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# Calculation of Glycolate Concentration Factors Across the 242-25H (3H) Evaporator System

**W. H. Woodham**

October 2020

SRNL-STI-2020-00385, Revision 0



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## REVIEWS AND APPROVALS

### AUTHORS:

---

W. H. Woodham, Chemical Flowsheet Development

### TECHNICAL REVIEW:

---

C. J. Martino, Chemical Flowsheet Development, Reviewed per E7 2.60

### APPROVAL:

---

F. M. Pennebaker, Program Manager  
Chemical Processing Sciences

---

S. D. Fink, Director  
Chemical Processing Sciences

---

J. E. Occhipinti, Manager  
Tank Farm Facility Engineering

## EXECUTIVE SUMMARY

Two models were developed to predict glycolate concentration factors across the 242-25H (3H) Evaporator system. The first model, identified as the Unrestricted Evaporation Model, was designed to evaluate concentration of typical 3H feed material to relatively high densities in order to calculate the concentration factor of glycolate in a given feed stream. This model may be used to understand the potential of glycolate to concentrate from a generic feed material through the 3H system. The second model, identified as the Holistic Evaporation Model, was designed to evaluate the evaporation of dilute, glycolate-containing Defense Waste Processing Facility (DWPF) waste with the requirement that high silicon concentrations typical of recycle material require dilution to adhere to current technical safety requirements. This model may be used to understand the potential of glycolate from DWPF recycle material to concentrate as a result of feeding through the 3H system.

The following conclusions are made as a result of the work described in this document.

- The 95<sup>th</sup> percentile of possible glycolate concentration factors across the 3H Evaporator system (Tanks 30, 32, 37, and the 3H evaporator pot) are 7.4 (assuming no thermolytic degradation) and 2.7 (assuming thermolytic degradation). These concentration factors are expected to be applicable to any feed transferred into the 3H system with the condition that the feed stream is chemically similar to material fed to the 3H system over the past decade.
- The 95<sup>th</sup> percentile of possible glycolate concentration factors of waste originating from the DWPF Recycle Collection Tank, fed to Tank 22, and proceeding via dilution in other tanks before transfer to the 3H system are 7.8 (assuming no thermolytic degradation) and 2.4 (assuming thermolytic degradation). These concentration factors are expected to be applicable to Tank 22 waste with the condition that the material in question retains silicon compositions similar to those observed over the past decade.
- Glycolate concentration factors across the 3H Evaporator system are expected to be lower than those predicted across the 2H Evaporator system, largely due to the elevated temperatures and concentrations achieved in the 3H Evaporator leading to higher rates of thermolytic degradation.

The following recommendations are made as a result of the work described in this document.

- Safety basis calculations for allowable glycolate in 3H Evaporator waste streams should take the concentration factors given above into consideration when calculating the impacts of glycolate thermolysis on flammability.
- Additional studies (including laboratory testing) should be performed to monitor total organic carbon (TOC) degradation to assess if a similar credit for decreased TOC thermolysis could be assumed in 3H evaporation. The degradation of antifoam products is of particular interest given the potential to form methane at high temperatures and should be studied independently.
- Periodic sampling should be performed to confirm the applicability of these models to ongoing radioactive waste processing. In particular, processing changes that may impact the conclusions of these models (e.g., transfers of silicon from frit to recycle material, modifications of silicon concentration in recycle material due to alternate antifoam use) should be assessed upon implementation to ensure consistency with the assumptions described in this document.

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## LIST OF ABBREVIATIONS

CSTF	Concentration, Storage, and Transfer Facilities
CSTR	Continuously-stirred tank reactor
DWPF	Defense Waste Processing Facility
EFQ	Evaporator Feed Qualification
NG	Nitric-Glycolic
RCT	Recycle Collection Tank
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
TOC	total organic carbon

## 1.0 Introduction

The Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS) is planning to switch to the Nitric-Glycolic (NG) flowsheet for enhanced waste processing. In the NG flowsheet, formic acid is replaced with glycolic acid as a reducing agent with the added benefit of greatly decreasing hydrogen formation in the DWPF.<sup>1</sup> Following implementation of the NG flowsheet at DWPF, some glycolate is expected to be lost via carryover or evaporation to the process condensate streams, ultimately leading to accumulation in the Recycle Collection Tank (RCT).

The recycled material from the RCT will transfer to the Concentration, Storage, and Transfer Facilities (CSTF) via Tank 22, where glycolate will be introduced into tank farm waste. Researchers at the Savannah River National Laboratory (SRNL) recently showed that the presence of glycolate in caustic tank waste is expected to lead to the evolution of hydrogen gas via thermolytic degradation of glycolate.<sup>2</sup> As a result, Savannah River Remediation (SRR) personnel began developing documentation and controls to mitigate the concern of flammable headspace generation in the CSTF. Part of this process is understanding how glycolate may be concentrated in and around the evaporators within the CSTF.

Work was requested by SRR to develop an understanding of glycolate concentration in the CSTF following implementation of the NG flowsheet and permanganate oxidation.<sup>3,4</sup> This includes analysis of a new permanganate oxidation process being developed by SRNL researchers to decrease the concentration of glycolate in the RCT before transfer to Tank 22.<sup>5</sup>

A document studying the concentration of glycolate across the 242-16H (2H) Evaporator was generated to assess the risk of glycolate concentration in common DWPF recycle streams.<sup>6</sup> This report is prepared as a complimentary work to assess the risk of glycolate concentration across the 242-25H (3H) Evaporator system, which is capable of higher temperatures and hydroxide concentrations than those typically seen in the 2H Evaporator system.

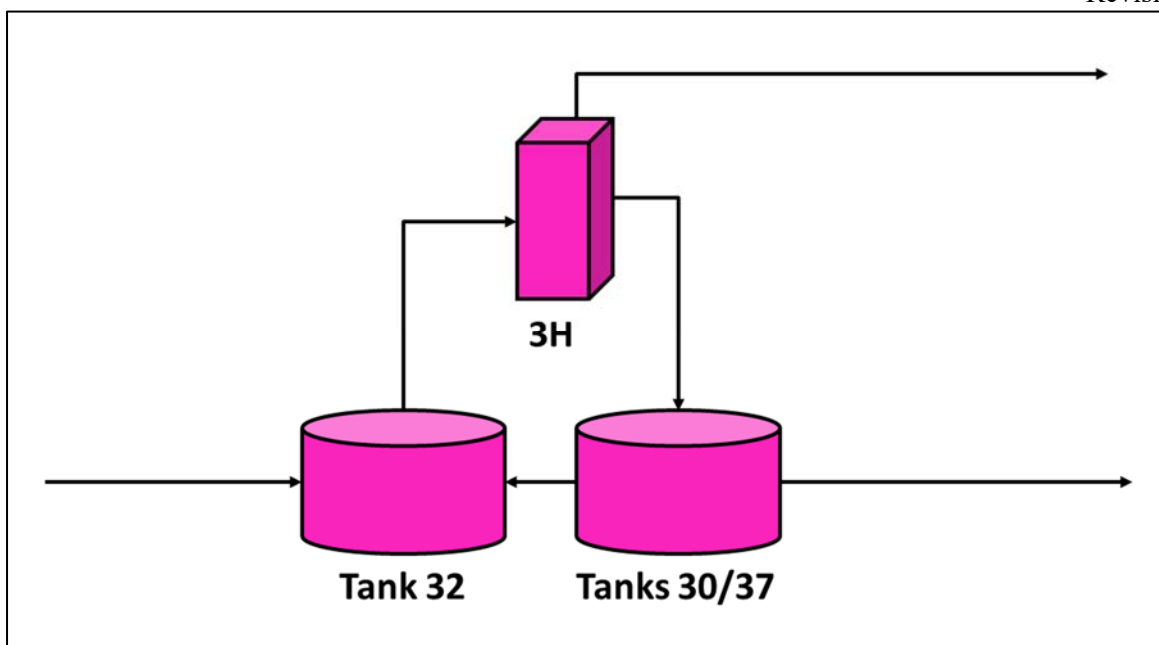
## 2.0 Calculation Procedure

### 2.1 Model Assumptions

The key assumptions made in this document are similar to those made in previous work simulating the concentration of glycolate in the 242-16H (2H).<sup>6</sup>

- Glycolate is introduced to the CSTF via DWPF recycle material transferred into Tank 22.
- The time-averaged behavior of glycolate in the CSTF may be approximated using a network of continuously stirred tank reactors (CSTRs).
- Glycolate will deteriorate via thermolysis at a rate equal to the rate of hydrogen formation from glycolate thermolysis.

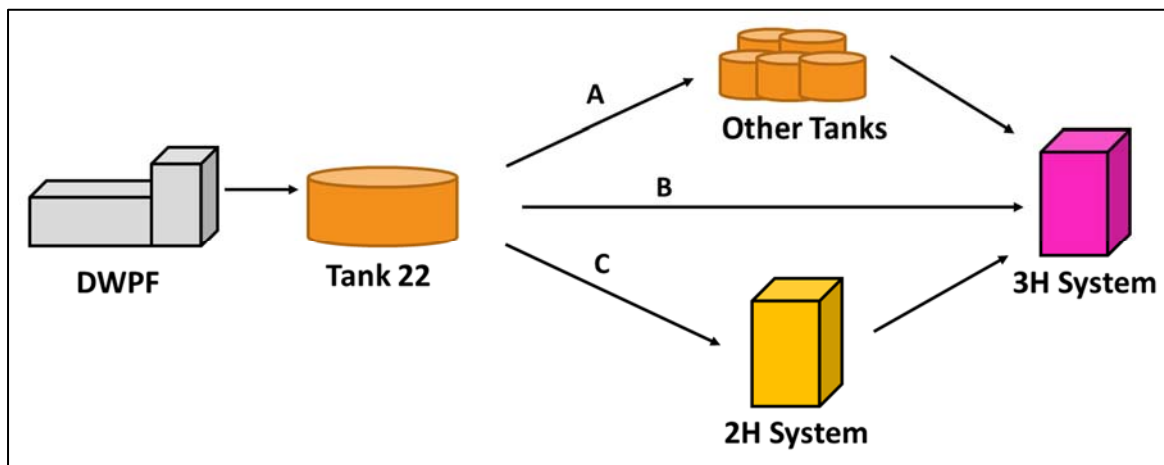
For the purposes of this report, a block flow diagram was generated to identify flow streams for glycolate-containing material through the 3H Evaporator system. That block flow diagram is given in Figure 2-1.



**Figure 2-1. Block Flow Diagram of 3H Evaporator System.**

Glycolate-containing material is assumed to first be transferred to Tank 32 (the 3H feed tank). Material is then transferred continuously from Tank 32 to the 3H Evaporator pot, where it is evaporated and the bottoms are subsequently transferred to Tanks 30 or 37. During an evaporator campaign, material in Tanks 30 or 37 (whichever is identified as the drop tank at the time of use) is recycled to Tank 32. At the conclusion of an evaporator campaign, 3H material may be removed from Tanks 30 or 37 and transferred to additional tanks throughout the CSTF. For the purposes of this analysis, Tanks 30 and 37 may be used interchangeably as drop tanks. Similarly, the conclusions drawn from this model would be expected to apply if the glycolate-containing material were fed to Tanks 30 or 37 and the 3H Evaporator drop material was transferred to Tank 32. This flowsheet is the basis for the Unrestricted Evaporation Model for glycolate-containing material (discussed later in this report).

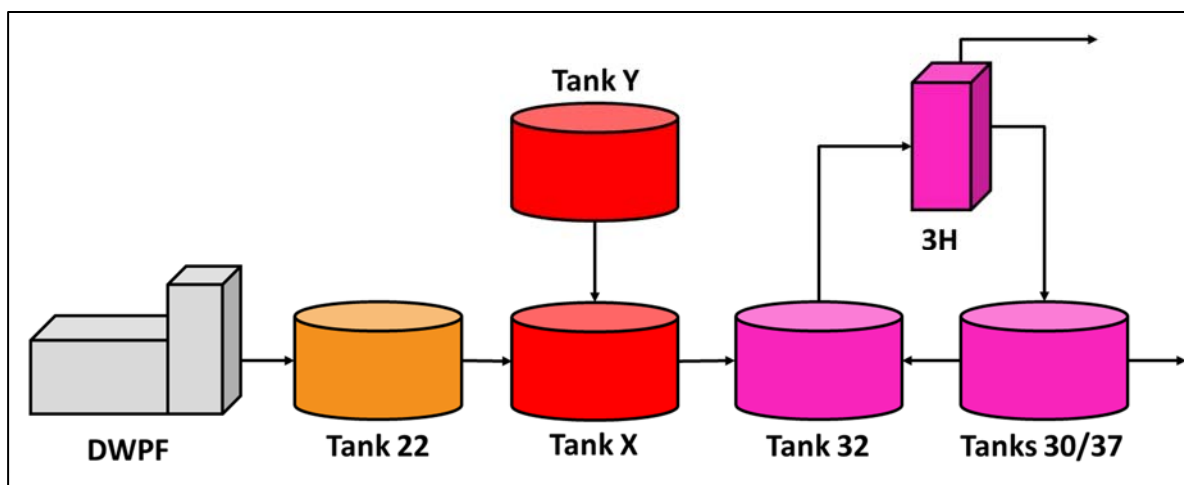
As stated above, a key assumption of this document is that glycolate is only introduced into the CSTF via DWPF recycle transfer into Tank 22. It is necessary to identify all possible flow paths of glycolate-containing material from Tank 22 to the 3H system to make a conservative estimate of the possible glycolate concentration factors. Figure 2-2 depicts the identified possible paths of glycolate in the CSTF.



**Figure 2-2. Possible Routes for Glycolate Transfer from Tank 22 to 3H System.**

Figure 2-2 identifies potential paths of glycolate reaching the 3H system by A) transfer to other tanks prior to transfer to the 3H system, B) direct transfer from Tank 22 to the 3H system, or C) transfer to the 2H system and subsequent evaporation prior to transfer to the 3H system.

At first glance, the possible pathways for glycolate to reach the 3H system add complexity. However, current Evaporator Feed Qualification (EFQ) program controls greatly limit the possible pathways.<sup>7</sup> The EFQ program requires that silicon content of any material transferred to the 3H system (feed tank or drop tank) must be “less than 120% of the historical maximum silicon concentration for feed to the evaporator”.<sup>8</sup> This historical maximum has been identified as 65.33 mg/L.<sup>9</sup> Therefore, material fed to the evaporator system must have an average silicon concentration less than of 78.4 mg/L. Over the past decade, Si concentrations in DWPF recycle material (as measured in Tank 22) greatly exceed this limit (135 – 265 mg/L vs. 78.4 mg/L). Therefore, the high-silicon, glycolate-containing material transferred from the DWPF RCT to Tank 22 will need appreciable dilution with low-Si waste before transfer can be made into the 3H system. This prohibits pathway B in Figure 2-2 (direct transfer of Tank 22 to the 3H system). Tank 22 material is typically transferred to the 2H system where it is further concentrated. The Si concentrations in the 2H system tend to be elevated and enhanced to the point of sodium aluminosilicate formation, which would be problematic for the 3H system, making pathway C equally unlikely (the possibility of a transfer from Tank 22 to the 2H system followed by dilution by other tank material and transfer to the 3H system was not considered due to the unlikelihood of this particular path, the similar restrictions due to increased silicon loading, and the inability of this unlikely pathway to sustain long-term concentration of glycolate-containing material).<sup>10-11</sup> The consideration of these factors allows reduction of the full block flow diagram in Figure 2-2 to the simplified version presented in Figure 2-3.



**Figure 2-3. Block Flow Diagram of Recycle Material Transfer to 3H System.**

It should be noted that an observation of a Tank 22 silicon concentration of less than 78.4 mg/L was made in 2014. However, this sample was drawn immediately following the transfer of Tank 51 material into Tank 22 and is therefore not representative of DWPF recycle material.

Figure 2-3 shows the flow of glycolate-containing material from DWPF into Tank 22. Afterwards, it is transferred to an arbitrary tank (here identified as “Tank X”) where it is combined with material containing no glycolate or silicon (assumed to be transferred from an arbitrary “Tank Y”). Following combination in Tank X, glycolate-containing material is transferred to Tank 32 and continues as usual throughout the 3H system. This flowchart is the basis for the Holistic Evaporation Model for glycolate-containing material (discussed later in this report).

## 2.2 Model Inputs

The inputs used for the unrestricted model are presented in Table 2-1.

**Table 2-1. Inputs Used for the Unrestricted Evaporation Model.**

Parameter	Description	Value	Sensitivity <sup>†</sup>
$\dot{V}_{32}^{in}$	Volumetric flow rate of feed into Tank 32	1.4 – 2.3 gal min <sup>-1</sup>	+
$[Gly]_{in}$	Concentration of glycolate in feed to Tank 32	10 mg L <sup>-1</sup>	N/A
$\omega_{32}^{in}$	Salt component compositions in feed to Tank 32	0.215 ≤ [NO <sub>2</sub> ] ≤ 1.0543 mol L <sup>-1</sup> 0.123 ≤ [NO <sub>3</sub> ] ≤ 1.2 mol L <sup>-1</sup> 0.2357 ≤ [OH] ≤ 3.8613 mol L <sup>-1</sup> 0.065 ≤ [CO <sub>3</sub> ] ≤ 0.312 mol L <sup>-1</sup>	-
$V_{32}$	Volume of Tank 32 supernatant phase	28,000 – 500,000 gal	-
$T_{32}$	Temperature of Tank 32 supernatant phase	25 – 54 °C	-
$V_{30}$	Volume of drop tank (Tank 30 or 37) supernatant phase	1,800 – 750,000 gal	-
$T_{30}$	Temperature of drop tank (Tank 30 or 37) supernatant phase	25 – 50 °C	-
$V_{3H}$	Volume of 3H evaporator pot	10,000 gal	-
$T_{3H}$	Temperature of liquid in 3H evaporator pot	140 °C	-
$\dot{V}_{3H}^{in}$	Volumetric flow rate of 3H evaporator feed	17 – 35 gal min <sup>-1</sup>	+
$\rho_f$	Target density in 3H evaporator pot	1.5 – 1.65 g mL <sup>-1</sup>	+

<sup>†</sup>Sensitivity is defined here as the sign of the derivative of the glycolate concentration factor with respect to the variable in question. A sign of “+” indicates that when the variable is increased, the glycolate concentration factor would be expected to increase. A sign of “-” indicates that when the variable is increased, the glycolate concentration factor would be expected to decrease.

The range of values for  $\dot{V}_{32}^{in}$  was chosen based on an assumption of glycolate-containing material being introduced to the CSTF at an anticipated rate of 750,000 to 1,200,000 gal yr<sup>-1</sup>. Ranges of values for  $V_{32}$ ,  $T_{32}$ ,  $V_{30}$ ,  $T_{30}$ ,  $V_{3H}$ ,  $\rho_f$ , and  $\dot{V}_{3H}^{in}$  were chosen based on recommendations made by SRR personnel. The value of 140 °C for  $T_{3H}$  was chosen based on a parameter review and recommendation provided by SRR personnel.<sup>12</sup>  $[Gly]_{in}$  was arbitrarily set to 10 mg L<sup>-1</sup> (and this value exhibits no influence on predicted concentration factors). Values for  $\omega_{32}^{in}$  were chosen statistically from sample records reported in the NTANK database.<sup>13</sup> Measured values of nitrite, nitrate, hydroxide, and carbonate from Tanks 29 and 51 in all samples taken since January 1<sup>st</sup>, 2010, were compiled and used to generate a distribution representative of tank chemistry that is typical of feed to the 3H Evaporator system.

More inputs are necessary for the holistic model. The required inputs are provided in Table 2-2.

**Table 2-2. Inputs Used for the Holistic Evaporation Model.**

Parameter	Description	Value	Sensitivity <sup>†</sup>
$\dot{V}_{22}^{in}$	Volumetric flow rate of DWPF recycle material into Tank 22	1.4 – 2.3 gal min <sup>-1</sup>	+
$[Gly]_{RCT}$	Concentration of glycolate in the recycle material	10 mg L <sup>-1</sup>	N/A
$\omega_{22}^{in}$	Salt component compositions in recycle material <sup>††</sup>	0.1507 ≤ [NO <sub>2</sub> ] ≤ 0.5528 mol L <sup>-1</sup> 0.0475 ≤ [NO <sub>3</sub> ] ≤ 0.5924 mol L <sup>-1</sup> 0.1519 ≤ [OH] ≤ 1.0065 mol L <sup>-1</sup> 0.0188 ≤ [CO <sub>3</sub> ] ≤ 0.1040 mol L <sup>-1</sup>	-
$V_{22}$	Volume of Tank 22 supernatant phase	250,000 – 950,000 gal	-
$T_{22}$	Temperature of Tank 22 supernatant phase	23 – 27 °C	-
$Si_{22}$	Concentration of silicon in Tank 22	135 mg L <sup>-1</sup>	-
$\omega_Y^{in}$	Salt component compositions in Tank Y material	0.215 ≤ [NO <sub>2</sub> ] ≤ 1.0543 mol L <sup>-1</sup> 0.123 ≤ [NO <sub>3</sub> ] ≤ 1.2 mol L <sup>-1</sup> 0.2357 ≤ [OH] ≤ 3.8613 mol L <sup>-1</sup> 0.065 ≤ [CO <sub>3</sub> ] ≤ 0.312 mol L <sup>-1</sup>	-
$V_X$	Volume of Tank X supernatant phase	50,000 – 1,000,000 gal	-
$T_X$	Temperature of Tank X supernatant phase	25 °C	-
$V_{32}$	Volume of Tank 32 supernatant phase	28,000 – 500,000 gal	-
$T_{32}$	Temperature of Tank 32 supernatant phase	25 – 54 °C	-
$V_{30}$	Volume of drop tank (Tank 30 or 37) supernatant phase	1,800 – 750,000 gal	-
$T_{30}$	Temperature of drop tank (Tank 30 or 37) supernatant phase	25 – 50 °C	-
$V_{3H}$	Volume of 3H Evaporator pot	10,000 gal	-
$T_{3H}$	Temperature of liquid in 3H Evaporator pot	140 °C	-
$\dot{V}_{3H}^{in}$	Volumetric flow rate of 3H Evaporator feed	17 – 35 gal min <sup>-1</sup>	+
$\rho_f$	Target density in 3H Evaporator pot	1.5 – 1.65 g mL <sup>-1</sup>	+

<sup>†</sup>Sensitivity is defined here as the sign of the derivative of the glycolate concentration factor with respect to the variable in question. A sign of “+” indicates that when the variable is increased, the glycolate concentration factor would be expected to increase. A sign of “-” indicates that when the variable is increased, the glycolate concentration factor would be expected to decrease.

<sup>††</sup>An artificial minimum of 0.15 M is assumed for nitrite and hydroxide concentrations of recycle material. This corresponds to the procedural requirement to remediate the DWPF recycle material with sodium nitrite and sodium hydroxide.

Values for  $V_{22}$  and  $T_{22}$  were chosen based on recommendation by SRR personnel. Values for  $\omega_{22}^{in}$  were chosen from previous measurements of nitrite, nitrate, hydroxide, and carbonate made from Tank 22 samples pulled since January 1<sup>st</sup>, 2010. The value for  $Si_{22}$  was chosen from the lowest silicon measurement made from Tank 22 material (excluding obvious outliers) since January 1<sup>st</sup>, 2010 (selection of the lowest Tank 22 silicon value requires less dilution to achieve a processable evaporator feed, thereby making the selection conservative). Values for  $V_X$  were chosen from an arbitrary range of possible supernatant volumes (representing possible levels in a random mixing Tank X),  $T_X$  is set to a defensible value of 25 °C.

Values for  $\omega_y^{in}$  were chosen from previous measurements of nitrite, nitrate, hydroxide, and carbonate made from Tank 51 samples pulled since January 1<sup>st</sup>, 2010 (Tank 51 was chosen as a representative source of typical feed to the 3H system).

## 2.3 Mathematical Treatment

### 2.3.1 Development of the Evaporation Model Around 3H Evaporator Tanks (Unrestricted Evaporator Model)

The mathematical development of 3H evaporation models is not unlike the development of 2H evaporation models described previously by SRNL.<sup>6</sup> The fundamental calculation is a mass balance of salt components around the 3H system, consisting of Tank 32, the drop tank (referred to as Tank 30), and the 3H pot. This mass balance is displayed graphically in Figure 2-4.

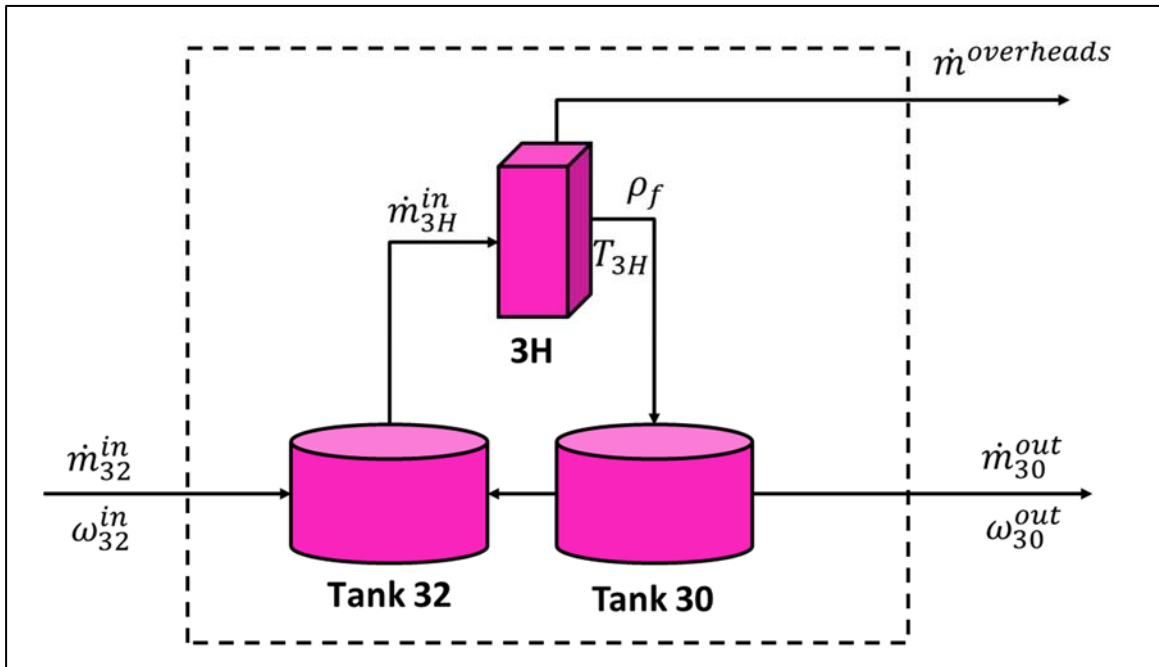


Figure 2-4. Mass Balance Around the 3H Evaporator System.

In the mass balance displayed in Figure 2-4, it is clear that the rate of salt components entering Tank 32 must be equal to the rate of salt components leaving Tank 30, as written in Equation [1].

$$\dot{m}_{32}^{in} \times \omega_{32}^{in} = \dot{m}_{30}^{out} \times \omega_{30}^{out} \quad [1]$$

By inspection, the mass fractions of salt components in Tank 30 (not necessarily the molarities) must be equal to the mass fractions of salt components in the 3H Evaporator pot. The bisection method was used to identify a mass fraction of water in the 3H Evaporator pot that yields the desired density  $\rho_f$  at a temperature of  $T_{3H}$ . For density calculations, the Laliberte-Cooper model previously used to simulate 2H evaporation was employed.<sup>14</sup> The expression used to calculate density is given in Equation [2].

$$\rho = \frac{1}{\frac{\omega_{H_2O}}{\rho_{H_2O}} + \sum_i \omega_i \bar{v}_{app,i}} \quad [2]$$

where  $\rho$  is the density of the target material in  $\text{kg m}^{-3}$ ,  $\omega_i$  is the weight fraction of component “i”,  $\rho_{H_2O}$  is the density of water in  $\text{kg m}^{-3}$ , and  $\bar{v}_{app,i}$  is the apparent specific volume of component “i” calculated according to Equation [3].

$$\bar{v}_{app,i} = \frac{\omega_i + c_2 + c_3 t}{(c_0 \omega_i + c_1) e^{(0.000001(t+c_4)^2)}} \quad [3]$$

where  $c_0$ ,  $c_1$ ,  $c_2$ ,  $c_3$ , and  $c_4$  are component-specific constants and  $t$  is the temperature in  $^{\circ}\text{C}$ .

Once the desired density is calculated, the composition of Tank 32 can be calculated according to Equation [4].

$$\omega_{32} = \frac{\dot{m}_{32}^{in} \omega_{32}^{in} + (\dot{m}_{3H}^{in} - \dot{m}^{overheads} - \dot{m}_{30}^{out}) \omega_{30}^{out}}{\dot{m}_{32}^{in} + \dot{m}_{3H}^{in} - \dot{m}^{overheads} - \dot{m}_{30}^{out}} \quad [4]$$

Degradation of glycolate was approximated in the same method described in previous work simulating evaporation around the 2H Evaporator. To evaluate glycolate in each of the 3H system tanks, a linear system of equations was established, as shown in Equation [5].

$$\begin{bmatrix} 1 & \frac{-(\dot{m}_{3H}^{in} - \dot{m}^{overheads} - \dot{m}_{30}^{out})}{(\dot{m}_{3H}^{in} + K_{32})} & 0 \\ 0 & 1 & \frac{-(\dot{m}_{3H}^{in} - \dot{m}^{overheads})}{(\dot{m}_{3H}^{in} - \dot{m}^{overheads} + K_{30})} \\ \frac{-\dot{m}_{3H}^{in}}{(\dot{m}_{3H}^{in} - \dot{m}^{overheads} + K_{3H})} & 0 & 1 \end{bmatrix} \times \begin{bmatrix} \omega_{32}^{gly} \\ \omega_{30}^{gly} \\ \omega_{3H}^{gly} \end{bmatrix} = \begin{bmatrix} \frac{\dot{m}_{32}^{in} \omega_{32}^{gly}}{\dot{m}_{3H}^{in} + K_{32}} \\ 0 \\ 0 \end{bmatrix} \quad [5]$$

Note that values indicated by  $K_i$  (in units of  $\text{kg min}^{-1}$ ) are reaction rate constants derived from conditions in Tank “i”, similar to earlier work simulating 2H evaporation.<sup>2, 6</sup>

After these calculations have been performed, a maximum theoretical concentration factor may be calculated by comparing the theoretical molarities of sodium (or any chemically inert species) in the outlet stream relative to the inlet stream, as shown in Equation [6]. This value is equivalent to a glycolate concentration factor without consideration of thermolysis. Note that these salt species concentrations are hypothetical, based on the assumption that the species of interest does not precipitate during or after evaporation. This assumption is safe for a glycolate maximum but may not accurately depict physical phenomena of highly concentrated salt species (i.e. sodium).

$$f_{Na} = f_{Gly}^{No Therm} = \frac{[Na]_{30}}{[Na]_{32}^{in}} \quad [6]$$

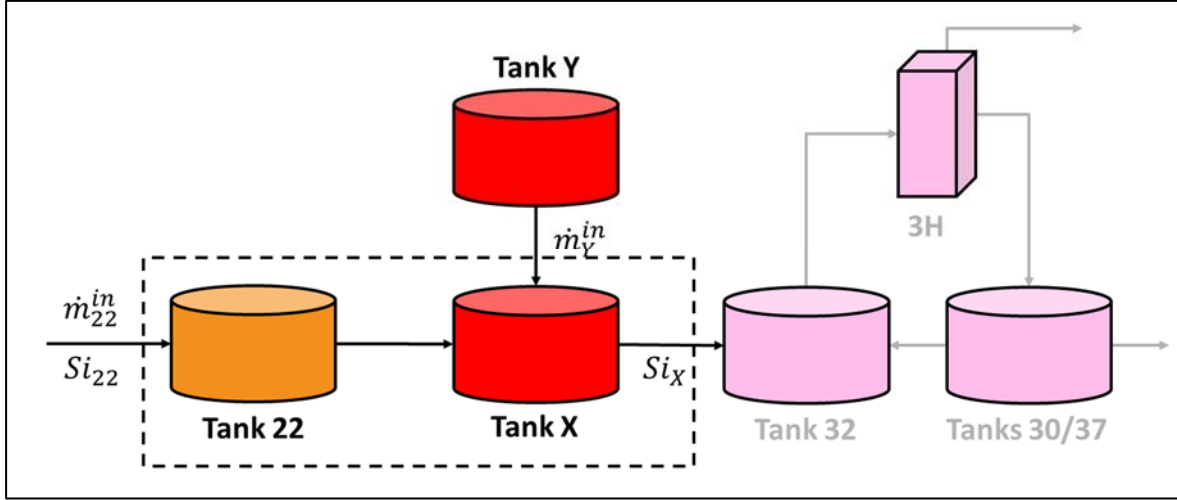
A glycolate concentration factor (with consideration of thermolytic degradation of glycolate) may be calculated according to Equation [7].



$$f_{Gly}^{w/Therm} = \frac{[Gly]_{30}}{[Gly]_{32}} = \frac{\omega_{30}^{gly} \times \rho_{30}}{\omega_{in}^{gly} \times \rho_{32}} \quad [7]$$

### 2.3.2 Modifications for the Holistic Evaporation Model

Additional calculations are required for the Holistic evaporation model. It is necessary to calculate the amount of additional feed material required to dilute Tank 22 material to sufficiently decrease the anticipated silicon concentration to allowable levels. The mass balance necessary for this calculation is shown graphically in Figure 2-5.



**Figure 2-5. Mass Balance Around the Dilution Tanks Prior to Transfer to 3H System.**

The amount of diluent from Tank Y needed to dilute Tank 22 material to a processable level can be calculated according to Equation [8].

$$\dot{m}_Y^{in} = \left( \frac{\rho_X Si_{22}}{\rho_{22} Si_X} - 1 \right) \dot{m}_{22}^{in} \quad [8]$$

The bisection method was used to identify a density  $\rho_X$  such that Equation [8] can be satisfied. Once the mass rate from Tank Y,  $\dot{m}_Y^{in}$ , has been calculated, the composition of Tank X can be calculated according to Equation [9].

$$\omega_X = \frac{\omega_{22} \dot{m}_{22}^{in} + \omega_Y \dot{m}_Y^{in}}{\dot{m}_{22}^{in} + \dot{m}_Y^{in}} \quad [9]$$

The concentration of glycolate in Tank 22 can be calculated with a simple CSTR equation, as shown in Equation [10].

$$\omega_{22}^{gly} = \frac{\dot{m}_{22}^{in} \times \omega_{RCT}^{gly}}{\dot{m}_{22}^{in} + K_{22}} \quad [10]$$

where  $K_{22}$  is the unit-corrected rate constant (in  $\text{kg min}^{-1}$ ) for glycolate thermolysis calculated from the density ( $\rho_{22}$ , in  $\text{g mL}^{-1}$ ), supernatant volume ( $V_{22}$ , in gal), sodium molarity ( $[Na]$ , in  $\text{mol L}^{-1}$ ), nitrate molarity ( $[NO_3]$ , in  $\text{mol L}^{-1}$ ), hydroxide molarity ( $[OH]$ , in  $\text{mol L}^{-1}$ ), and temperature ( $T$ , in K) in Tank 22. The calculation of this rate constant is given in Equation [11].

$$K_{22} = 2.416 \times 10^4 \cdot \rho_{22} V_{22} [Na]^{1.520} [NO_3]^{0.282} [OH]^{1.441} e^{-82,300/RT} \quad [11]$$

Once the concentration of glycolate exiting Tank 22,  $\omega_{22}^{gly}$ , has been calculated, the concentration of glycolate exiting Tank X may be easily solved using Equation [12].

$$\omega_X^{gly} = \frac{\dot{m}_{22}^{in} \times \omega_{22}^{gly}}{(\dot{m}_{22}^{in} + \dot{m}_Y^{in} + K_X)} \quad [12]$$

After these calculations have been performed, the output from Tank X may be assumed to be the input into Tank 32 and Equations [1] through [7] may be used to calculate the concentration across the 3H system.

To calculate a maximum theoretical concentration factor, silicon may be used as a substitute for glycolate (as a non-reactive species). The theoretical concentration factor may be calculated according to Equation [13].

$$f_{Si} = f_{Gly}^{No Therm} = \frac{[Si]_X [Na]_{30}}{[Si]_{22} [Na]_X} \quad [13]$$

A holistic glycolate concentration factor (with consideration of thermolytic degradation of glycolate) may be calculated according to Equation [14].

$$f_{Gly}^{w/Therm} = \frac{[Gly]_{30}}{[Gly]_{RCT}} = \frac{\omega_{30}^{gly} \rho_{30}}{\omega_{RCT}^{gly} \rho_{22}} \quad [14]$$

## 2.4 Quality Assurance

The TTR for this work specifies a functional classification of Safety Class. Requirements for performing reviews of technical reports and the extent of review established in manual E7 2.60 designate a Design Verification as the necessary form of technical review. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.<sup>15</sup>

Modeling of the 3H Evaporator concentration factors was accomplished by performing 50,000 calculations of the concentration factors according to the mathematical structures outlined in this report using randomly-generated initial conditions. These calculations were performed en masse using the Octave 5.2.0 Integrated Development Environment. Octave 5.2.0 has not been evaluated for a software classification for use at SRS, so a subset of calculations was technically reviewed via design verification by alternate calculations (per technical review requirements outlined in E7, 2.60)<sup>16</sup> to ensure accuracy.

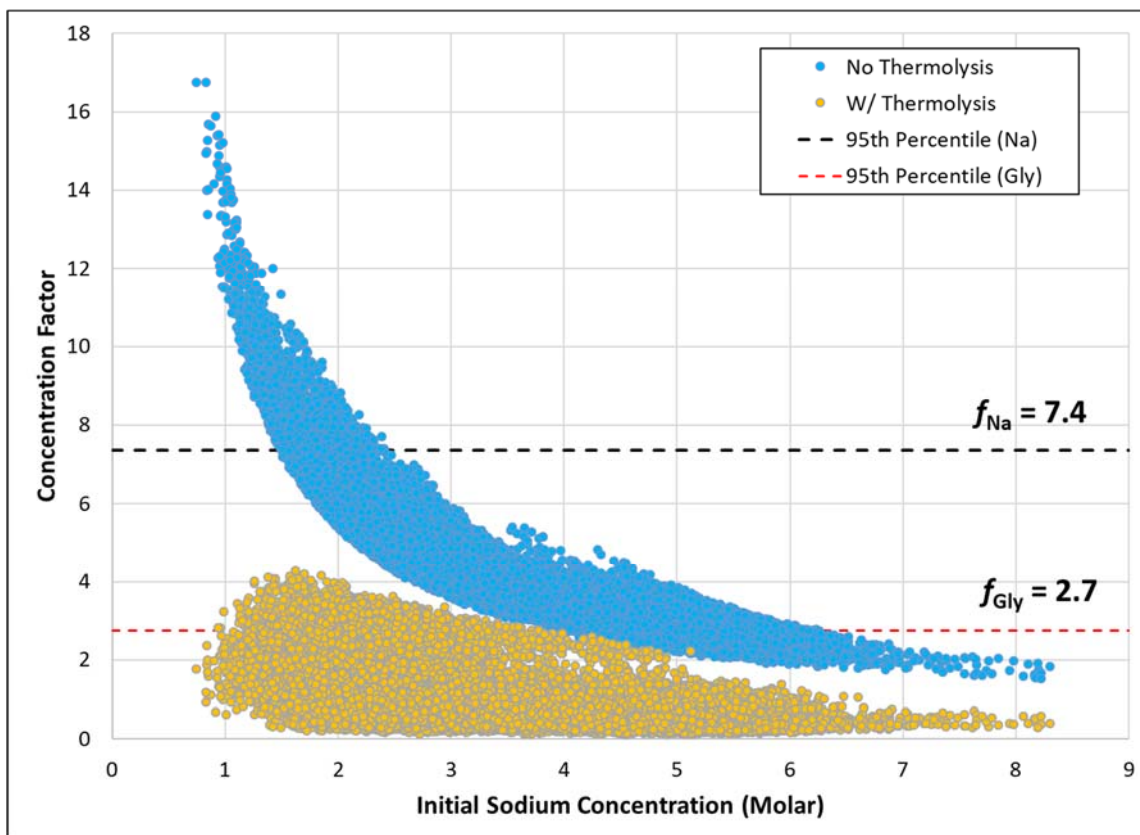
## 3.0 Results and Discussion

This section presents the results of simulations performed for each of the two models described above as well as the calculated concentration factors. Section 3.1 describes the simulation results from the Unrestricted Evaporation Model, which considers transfer of material from an unspecified glycolate-containing tank and does not consider restrictions on silicon concentration. Section 3.2 describes the

simulation results from the Holistic Evaporation Model, which considers DWPf recycle material as the source of glycolate in the CSTF and is assumed to contain measurable silicon, necessitating evaluation of silicon dilution before transfer to the 3H system. Section 3.3 describes the various conservatisms associated with these models.

### 3.1 Results from the Unrestricted Evaporation Model

The results of 50,000 simulations using the Unrestricted Evaporation Model are given in Figure 3-1. Data in blue are sodium concentration factors (which represent theoretical maximum glycolate concentration factors assuming no glycolate degradation) while data in yellow are glycolate concentration factors (calculated assuming thermolytic degradation of glycolate). The concentration factors are plotted against the initial sodium concentration in the evaporator feed stream (plotted along the x-axis).



**Figure 3-1. Concentration Factors from the Unrestricted Evaporation Model.**

The data in Figure 3-1 suggest that 95% of anticipated processing variables (e.g., operating temperatures, solution concentrations, supernatant volumes, etc.) would limit sodium concentration factors to a value of 7.4 (relative to the concentration of sodium in the material being fed to the 3H feed tank). It should be noted that this result is based on the model assumption that the composition of material fed to the 3H system will not vary significantly from the material that has been fed over the past decade. If significant processing changes are made that impact the average feed composition to the 3H Evaporator (i.e., changes to the compositions assumed in Table 2-1), this model should be re-evaluated.

The data in Figure 3-1 also indicate that, despite sodium concentration factors as high as 17, glycolate concentration factors tend to be lower than 4, with 95% of observed glycolate concentration factors falling below 2.7. This suggests that glycolate concentration from any individual stream sent to the 3H system would reasonably be expected to increase by no more than a factor of 2.7.

The apparent difference between the observed glycolate concentration factor (2.7) and the theoretical maximum calculated from sodium (7.4) is larger than the differences predicted in previous 2H evaporator modeling work.<sup>6</sup> This is due primarily to the higher temperatures and salt concentrations inherent to 3H processing leading to higher destruction of glycolate and suggests that glycolate is not as easily concentrated in the 3H system as it is in the 2H system.

The Unrestricted Evaporation Model provides a basis for glycolate concentration control at the point of use (i.e., before transferring to the 3H Evaporator system). The following constraints are placed on the Unrestricted Evaporation Model to ensure consistency with its derivation.

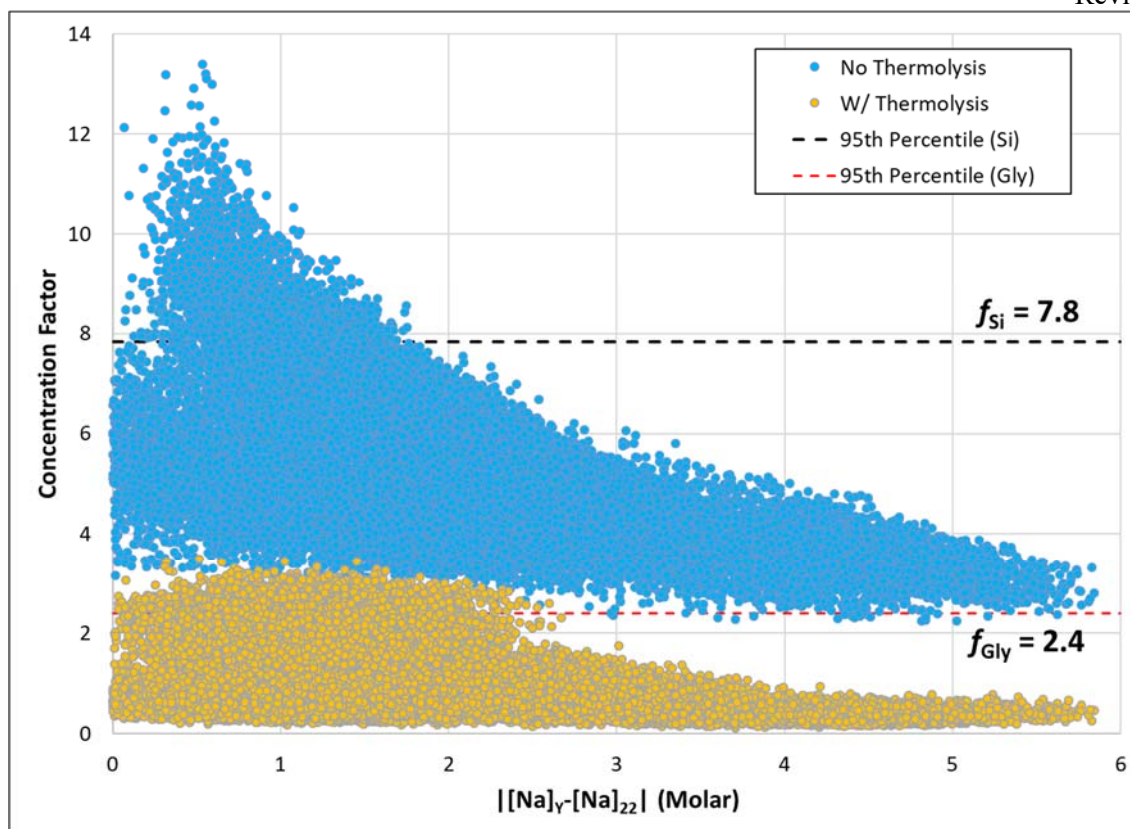
For the Unrestricted Evaporation Model to be considered accurate, these underlying assumptions must be valid:

- Feed to the 3H Evaporator system is assumed to be similar to material transferred to 3H Evaporator tanks over the past decade. A change in average feed composition (i.e., the salt solution compositions typically fed to Tank 32) would require a re-evaluation of this model.
- Feed of glycolate-containing material to the 3H Evaporator system is assumed to originate solely from DWPF recycle material. If additional glycolate-containing waste streams are later incorporated into the CSTF flowsheet, a re-evaluation of this model should be performed.
- The only role of glycolate in chemical reactions throughout the CSTF is assumed to be destruction. If evidence of glycolate-producing reactions is found, a re-evaluation of this model should be performed.

It should be noted that, while this model is an adequate picture of the thermodynamic changes around the 3H Evaporator itself, there are several restrictions on evaporation in the 3H Evaporator that complicate the accurate calculation of expected glycolate concentrations achievable in the 3H system (e.g., the restriction on silicon concentration, which is inherently coupled with DWPF operations). These restrictions are considered in the following section.

### 3.2 Results from the Holistic Evaporation Model

The results of 50,000 simulations using the Holistic Evaporation Model are shown in Figure 3-2. Data in blue are theoretical silicon concentration factors (which represent a theoretical maximum for glycolate if no degradation was assumed) while data in yellow are glycolate concentration factors (calculated assuming thermolytic degradation of glycolate).



**Figure 3-2. Concentration Factors from the Holistic Evaporation Model.**

The concentration factors predicted by the Holistic Evaporation Model are driven primarily by the difference in sodium concentration between Tank 22 material and Tank Y material (rather than feed tank sodium concentration, as in the Unrestricted Evaporation Model), the absolute value of which is plotted along the x-axis. The results in Figure 3-2 suggest that theoretical silicon concentration factors reach as high as 14, but generally fall below 7.8 (95<sup>th</sup> percentile). The value of 7.8 suggests a preliminary dilution by ~50% (upon transfer of Tank 22 material to Tank X) followed by an apparent concentration of ~15x across the 3H Evaporator. This apparent concentration of ~15 is consistent with the notion of feeding more dilute feed material to the 3H Evaporator system.

The observed glycolate concentration factors reach as high as ~3.5 but tend to stay below 2.4 (95<sup>th</sup> percentile). This suggests that glycolate originating from Tank 22 would reasonably be expected to concentrate through the 3H system by a factor of 2.4 or less. Again, this result is markedly different from those seen in 2H modeling efforts, largely because of the higher temperatures and concentrations achievable in the 3H system leading to an elevated rate of glycolate destruction and the requirement to dilute the silicon-rich DWPF recycle material.<sup>6</sup>

The Holistic Evaporation Model provides a basis for glycolate concentration control at the point of addition (i.e., before/after transferring DWPF recycle material to Tank 22). The following constraints are placed on the Holistic Evaporation Model to ensure consistency with its derivation.

For the Holistic Evaporation Model to be considered accurate, these underlying assumptions must be valid:

- DWPF recycle material fed to Tank 22 is assumed to contain relatively high levels of silicon (with respect to waste typically processed in the 3H Evaporator system). While the silicon value assumed in this calculation is the lowest observed in recent Tank 22 samples, the presence of Si at concentrations that require dilution is critical to this model. A processing change leading to a significant decrease in Tank 22 silicon concentration would decrease the amount of dilution

necessary to feed material to the 3H Evaporator system (potentially increasing the possible concentration factor by as much as a factor of 2 or more and would therefore necessitate a re-evaluation of this model.

- The silicon concentration in DWPF recycle material is inherently linked to historic silicon-containing antifoam and frit use in DWPF. If any processing changes are expected to result in significant differences in the influence of antifoam or frit chemistry on silicon concentration, an assessment should be performed upon implementation to confirm the applicability of this model.
- The only role of glycolate in chemical reactions throughout the CSTF is assumed to be destruction. If evidence of glycolate-producing reactions is found, a re-evaluation of this model should be performed.

### 3.3 Model Conservatisms

The models described above include several conservative assumptions. These conservatisms are quantified here.

- In both the Unrestricted and Holistic Evaporation Models, the temperature of evaporation is assumed to be 140 °C.<sup>12</sup> The maximum operating temperature of the 3H Evaporator is expected to be higher than 140 °C during operation. For every 10 °C increase in average temperature, a decrease of 25% is expected in glycolate concentration factor due to the accelerated thermolytic degradation.
  - In the event that operating temperatures fall below 140 °C, the above conservatism should not be credited. For every 10 °C decrease in temperature, an increase of 36% is expected for the glycolate concentration factor.
- In both the Unrestricted and Holistic Evaporation Models, the rate of glycolate degradation is assumed to be equal to the rate of hydrogen generation by glycolate thermolysis (on a molar basis). While this is inherently conservative (the rate of glycolate destruction must be greater than or equal to the rate of formation of product hydrogen), it is unlikely that this conservatism can be credited to decrease the glycolate concentration factor. This is because the glycolate HGR expression is slightly conservative with respect to hydrogen generation (and therefore slightly unconservative for glycolate destruction).<sup>2</sup> The impacts of these differences are likely to negate, resulting in no change to an assumed glycolate concentration factor.
- In both the Unrestricted and Holistic Evaporation Models, the concentration of hydroxide in material fed to the 3H Evaporator system is assumed to be representative of tank sample histories over the last decade.<sup>13</sup> However, transfers of material to the 3H system are often required to contain hydroxide at concentrations greater than 1 M. If this limit is universally imposed on the 3H system, the glycolate concentration factor may reasonably be expected to decrease by 72% (from 2.4 to 0.7). Similarly, the theoretical maximum (assuming no glycolate degradation) may also be expected to decrease by 36% (from 7.8 to 5.0).
- In the Holistic Evaporation Model, the assumed silicon concentration in Tank X (the material intended for feed to the 3H Evaporator system) is assumed to be equal to 78.4 mg/L. The expected silicon concentration in 3H Evaporator feed is expected to be (and has historically been) much less than 78.4 mg/L. For every 10 mg/L decrease in silicon concentration in evaporator feed, at least 7% decrease in glycolate concentration factor is expected.
- In the Holistic Evaporation Model, the assumed silicon concentration in Tank 22 is 135 mg/L. This value is the lowest Tank 22 silicon measurement recorded in recent history (not counting samples following a Tank 51 to Tank 22 transfer). The actual concentration of silicon in Tank 22 may reasonably be expected to be greater than 135 mg/L. For every 50 mg/L increase in Tank 22 silicon concentration, a glycolate concentration factor decrease of 12% may be expected.

## 4.0 Conclusions

The following conclusions are made as a result of the work described in this document.

- The 95<sup>th</sup> percentile of possible glycolate concentration factors across the 3H Evaporator system (Tanks 30, 32, 37, and the 3H Evaporator pot) are 7.4 (assuming no thermolytic degradation) and 2.7 (assuming thermolytic degradation). These concentration factors are expected to be applicable to any feed being transferred into the 3H system with the condition that the feed stream is chemically similar to material fed to the 3H system over the past decade.
- The 95<sup>th</sup> percentile of possible glycolate concentration factors of waste originating from the DWPF Recycle Collection Tank, fed to Tank 22, and proceeding via dilution in other tanks before transfer to the 3H system are 7.8 (assuming no thermolytic degradation) and 2.4 (assuming thermolytic degradation). These concentration factors are expected to be applicable to Tank 22 waste with the condition that the material in question retains silicon compositions similar to those observed over the past decade.
- Glycolate concentration factors across the 3H Evaporator system are expected to be lower than those predicted across the 2H Evaporator system, largely due to the elevated temperatures, residence times, and concentrations achieved in the 3H Evaporator leading to higher rates of thermolytic degradation.

## 5.0 Recommendations

The following recommendations are made as a result of the work described in this document.

- Safety basis calculations for allowable glycolate in 3H Evaporator waste streams should take the concentration factors given above into consideration when calculating the impacts of glycolate thermolysis on flammability.
- Additional studies (including laboratory testing) should be performed to monitor total organic carbon (TOC) degradation to assess if a similar credit for decreased TOC thermolysis could be assumed in 3H evaporation. The degradation of antifoam products is of particular interest given the potential to form methane at high temperatures and should be studied independently.
- Periodic sampling should be performed to confirm the applicability of these models to ongoing radioactive waste processing. In particular, processing changes that may impact the conclusions of these models (e.g., transfers of silicon from frit to recycle material, modifications of silicon concentration in recycle material due to alternate antifoam use) should be assessed upon implementation to ensure consistency with the assumptions described in this document.

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**Distribution:**

[cj.bannochie@srnl.doe.gov](mailto:cj.bannochie@srnl.doe.gov)  
[alex.cozzi@srnl.doe.gov](mailto:alex.cozzi@srnl.doe.gov)  
[a.fellinger@srnl.doe.gov](mailto:a.fellinger@srnl.doe.gov)  
[samuel.fink@srnl.doe.gov](mailto:samuel.fink@srnl.doe.gov)  
[Brenda.Garcia-Diaz@srnl.doe.gov](mailto:Brenda.Garcia-Diaz@srnl.doe.gov)  
[connie.herman@srnl.doe.gov](mailto:connie.herman@srnl.doe.gov)  
[dennis.jackson@srnl.doe.gov](mailto:dennis.jackson@srnl.doe.gov)  
[brady.lee@srnl.doe.gov](mailto:brady.lee@srnl.doe.gov)  
[Joseph.Manna@srnl.doe.gov](mailto:Joseph.Manna@srnl.doe.gov)  
[daniel.mccabe@srnl.doe.gov](mailto:daniel.mccabe@srnl.doe.gov)  
[Gregg.Morgan@srnl.doe.gov](mailto:Gregg.Morgan@srnl.doe.gov)  
[frank.pennebaker@srnl.doe.gov](mailto:frank.pennebaker@srnl.doe.gov)  
[Amy.Ramsey@srnl.doe.gov](mailto:Amy.Ramsey@srnl.doe.gov)  
[William.Ramsey@SRNL.DOE.gov](mailto:William.Ramsey@SRNL.DOE.gov)  
[eric.skidmore@srnl.doe.gov](mailto:eric.skidmore@srnl.doe.gov)  
[michael.stone@srnl.doe.gov](mailto:michael.stone@srnl.doe.gov)  
[Boyd.Wiedenman@srnl.doe.gov](mailto:Boyd.Wiedenman@srnl.doe.gov)  
Records Administration (EDWS)  
[bill.clark@srs.gov](mailto:bill.clark@srs.gov)  
[jeffrey.crenshaw@srs.gov](mailto:jeffrey.crenshaw@srs.gov)  
[james.folk@srs.gov](mailto:james.folk@srs.gov)  
[Curtis.Gardner@srs.gov](mailto:Curtis.Gardner@srs.gov)  
[Pauline.hang@srs.gov](mailto:Pauline.hang@srs.gov)  
[Anna.Murphy@srs.gov](mailto:Anna.Murphy@srs.gov)  
[tony.polk@srs.gov](mailto:tony.polk@srs.gov)  
[Anthony.Robinson@srs.gov](mailto:Anthony.Robinson@srs.gov)  
[mark-a.smith@srs.gov](mailto:mark-a.smith@srs.gov)  
[patricia.suggs@srs.gov](mailto:patricia.suggs@srs.gov)  
[thomas.temple@srs.gov](mailto:thomas.temple@srs.gov)  
[celia.aponte@srs.gov](mailto:celia.aponte@srs.gov)  
[timothy.baughman@srs.gov](mailto:timothy.baughman@srs.gov)  
[earl.brass@srs.gov](mailto:earl.brass@srs.gov)  
[Richard.Edwards@srs.gov](mailto:Richard.Edwards@srs.gov)  
[Azikiwe.hooker@srs.gov](mailto:Azikiwe.hooker@srs.gov)  
[Ryan.McNew@srs.gov](mailto:Ryan.McNew@srs.gov)  
[phillip.norris@srs.gov](mailto:phillip.norris@srs.gov)  
[Christine.Ridgeway@srs.gov](mailto:Christine.Ridgeway@srs.gov)  
[Azadeh.Samadi-Dezfouli@srs.gov](mailto:Azadeh.Samadi-Dezfouli@srs.gov)  
[arthur.wiggins@srs.gov](mailto:arthur.wiggins@srs.gov)  
[Kevin.Brotherton@srs.gov](mailto:Kevin.Brotherton@srs.gov)  
[Grace.Chen@srs.gov](mailto:Grace.Chen@srs.gov)  
[thomas.colleran@srs.gov](mailto:thomas.colleran@srs.gov)  
[robert.hoeppel@srs.gov](mailto:robert.hoeppel@srs.gov)  
[bill.holtzscheiter@srs.gov](mailto:bill.holtzscheiter@srs.gov)  
[Thomas.Huff@srs.gov](mailto:Thomas.Huff@srs.gov)  
[Spencer.Isom@srs.gov](mailto:Spencer.Isom@srs.gov)  
[Vijay.Jain@srs.gov](mailto:Vijay.Jain@srs.gov)  
[Jeremiah.Ledbetter@srs.gov](mailto:Jeremiah.Ledbetter@srs.gov)

[MARIA.RIOS-ARMSTRONG@SRS.GOV](mailto:MARIA.RIOS-ARMSTRONG@SRS.GOV)  
[Wesley.Woodham@srnl.doe.gov](mailto:Wesley.Woodham@srnl.doe.gov)  
[charles.nash@srnl.doe.gov](mailto:charles.nash@srnl.doe.gov)  
[chris.martino@srnl.doe.gov](mailto:chris.martino@srnl.doe.gov)  
[Daniel.Eitreim@srs.gov](mailto:Daniel.Eitreim@srs.gov)  
[Edwin.Ball@srs.gov](mailto:Edwin.Ball@srs.gov)  
[hilary.bui@srs.gov](mailto:hilary.bui@srs.gov)  
[john.iaukea@srs.gov](mailto:john.iaukea@srs.gov)  
[john.occhipinti@srs.gov](mailto:john.occhipinti@srs.gov)  
[William.Satterfield@srs.gov](mailto:William.Satterfield@srs.gov)  
[joseph.copeland@srs.gov](mailto:joseph.copeland@srs.gov)  
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