposed processing facilities, including the Salt Waste Processing Facility, Defense Waste Processing Facility, Actinide Removal Process, Tank Farms, Saltstone Facility, or the HLW evaporators. This list was compiled so that researchers and modelers could target their future work to determine whether these postulated interactions will truly impact the downstream facilities. **Table 1** summarizes the possible downstream impacts noted in this document and attempts to compare the processing alternatives. Note, a "1" signifies the best alternative while a "4" signifies the worst of the options.

Based on an assessment by the author, the out-of-tank Fenton process finishes high in most categories as it most completely destroys the organic, does not require a post processing pH and nitrite adjustment if fed directly to DWPF, and is the safest relative to flammability and processing, since processing is completed in a smaller, well designed facility. The in-tank thermal hydrolysis process scores well in many categories, is the simplest of the in-tank processing schemes, and likely would minimize corrosion to the tank compared to the other in-tank processes.

**Table 1 – Comparison of Processing Options based on Downstream Impacts to SRS Processing Facilities**

	In-tank Fenton	Out-of- tank Fenton	In-tank Thermal Hydrolysis	In-tank Catalyzed Thermal Hydrolysis
High Organic	2	1	3	4
High Sodium	2	1	2	2
High Nitrate	1	2	1	1
Flammability	2	1	3	4
Corrosion	4	1	2	3
Glass Quality	2	2	1	4
Process Safety	4	1	2	3

## Introduction

The limited volumes available to store wastes currently hamper acceleration of salt waste processing at the Savannah River Site. Tank 48H contains only 230,000 gallons of waste with a freeboard of about 1 million gallons. However, the organic contents of the waste place severe restrictions on additions or removals of this waste. Currently, personnel are investigating various processing options for treatment of this waste.<sup>1,2</sup> These options consider processing within the tank as well as fabrication of a purpose-built facility for the treatment. Available treatment technologies for the organic waste include, but are not limited to, acid hydrolysis, thermal destruction, catalytically induced destruction, and destruction based on Fenton's chemistry [i.e., use of hydrogen peroxide and an appropriate metal catalyst]. This report evaluates the potential impact of treated waste from these processes on interfacing facilities that may receive part or all of the treated material.

The in-tank and out-of-tank processes will be evaluated separately as they will have significantly different impacts on current processing. Portions of the waste may be processed in the Salt Waste Processing Facility, Defense Waste Processing Facility, Actinide Removal Process, Saltstone Facility or concentrated in the HLW evaporators. The in-tank products will likely be stored in a high level waste tank prior to the ultimate processing. To minimize the impact and identify the more significant risks, testing will

need to be completed to ensure that the product of the TPB decomposition is compatible with current HLW facilities.

## **Details**

### **Out-of-tank Fenton's Chemistry Process**

The product of an out-of-tank Fenton's Chemistry Process is very similar to the product of the acid hydrolysis process, the reference process for precipitate processing. Both processes produce a product with a pH of ~3.5, destroy the TPB, use a copper catalyst, and produce a product low in TOC [i.e., total organic carbon]. The Fenton's chemistry product also contains peroxide in low concentrations since it is too unstable to persist at significant concentrations. The out-of-tank process produces the least organic species in its product due to the combination of Fenton's Chemistry, hydrolysis reactions, and steam stripping to remove virtually all of the insoluble organic species.

#### **Advantages:**

1. Most complete organic destruction of any chemical process.
2. No corrosion potential to tank

#### **Disadvantages**

1. Not demonstrated with actual waste (has worked with simulant)
2. Most expensive processing method
3. May slow DWPF processing (if coupled with DWPF)

### **In-tank Fenton's Chemistry Process**

The product of an in-tank Fenton's Chemistry Process is very similar to a dilute high nitrate salt waste. The main addition to the waste is hydrogen peroxide, water, nitrate and catalyst. After processing, the waste will be returned to 1 M free hydroxide to ensure corrosion control. However, the following is a list of treated waste attributes that might pose complications with interfacing facilities

#### **Advantages:**

1. More complete destruction of organic than hydrolysis processes.

#### **Disadvantages**

1. Not demonstrated with actual waste (has worked with simulant)
2. Catalyst expensive (\$500K) and is not yet commercially available
3. Most corrosion potential to tank

### **In-tank Thermal Hydrolysis Process**

Many of the downstream impacts for the In-tank Thermal Hydrolysis Process are identical to the in-tank Fenton process impacts. The main addition to the waste is water, and nitrate. After processing, the waste will be returned to 1 M free hydroxide to ensure corrosion control. Tank 50H used thermal hydrolysis to assist in the removal of the residual potassium tetraphenylborate.

**Advantages:**

1. Simplest in-tank process
2. Lowest corrosion potential of in-tank process
3. Inexpensive processing method

**Disadvantages**

1. May not completely destroy TPB
2. Not demonstrated with actual waste (has worked with simulant)

**In-tank Catalyzed Thermal Hydrolysis Process**

Many of the downstream impacts for the In-tank Catalyzed Thermal Hydrolysis Process are identical to the in-tank Thermal Hydrolysis Process impacts. The main addition to the waste is water, catalyst and nitrate. After processing, the waste will be returned to 1 M free hydroxide to ensure corrosion control. Tank 49H used copper catalyzed thermal hydrolysis to destroy the residual sodium tetraphenylborate and allow the tank to return to routine HLW service. In FY03 testing, the catalyzed hydrolysis actually led to slower TPB decomposition than the thermal hydrolysis.

1. Simple in-tank process
2. Lower corrosion potential than in-tank Fenton
3. Inexpensive processing method

**Disadvantages**

1. May not completely destroy TPB
2. Not demonstrated with actual waste (has worked with simulant)
3. May take longer than thermal hydrolysis

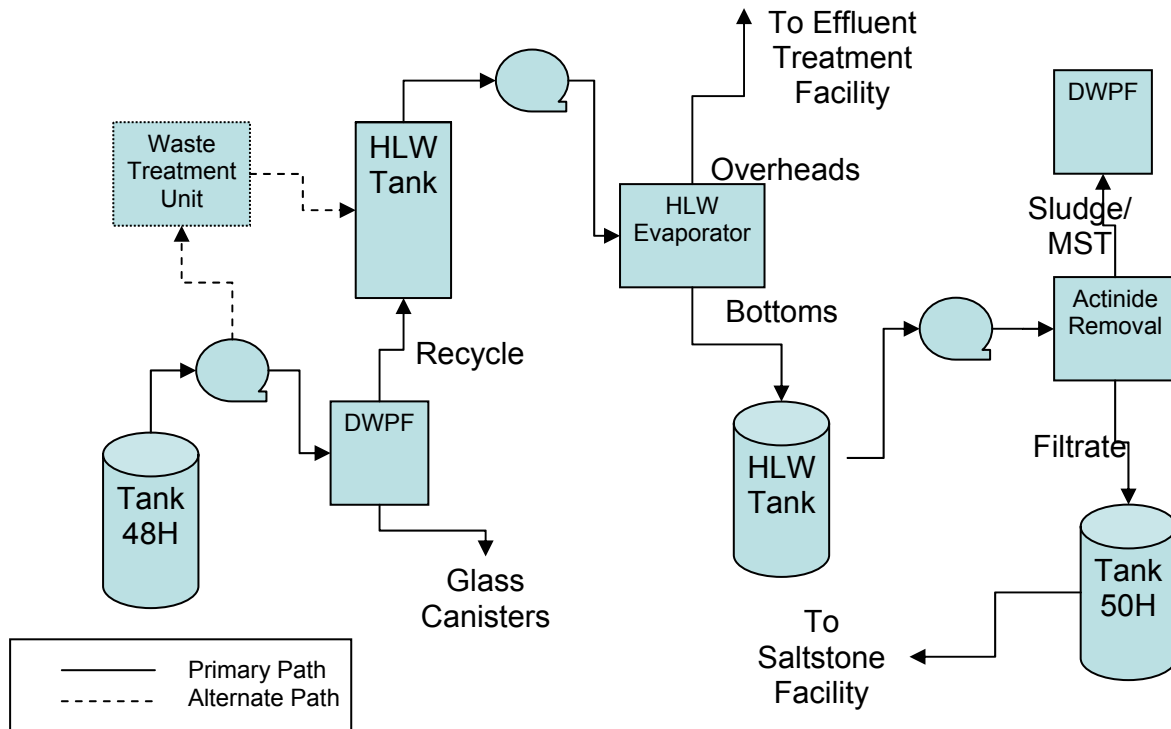
**Downstream Impacts identified by team**

A meeting was held on October 14, 2003 to identify downstream impacts of in-tank and out-of-tank Fenton Processing. A number of site experts (listed in acknowledgements), representing the various downstream processing facilities at SRS, were present. A summary of the meeting is attached (Attachment 1).

The likely processing path for an out-of-tank process is identified in Figure 1. The most notable impacts identified by the team were primarily impacts to DWPF, assuming the product from the Fenton Process was fed directly to DWPF. Any high organic stream has the potential to impact DWPF by fouling processing equipment, leading to flammability issues in the melter and requiring an earlier melter replacement. A high nitrate stream has the potential to lower the melt rate and lead to higher offgas production in the melter offgas system. A high sodium feed may require a frit change to ensure glass quality.

Figure 1

# Out-of-tank Treatment Flowpath

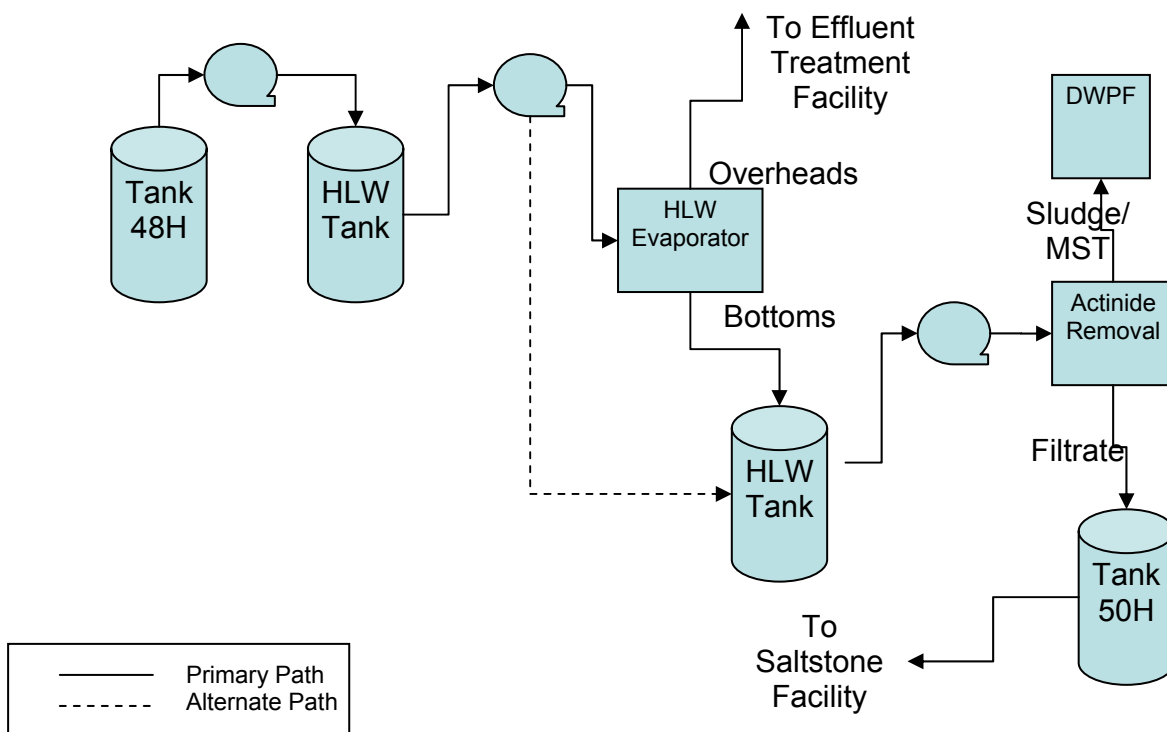


The likely processing path for an in-tank process is identified in Figure 2. The primary concern with in-tank processing is tank and cooling coil corrosion. Tank 48H is needed to serve as the feed tank for the Actinide Removal Process. Significant corrosion to the tank or cooling coils would be a distinct disadvantage. The advantage of in-tank processing is that Tank 48H waste after treatment would likely be returned to the tank farm and evaporated prior to being fed to the Actinide Removal Process (ARP) or Salt Waste Processing Facility (SWPF). This would result in sending the vast majority of the sodium and salts (including the high nitrate) to the Effluent Treatment Facility (ETF) and Saltstone, less expensive processing facilities than DWPF. Another advantage is the waste would be decoupled from DWPF processing. However, most of the cesium (now separate and concentrated) would also be fed to Saltstone (unless this feed is sent to the SWPF) as most of the soluble salts are fed to the Saltstone facility. The in-tank processing products are likely to be higher in organic content which could lead to fouling of filters in ARP or SWPF, or fouling of the HLW evaporators.



Figure 2

# In-tank Treatment Flowpath



Future testing or engineering analysis should identify whether any of these postulated problems will impact processing. Integrated testing of the flowsheet with simulated waste and actual waste should occur to identify any major issues related to processing the Fenton's Chemistry Process product. For example, completing a series of DWPF Sludge Receipt and Adjustment Tank (SRAT)/Slurry Mix Evaporator (SME) cycles, feeding to a melter, and evaporating the condensate are likely to identify major processing problems. Personnel should also conduct testing to confirm that the organic byproducts formed in actual waste agree with those detected during tests with simulated waste. Thermal analysis would be used to determine if any energetic compounds are produced. Rheology testing would examine the impact on transfer behavior. However, there is a potential risk that additional process problems will become an issue only after many batches are processed. For example, the original Salt Cell process discovered increased concentrations of residual products in some of the process lines only after many batches had been completed.

## 1. Corrosion impacts

Additional corrosion testing should be performed to ensure Tank 48H is not damaged during processing. Testing in 2003 by Zapp and Mickalonis<sup>3</sup> identified pitting as a potential concern to in-tank processing. Temperature, pH and time

limits may be required to protect the tank. The oxidizing process may be more aggressive on the tank, coils, and offgas components than just lowering the pH.

Corrosion testing should also be performed to ensure the salt cell materials of construction are acceptable for Fenton's Chemistry Processing. The oxidizing process may be more aggressive on the existing Hastelloy reactor, coils, and offgas components than the original hydrolysis process.

## 2. Organic Decomposition Products:

Ideally TPB will decompose to  $\text{CO}_2$  in the Fenton's Chemistry Process or benzene in the hydrolysis processes. However, other reactions compete and these reactions may produce tar-like organic species. In addition, the Fenton's reaction becomes less efficient as the carbon concentration decreases so there will always be some organic species remaining at the completion of processing. The hydrolysis reactions are also first order in TPB concentration so will become less efficient as the TPB concentration decreases. For the in-tank processes, the soluble organic species will likely be fed to Saltstone and the insoluble species will be likely to be removed through filtration in the Actinide Removal Process.

These organic species may cause problems in several places

### a. Organic species in DWPF Chemical Cell

The radioactive product of the Fenton's Chemistry Process will have some organic species present after completion (estimated as 200-500 mg/L carbon based on testing to date). These organic species could steam strip during the extensive boiling period in the SRAT and SME processing, or could react with the nitric or formic acid in the Chemical Cell to produce new organic species. The need to clean the accumulated organic deposits is much less likely in the Chemical Cell than it is in the Fenton's Chemistry processing equipment because the bulk of the insoluble organics will be removed by steam stripping during Fenton processing.

### b. Organic species in DWPF Melter

The organic species in the melter feed affect the redox of the slurry. One concern is that too much organic will lead to a very reducing melter feed which may lead to the reduction of noble metals, copper and nickel in the melter. This has the potential to reduce the melter's life by shorting out the melter. The redox is currently balanced by having the proper mix of formic acid and nitric acid.

### c. Organic species in HLW evaporators

The organic species in the condensate from the Fenton's Chemistry Process will be returned to the High Level Waste System and will be evaporated using a High Level Waste Evaporator. The organic species left in the condensate could foul the evaporator coils, evaporator, or condenser or cause processing problems in ETF or Saltstone.

### d. Organic species in filtration in Actinide Removal

Tar-like organic species could coat or foul the filters planned for use in actinide removal. If this happens, the oxalic acid cleaning is unlikely to remove the organics from the filter as this cleaning is designed to dissolve sludge and will not dissolve the organic deposits.

- e. Organics in Tank 48H  
Identification of the organic products, along with tests to determine the impact of these organic species will be required. The organic species will be different in the Fenton's Chemistry Product, which will be returned to the High Level Waste system and will be evaporated using a High Level Waste Evaporator. The organic species left in the condensate could foul the evaporator coils, evaporator, or condenser or cause processing problems in ETF.
3. High sodium content
- The treated waste will have much higher sodium content than originally planned with the reference precipitate hydrolysis process. For example, the nitrite concentration was expected to be 0.01 M in the original process but is ~0.50 M in the current precipitate feed. As a result, if the waste is fed to DWPF, more sodium will be fed to the DWPF melter. Operations may compensate by altering the washing point of the sludge or by modifying the frit to contain less alkali. Alternatively, processing of this waste may result in more canisters of glass (approximately 250 more if no frit change is made). However, the higher sodium content may be beneficial in increasing melt rate. Although DWPF process models will likely predict that quality glass can be produced with a frit change, these models can not predict how well DWPF can process this new feed.
- a. Frit change required  
Model calculations should be completed to ensure the current frit is acceptable. If it is not acceptable, model calculations and crucible studies will be required to identify a frit that will allow optimum melter performance with the new Fenton's Chemistry product. If major processing changes are required, integrated testing through the minimelter should be completed.
4. High nitrate concentration
- Nitric acid is used in the Fenton's Chemistry Process to acidify the waste and control pH. The acid used is nitric, not formic acid. This is because the formic acid would compete with the TPB destruction in the process making pH control more difficult. As a result, the nitrate concentration in the product will be an order of magnitude higher than produced in the Acid Hydrolysis Process.
- a. Formic acid process in chemical cell  
The SRAT process will likely require the addition of formic acid as the only acid to produce a melter feed with the appropriate redox. If a formic acid only process in the SRAT produces a melter feed that is too oxidizing, an additional reductant such as sugar may be required. Another option is to use formic acid or CO<sub>2</sub> to reduce the pH to minimize the nitric acid used. This may help in producing a melter feed with the appropriate redox.
  - b. Higher hydrogen generation  
The addition of more formic acid in the SRAT may lead to higher hydrogen production. The optimum SRAT processing should be developed using a series of small-scale experiments, demonstrated in larger scale integrated processing and actual waste testing.

5. Impacts to DWPF processing

The addition of a new stream, the Fenton's Chemistry product, to the SRAT is a significant change in the SRAT process. This stream may have to be fed at boiling (like originally planned for the hydrolysis product) and may extend the cycle time. In addition the waste is likely to change the rheology of the slurry in the SRAT and SME. As a result, integrated testing and actual waste testing is recommended.

6. Recycle impacts

The condensate collected during vitrification and processing of the Fenton's Chemistry product will be combined with other DWPF condensate, caustic and nitrite adjusted, and returned to a HLW tank. It is estimated that the additional recycle volume is less than 20% of the current DWPF recycle volume. As the condensate is very dilute, it may be evaporated prior to future processing. Testing of the evaporation of the DWPF recycle stream, including Fenton's Chemistry product condensate, Chemical Cell condensate, and melter condensate should be considered using condensate collected during large-scale integrated testing to determine whether there are issues related to the evaporation of this stream. However, this is unlikely to be a problem based on previous recycle testing that used recycle stream simulating that produced during vitrification of precipitate hydrolysis product.

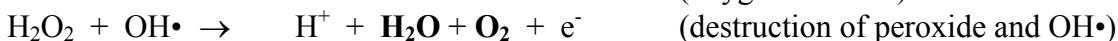
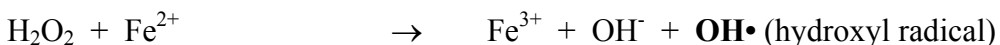
### **Acknowledgements**

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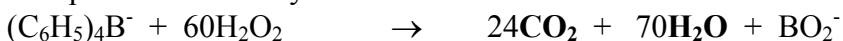
**Appendix A**  
**Results of Downstream Impacts Meeting**  
**October 14, 2003**

**Introduction**

Fenton's Chemistry is being considered to destroy TPB and oxidize the carbon to CO<sub>2</sub>.



Complete destruction yields



- Reaction works best at pH 3-5. Nitric acid added to reduce pH.
- Reaction kinetics faster at higher temperatures.
- Catalyst needed (200 ppm Cu or Fe at pH 3.5 or ~100 ppm TAML at pH 11)
- Meter in peroxide slowly to control reaction
  - Exothermic reaction
  - Large volume of gas generated (CO<sub>2</sub> + O<sub>2</sub>)
- Processing dilutes feed (doubles original volume)
- In-tank processing will require pH adjustment and nitrite addition at completion of processing.

Parameter	In-Tank	Out-of-tank
Where?	Tank 48H	DWPF Salt Cell
How many batches	1	100-200
Initial pH	11	7.5
Final pH	11	3.5
pH adjustment post processing	>1 M [OH <sup>-</sup> ]	Send to SRAT
Temperature	45 °C	100 °C
Mol H <sub>2</sub> O <sub>2</sub> :mol C	3-9	5
Complete TPB destruction	Yes?	Definitely
TOC remaining	unknown	<500 ppm
Peroxide remaining	None	None
Final Na	2.5 M	1.5 M
Final NO <sub>3</sub> <sup>-</sup>	1 M	1.1 M
Added [OH <sup>-</sup> ]	1 M	None
Added NO <sub>2</sub> <sup>-</sup>	As needed	None
Catalyst	Cu, Fe or TAML <sup>®</sup>	Cu

**What will happen to product of Fenton Destruction?**

1. Out-of-Tank (DWPF Salt Cell)
  - a. Fed to SRAT (Condensate will go to SMECT/RCT/HLW Tank)
    - i. Condensate will eventually be concentrated in HLW Evaporator
      1. HLW Evaporator Overheads will go to ETF
      2. HLW Evaporator Bottoms will go to drop tank.
    - ii. Drop tank will be fed to Actinide Removal or Salt Waste Processing Facility
      1. Decontaminated Salt Solution will be fed to Saltstone
      2. Sludge/MST/cesium will be fed to DWPF
  - b. Fed directly to Saltstone Facility
  - c. Use condensate to dissolve Saltcake
    - i. Dissolved saltcake will be fed to Actinide Removal or Salt Waste Processing Facility
    - ii. Decontaminated Salt Solution will be fed to Saltstone
    - iii. Sludge/MST/cesium will be fed to DWPF
2. In-Tank (Tank 48H)
  - a. Add caustic and nitrite for corrosion control and transfer to a HLW Tank
    - i. Eventually slurry will be concentrated in HLW Evaporator
      1. HLW Evaporator Overheads will go to ETF
      2. HLW Evaporator Bottoms will go to drop tank.
    - ii. Drop tank will be fed to Salt Waste Processing Facility
      1. Decontaminated Salt Solution will be fed to Saltstone
      2. Sludge/MST/cesium will be fed to DWPF
  - b. Fed directly to Saltstone Facility

**Concerns with Fenton Product**

1. What will happen to organic?
  - a. How will it partition between condensate and bottoms?
  - b. Will it foul evaporator vessels (SRAT, SME, HLW evaporators)?
  - c. Will it increase organic load to DWPF melter?
  - d. Will degradation products cause a flammability concern (more likely in-tank)?
  - e. Will degradation products cause a foaming problem?
2. What will happen to the bulk of the waste?
  - a. Will catalysts impact downstream processing?
  - b. Will high nitrate complicate processing (especially DWPF)?
    - i. REDOX issues
    - ii. High offgas generation in melter
  - c. Will high sodium complicate processing (especially DWPF)?

### Downstream Impacts Data Sheets

Facility/Equipment	What is postulated impact?	How could this be answered?
DWPF Melter	Melter feed is too reducing; noble metals are reduced and short out melter. Melter must be replaced.	1. paper study 2. stimulant study 3. actual waste demo
DWPF Melter	Offgas flammability due to high organic content	1. paper study 2. stimulant study 3. actual waste demo
DWPF SRAT and SME	High nitrite, nitrate impact hydrogen generation	1. stimulant study 2. actual waste demo
DWPF Melter	High sodium impacts	Paper study
DWPF SRAT/SME HLW Tank HLW Evaporator	Do remaining organic species react with other waste	1. paper study 2. stimulant study
DWPF SRAT/SME HLW Tank HLW Evaporator	Do tar-like substances foul equipment	Stimulant study
DWPF SRAT/SME HLW Evaporator	Excessive foaming during evaporation	1. paper study 2. stimulant study 3. actual waste demo
HLW Tank	Flammable organic species	Stimulant study
DWPF Fenton Reactor	Flammable vapor space	1. stimulant study 2. actual waste demo
DWPF Fenton Reactor Tank 48H HLW Tank	Corrosion	1. paper study 2. stimulant study
Tank 48H	Would there be a better acid than nitric acid (formic, oxalic)	Simulant testing
HLW Tank	Solids are not packed sufficiently so solids take up excess volume	simulant study
DWPF Melter	Does catalyst impact glass quality	Paper study if iron Simulant study if other
HLW Tank DWPF SRAT	Does peroxide impact downstream processing facility	Paper study
DWPF Fenton Reactor	Does gas generation impact processing	1. stimulant study 2. actual waste demo
ARP Filtration SWPF Filtration	Do tar-like organic species foul filters or make more difficult to clean.	Simulant Study
All	Do processing changes impact DSA	Paper study with flowsheet

<b>Facility/Equipment</b>	<b>What is postulated impact?</b>	<b>How could this be answered?</b>
HLW evaporators	Do new scaling species foul heat exchanger surfaces	Paper study with flowsheet. Testing if needed
HLW Evaporators	Mercury reactions form new mercury species	Have Donnie Thaxton's task team evaluate. Testing if required.



## References

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- <sup>1</sup> T. B. Peters, D. P. Lambert, M. E. Smith, and S. D. Fink, "Task Technical and Quality Assurance Plan for Destruction of Tetraphenylborate in Tank 48H," WSRC-RP-2003-00396, May 27, 2003.
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- <sup>3</sup> P. E. Zapp and J. I. Mickalonis, Electrochemical Tests Of Carbon Steel In Simulated Waste Containing Fenton's Reagent, WSRC-TR-2003-00445, October, 2003.