Optimization of Saltcake Removal Flowsheet at SRS through Incorporation of Testing and In-Tank Waste Experience - 15263

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ABSTRACT

Saltcake removal at SRS may be performed for several reasons: to provide space for evaporator operation (i.e., to precipitate more salt in the drop tank), to provide feed for salt processing (i.e. immobilize the waste), or to remove the salt for tank closure. Many different salt dissolution techniques have been employed in the 40 years that SRS has been performing salt removal, from a basic “Add, Sit, Remove” method (water is added on top of the saltcake and time is allowed for diffusion), to performing interstitial liquid removal, or using mixing devices to promote contact with the liquid. Lessons learned from previous saltcake removal campaigns, in addition to testing and modeling, have led to opportunities for improvements to the overall saltcake removal process. This includes better understanding of salt properties and behavior during dissolution; the primary concerns for salt dissolution are the release of radiolytic hydrogen and criticality prevention (post-dissolution). Recent developments in salt dissolution include the reuse of dilute supernate and a semi-continuous dissolution (SCD) process, where low volume mixing eductors are used to deliver water near the surface of the saltcake at the same rate as the salt solution is removed and transferred to a receipt tank.

INTRODUCTION

Radioactive wastes from the Savannah River Site (SRS) are stored in large underground carbon steel tanks. Fifty-one waste tanks have been in service at SRS, with capacities ranging from approximately 2,800 m³ (gallons) to 4,900 m³ (0.75 to 1.3 million gallons). Since beginning operations in 1954, the Tank Farms have received more than 5.7E+05 m³ (150 million gallons) of waste. Due to a combination of evaporation and disposition the current inventory is approximately 1.4E+05 m³ (37 million gallons) of waste, with a total activity of almost 1E+19 Bq (290 million Curies). Waste is stored in three primary forms: liquid supernate, sludge waste consisting of precipitated metal oxides, and saltcake formed due to evaporator operation. The Liquid Waste mission at SRS involves the safe storage of waste, closure of waste tanks, and immobilization of the waste into grout (low level waste) or glass (high level waste). Saltcake removal is a necessary step to tank closure and waste immobilization, but is also vital to waste storage and processing.

Evaporators are operated in the Tank Farms at SRS in order to concentrate liquid waste and preserve space for waste storage. As the evaporator bottoms streams cool, salt precipitates and forms a hard saltcake on the bottom of the drop tanks. A dense supernatant layer, or liquor, typically remains above the saltcake layer in operating drop tanks, although some tanks may have no supernate cover above the saltcake when not in active use in an evaporator system (due to evaporation in the tank or a decant of the liquor).

Saltcake removal may be performed for several reasons: to provide space for evaporator operation (i.e., to precipitate more salt in the drop tank), to provide feed for salt processing (i.e. immobilize the waste), or to remove the saltcake for tank closure. Many different salt dissolution techniques have been employed, from a basic “Add, Sit, Remove” method (water is added on top of the saltcake and time is allowed for diffusion), to performing interstitial liquid removal, or using mixing devices to promote contact with the liquid. The most recent development in salt dissolution is a semi-continuous dissolution (SCD) process,
where low volume mixing eductors are used to deliver water near the surface of the saltcake at the same rate as the salt solution is removed and transferred to a receipt tank. In general, saltcake is found to dissolve quickly when contacted by dilute supernate or water. The effectiveness of the saltcake removal process has improved over the years and continues to improve through testing, operational experience, and technology.

SRS WASTE TANKS

Construction of waste tanks at SRS began in 1951. A total of fifty one carbon steel waste tanks were built, with the final tank placed into service in 1981. TABLE I shows the distribution of waste tanks by waste type (not counting residual heel volumes). To date, six tanks have been operationally closed and filled with grout; two additional tanks are in preparation for grouting [1]. TABLE II shows the total inventory of each waste type by volume and total activity. Saltcake accounts for nearly half of the total volume in the Tank Farms, but less than 5% of the total activity.

<table>
<thead>
<tr>
<th>Waste Type</th>
<th>Count</th>
<th>Closure Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Saltcake</td>
<td>17</td>
<td>Closure Prep.</td>
</tr>
<tr>
<td>Sludge</td>
<td>16</td>
<td>Closed</td>
</tr>
<tr>
<td>Saltcake &amp; Sludge</td>
<td>6</td>
<td>Precipitate</td>
</tr>
<tr>
<td>Supernate</td>
<td>3</td>
<td>Total</td>
</tr>
</tbody>
</table>

There are four major tank design types, as can be seen in Figure 1. The twenty-four “Old Style” tanks do not meet all current Federal requirements for secondary containment, and welds were not stress relieved during construction. Of the six tanks that have been closed, four were Type IV tanks, and two were Type I tanks. One additional Type I tank and one Type II tank are in preparation for grouting and final closure now.

<table>
<thead>
<tr>
<th>Waste Type</th>
<th>Volume (m³)</th>
<th>Activity (Bq)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid</td>
<td>68,138</td>
<td>4.63E+18</td>
</tr>
<tr>
<td>Sludge</td>
<td>10,296</td>
<td>5.00E+18</td>
</tr>
<tr>
<td>Saltcake</td>
<td>61,324</td>
<td>4.48E+17</td>
</tr>
<tr>
<td>Total</td>
<td>139,758</td>
<td>1.01E+19</td>
</tr>
</tbody>
</table>
Figure 1. SRS Waste Tanks. Number of each type, capacities, and years of construction are shown. Type III tanks have insertable or deployable cooling coils, such as shown on the left of the tank, and Type IIIA tanks have permanently installed coils, such as shown on the right of the tank.

Type I and II tanks have a 1.5 m annulus pan, and an extensive array of installed cooling coils. Type I tanks are 23m in diameter, have twelve support columns, and are covered by 2.7m of earth. Type II tanks are 25.9m in diameter, have a 2.1m center column and no earth overburden.

Type IV tanks are primarily used for low heat waste, and have a domed roof, no cooling coils, and have a leak detection system, but no secondary containment. They are 25.9m in diameter, with no internal supports.

The Type III/IIIA have full secondary containment and stress relieved welds. They are 25.9m in diameter with a 2.1m center column, and are essentially the same design, with a few exceptions: Type III tanks use insertable or deployable cooling coils installed through tank risers, where Type IIIA tanks have bottom-supported installed cooling coils (both types of cooling coils are shown in Figure 1). Type IIIA tanks have improved airflow around the primary tank as well.

SRS SALTCAKE

The Evaporator Systems operate at typical temperatures in the range of 110°C to 145°C; the Bottoms stream, hot and near saturation with dissolved salts, is delivered to a drop tank where the bulk temperature is less than 100 °C. Salts precipitate as the liquid cools and form saltcake or gather on cooling coils. The saltcake is porous, with a void fraction of approximately 40%; wet saltcake contains approximately 30% liquid by volume, and the balance of the void space may be occupied by trapped gas (a portion of which may be hydrogen produced via radiolysis). The salt solids are typically low-activity, but a small amount of high-activity sludge particles may be trapped within the saltcake matrix. In addition, the supernate and interstitial liquid typically contains significant concentrations of highly-soluble Cesium-137; concentrations of 5E+13 Bq/m³ (5 Ci/gal) are typical. Saltcake at SRS is very strong, with yield strength of around 1500 Pa for wet saltcake and several thousand Pascal for dry saltcake [3].
Figure 2. Dry Saltcake Prior to Dissolution in Tank 10 (left) and “Christmas Tree” Saltcake Formation on Tank 3 Cooling Coils Following Interstitial Liquid Draining (right)

Saltcake Composition

Salts precipitate in accordance with the chemistry of the Evaporator Bottoms and the temperature profile within the drop tanks; saltcake composition can vary significantly between waste tanks and even between locations within a single waste tank. Early efforts were made to establish a general saltcake composition for flowsheet modeling; these were based on limited data and calculations based on supernate composition. Since that time, more extensive saltcake data has been obtained, so that the overall average composition of undissolved saltcake can be more accurately estimated. Predicted compositions are shown in TABLE III and TABLE IV. The dominant single salt species in SRS saltcake is sodium nitrate.

**TABLE III. General SRS Saltcake Composition [4]**

<table>
<thead>
<tr>
<th>Chemical Compound</th>
<th>Saltcake (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaNO3</td>
<td>86</td>
</tr>
<tr>
<td>Na2CO3·H2O</td>
<td>5.7</td>
</tr>
<tr>
<td>NaNO2</td>
<td>0.82</td>
</tr>
<tr>
<td>NaAlO2·2H2O</td>
<td>2.2</td>
</tr>
<tr>
<td>Na2C2O4</td>
<td>0.45</td>
</tr>
<tr>
<td>Na2SO4</td>
<td>2.9</td>
</tr>
<tr>
<td>NaCl</td>
<td>0.0068</td>
</tr>
<tr>
<td>NaF</td>
<td>0.17</td>
</tr>
<tr>
<td>NaOH</td>
<td>0.73</td>
</tr>
<tr>
<td>Na3PO4</td>
<td>0.59</td>
</tr>
<tr>
<td>Total Na</td>
<td>28</td>
</tr>
</tbody>
</table>

The salts listed in TABLE III are based on digestion of saltcake, and so appear as single salt species. X-Ray Diffraction Spectroscopy analysis shows that these salt species often form more complex double and triple salts or aluminosilicates; some of these present challenges to dissolution (e.g. burkeite, which is less soluble at higher temperatures), while others are insoluble and cannot be removed at all by typical salt dissolution processes. The compositions in TABLE III and TABLE IV are useful for modeling purposes where limited data is available; however, tank-specific data is preferred to give the most accurate results for flowsheet development.

**TABLE IV. General Concentration of Radionuclides in SRS Saltcake [4]**

<table>
<thead>
<tr>
<th>Radionuclide</th>
<th>Bq/m³</th>
<th>Ci/gal</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-14</td>
<td>2.23E+08</td>
<td>2.29E-05</td>
</tr>
<tr>
<td>Sr-90</td>
<td>3.48E+12</td>
<td>3.56E-01</td>
</tr>
<tr>
<td>Y-90</td>
<td>3.48E+12</td>
<td>3.56E-01</td>
</tr>
<tr>
<td>Cs-137</td>
<td>1.55E+11</td>
<td>1.59E-02</td>
</tr>
<tr>
<td>Ba-137m</td>
<td>1.47E+11</td>
<td>1.50E-02</td>
</tr>
<tr>
<td>U-235</td>
<td>1.37E+05</td>
<td>1.40E-08</td>
</tr>
<tr>
<td>U-238</td>
<td>3.07E+06</td>
<td>3.14E-07</td>
</tr>
<tr>
<td>Pu-238</td>
<td>4.44E+10</td>
<td>4.54E-03</td>
</tr>
<tr>
<td>Pu-239</td>
<td>1.48E+09</td>
<td>1.51E-04</td>
</tr>
</tbody>
</table>
DISSOLUTION TECHNIQUES

Several salt dissolution techniques have been used at SRS, which can be generally placed into one of the following categories. As can be seen in the later section on Salt Dissolution History, actual implementation can combine aspects of several techniques, either by design or due to operational challenges.

Add, Sit, Remove / Molecular Diffusion (ASR / MD)

In this method, dissolution water (DW) is added to a tank with some amount of existing supernate above the saltcake. This method relies on concentration gradients within the tank to promote contact between the saltcake and the water. This method is slow, inefficient, and tends to result in a saturated layer on top of the saltcake that allows little contact of the saltcake with dissolution water.

Modified Density Gradient (MDG)

In the Modified Density Gradient method, the transfer pump/jet is mined to a low position in the tank. Dissolution water is added on top of the remaining supernate. The best results with this method are achieved by maintaining the liquid level close to the saltcake surface in a steady-state “feed and bleed” type of operation (or in small batches in and out that approximate such a process) [5]. Larger volumes of supernate can result in saturated layers above the saltcake, and the dissolution water tends to travel across the saltcake and down into the salt well, such that the pump removes more dilute material.
**Drain, Add, Sit, Remove (DASR)**

DASR is a special type of MDG with interstitial liquid removal. In this method, a pump/jet is mined into the saltcake and the saturated interstitial liquid is removed. Dissolution water is added to the saltcake and allowed to sit for some time and then removed again. This method promotes excellent contact with the saltcake, and the wait time is minimal. However, the pump suction elevation is the only mechanism for controlling the release of hydrogen resulting from interstitial liquid removal; this requires the pump to be mined between stages, or the tank must enter Gas Release Mode (GRM) as described in the section below on Flammability.

![Diagram of DASR method]

**Agitation**

Agitation has been employed in conjunction with adaptations of the above methods to attempt to improve contact between the saltcake and the dissolution water. Slurry pumps, mixers, and steam jet recirculation have all been used for salt dissolution at SRS. This is further covered in the Discussion of dissolution in Tank 22 and Tank 19.

**Semi-Continuous Dissolution (SCD)**

The most recent approach to bulk salt dissolution has been successfully used for two campaigns since 2010, and is planned for a third campaign this year. SCD adds water at a low flow rate (approximately 0.11m³/min or 30 gpm) at the same rate as dissolved salt solution is removed. Low volume mixing eductors are used to add the dissolution water to multiple locations in the tank to maximize contact with saltcake. The water being added through the eductors entrain the supernate surrounding the nozzle which mixes the water with the free liquid in the tank, preventing stratification and distributing the water across the surface of the saltcake [6]. The Dissolution Water Skid (DWS) controls the water addition to within the limits allowed by the Flammability program, while allowing the process to operate continuously. The water is added via two 11 m³ (3,000 gallon) tanks; a programmable logic controller (PLC) empties one tank, then swaps to the other tank as the first tank is filled. Among the methods where interstitial liquid is not removed from the saltcake, this method has achieved excellent results [7].
SAFETY CONCERNS DURING SALT DISSOLUTION

Flammability

As previously mentioned, up to 11% of the saltcake volume can be trapped gas. A portion of this trapped gas is radiolytic hydrogen; the actual composition depends on the inventory of radionuclides within the tank as well as the chemical makeup of the interstitial liquid (i.e. concentration of nitrite and nitrate ions, which act as hydrogen scavengers). Concentrations of less than 35% hydrogen by volume are typical in the saltcake trapped gas.

Radiolytic hydrogen is generated over time in liquid waste. Waste tanks are classified based on the time in which the radiolytic hydrogen could accumulate in the tank vapor space such that the lower flammability limit (LFL) could be reached upon loss of ventilation (starting from an assumed initial concentration equal to the Safety Analysis Value, or SAV).
Trapped gas is released from saltcake due to dissolution of the salt, removal of supernate or interstitial liquid, or agitation of the solids (by intentional mixing or due to a seismic event). Prior to performing an activity that has the potential to release a significant amount of trapped hydrogen, the activity must be evaluated to show that the release is acceptable (e.g. does not exceed the SAV). This evaluation is performed using conservative assumptions for hydrogen release volume, temperature effects on LFL, hydrogen release rate, and vapor space volume. Alternatively, the tank may perform salt dissolution or agitation in Gas Release Mode (GRM); this mode allows for assumptions that are not as conservative in exchange for additional controls, such as constant hydrogen monitoring and safety class interlocks. Tank 41 and Tank 25 have dissolved saltcake while in GRM.

Flammability requirements are generally met for salt dissolution by controlling the liquid addition volume; after adding a calculated batch volume of water or dilute supernate, subsequent additions cannot be added until the vapor space is verified to meet the initial conditions in the evaluation (e.g. hydrogen concentration < 2.5% LFL). The batch volume is controlled by the capacity of the water source. The PLC and flow totalizer on the DWS used during the SCD process meet this requirement in a way that minimizes the impact to operations while allowing the process to continue to run in a semi-continuous fashion. SCD allows for efficient dissolution while avoiding GRM controls, which can be costly and labor-intensive to implement.

Corrosion

The Corrosion Control Program at SRS establishes limits on both chemistry and temperature within the waste tanks to limit corrosion. In general during salt dissolution, nitrate concentrations in the supernate increase, and hydroxide and nitrite concentrations decrease. In these conditions, Stress Corrosion Cracking is a potential concern. Salt dissolution temperatures are typically required to be less than 50°C to reduce the potential for cracking. Salt dissolution is an endothermic process, and the initial temperatures are typically less than 50°C for saltcake and 30°C for the dissolution water or supernate, so temperature requirements are often met without any additional operational constraints.

During active salt dissolution, the supernate chemistry and the air-liquid interface are both constantly changing; the tank chemistry may not meet chemistry requirements throughout salt dissolution. If the salt dissolution process ends or is stalled, both the dissolution tank and the receipt tank must be sampled and brought back into strict adherence with the Corrosion Control Program chemistry limits within 45 days. This is usually accomplished by adding sodium hydroxide, either by a transfer of concentrated supernate or by adding chemicals via a tanker or tote.

The following improvements in the Corrosion Control Program have helped to optimize the salt dissolution flowsheet:

- Supernate chemistry is predicted as part of flowsheet development to better manage corrosion and temperature limits. This is improved by better predicting the saltcake composition.
- Corrosion mitigation after completion or suspension of dissolution has been improved by matching the specific gravity of the chemical treatment with the surface chemistry of the treatment tank. Previous attempts to add concentrated hydroxide solution (ρ ≈ 1.5) to sub-saturated salt solution (ρ ≈ 1.25) have, in some cases, resulted in the heavier solution forming a stratified layer deep in the tank without adequately conditioning the liquid surface.
- An “old-style” tank may be placed into the Cleaning Activities Lifecycle to suspend chemistry requirements for salt dissolution; this allows up to four years for the latter stages of the tank closure process (this includes all cleaning activities for that tank). This allows operational
flexibility at the cost of a significant time limitation. This has not been employed during salt dissolution at SRS.

Criticality

Fissile material occurs in saltcake in two ways; it is either a result of small amounts of insoluble sludge that were entrained through the evaporator system (controls are in place to limit the entrainment of sludge solids into the evaporators, but a small amount can potentially be carried through), or it is a result of dissolved fissile isotopes that precipitate in the drop tank. Prior to performing saltcake removal in a waste tank, the planned operations must be evaluated and shown to not pose a criticality concern. This is currently implemented by tailoring the criticality evaluation to a specific tank and process. Criticality safety strategies for salt dissolution include [8]:

- A waste tank can be shown to not pose a criticality safety concern based on process knowledge of the saltcake fissile inventory. For example, an analysis of transfer histories can be used to show that a tank contains a limited inventory of low enrichment fissile material; such that no normal or credible abnormal upset conditions in the salt dissolution process would result in a criticality.
- A waste tank can be shown to not pose a criticality safety concern based on the presence of neutron poisons during the bulk salt dissolution/agitation process. The type of poison (e.g., iron, manganese) credited in the criticality evaluation may differ by waste tank. This method requires verification by sampling.
- A waste tank can be shown to be subcritical using an areal density argument based on process knowledge of the tank contents to demonstrate subcriticality of the proposed saltcake dissolution in the tank. Verification by sampling may be required for certain waste streams.

Radiological Control

The primary radiological concern during saltcake removal activities is gamma radiation exposure; interstitial liquid associated with saltcake can have high concentrations of Cs-137. This can be largely mitigated for waste tanks that have viable transfer lines below grade – the ground provides radiation shielding. However, many of the “old-style” waste tanks have installed transfer lines that have not been used in decades and cannot be qualified for use. Temporary above ground transfer lines will be used for these tanks. The recent campaign in Tank 10 used three layers of lead blankets on the above-grade hose-in-hose transfer line in addition to installed fencing to prevent personnel access. These measures were taken based on a calculated maximum Cs-137 concentration of 1.1E13 Bq/m³ (1.13 Ci/gal) without interstitial liquid removal; had interstitial liquid removal been performed, the Cs-137 maximum concentration could have been as high as 4.0E13 Bq/m³ (4.07 Ci/gal) [9]. The final concentration at the end of salt dissolution was approximately 1.56E12 Bq/m³ (1.6 Ci/gal) [10]; unshielded dose rates at 30 cm reached a maximum of approximately 1 mSv (100 mrem) [7].
Waste transfers at SRS are placed into two categories with respect to inhalation dose potential (IDP) for transfer accident analysis. Transfers with an IDP above 5.28E8 Sv/m³ (2.0E8 rem/gal) are considered High-Rem, and lower doses are considered Low-Rem. High-Rem transfers are not permitted in many facilities and transfer lines, and have increased requirements for instrumentation, surveillances, equipment to isolate and terminate the transfer, and ventilation. Salt dissolution transfers to date have been well below this limit and have been Low-Rem; significant sludge content is necessary to require High-Rem classification. Future processing of tanks with a combination of sludge and saltcake will have to be evaluated to determine whether this limit is challenged, but it is unlikely that saltcake removal transfers will ever be classified as High-Rem.

**SRS SALT DISSOLUTION HISTORY, TESTING, AND IMPROVEMENTS**

**Tank 22 Salt Dissolution, ASR with Steam Recirculation (1971-1974)**

The first two salt dissolution campaigns were performed in Tank 22, which is a Type IV Tank with no cooling coils. Dilute waste was initially added to the tank and allowed to sit, then steam jets were used to recirculate the supernate (thus increasing the temperature as well as adding water as the steam condensed). This campaign removed approximately 1,000 m³ (273,000 gallons) of saltcake at a rate of dissolution not achieved since in the Tank Farms (14.5 m³/hr) [11]. The high dissolution rate was a result of the combination of agitation, high temperatures and added water. The tank reached 90°C, and required 24 months of cooling before the dissolved salt solution could be removed; Tank 22 has no cooling capacity other than air circulating through the top of the tank. It is estimated that the cooling duration would have been 10 months in a Type I tank with cooling [12]. In addition to transfer restrictions, the elevated temperatures and dissolved nitrate increased the risk of stress corrosion cracking of the tank wall. No tank has used steam jet recirculation since.

Following Tank 22 salt dissolution, a series of studies were employed to determine the best method for saltcake removal. Bench-scale dissolution tests compared density-driven salt dissolution (MDG), steam jet recirculation, and mechanical agitation. Demonstration tests were performed using MDG in Tank 10 (Type I) and mechanical agitation in Tank 19 (Type IV) for comparison of the methods as well as comparison of laboratory tests to in-tank results. Agitation in Tank 19 was accomplished initially by a single long-shafted Bingham-Willamette 112 kW (150 hp) slurry pump; a second slurry pump was installed for the last dissolution batch (out of four total batches). Lab results were found to agree closely with the full-scale demonstrations. Both techniques achieved approximately the same specific gravity (1.38 for Tank 10, 1.40 for Tank 19), so the amount of saltcake removed per gallon of water was similar. The MDG method doesn’t prevent overheating issues and is simple to implement compared to mechanical agitation, but it was found to leave insoluble material behind as it dissolves, which could build up on top of the saltcake to the extent that it would inhibit or prevent further dissolution using that method. Mechanical agitation was recommended for salt dissolution due to a better overall rate of dissolution and because of the potential build-up of insolubles with MDG [12].

Salt dissolution using mixing devices promotes contact, and does increase dissolution rates (Tank 24 also used mechanical agitation, and achieved similar results to Tank 19 [11]). However, there is significant cost to install and maintain mixing devices.

Tank 33, DASR (1982)

Bulk salt dissolution by DASR method in Tank 33 was performed in December 1982. The saltcake was drained, and then low-heat supernate was added from Tank 47 (via tank 26) [13]. The supernate was allowed to sit for 9 days and was then transferred back to Tank 26. Approximately 427 m³ (113,000 gal) of saltcake was dissolved at a rate of 2.0 m³/hr [11].

Further Study and Ongoing Salt Dissolution

In 1996, testing was conducted to compare DASR, MDG, and a proposed method called Continuous Salt Mining. Continuous Salt Mining was similar to MDG, but the dissolution water was added through a downcomer mined into the saltcake. This experiment found that all three methods achieved similar chemistry and density. Critical factors in improving salt dissolution efficiency were found to be:

- Suction depth – For these techniques, deeper suction depths promote better contact with the saltcake. If the suction depth is too shallow, short circuiting of the dissolution water results in poor dissolution.
- Removal rate – Faster removal rates also can cause short circuiting of the dissolution water and affects the amount of interstitial liquid that can be removed via DASR due to limited flow through the saltcake matrix. For a continuous “feed and bleed” type of operation, adjusting the outlet rate to maintain a consistent liquid level is important.
- Liquid level – Maintaining the liquid level just above the saltcake is optimal. Excess liquid tends to reduce efficiency due to stratification. If the liquid level is not maintained above the saltcake level, salt accumulation may remain on tank structures (primarily cooling coils) above the liquid level, and will require that the tank be filled again to cover the accumulated salt (or spraying/lancing activities if the accumulations are in a limited, accessible area) [5].

A study conducted in 2001 compared MD, MDG, and agitation (using one or more 5.6 kW / 7.5 hp Flygt 4650 mixers or a single slurry pump such as used in Tank 19) in preparation for salt dissolution in Tank
37. The stated objective was to dissolve 1.27 m (50 in.) of saltcake in Tank 37 within 6 months. This study found that MD was unlikely to be successful. Dissolution via MDG was estimated to be capable of dissolving salt faster than using a single Flygt mixer; multiple Flygt mixers or a single slurry pump increase dissolution rates [14].

Based on the testing performed, and the process history of salt dissolution, MDG and DASR were well established as the preferred salt dissolution methods. While agitation does promote contact with saltcake and increases the dissolution rate, this is offset by the time and cost to install and maintain the agitation equipment during bulk salt dissolution. However, as salt dissolution approaches the final stages in a given tank, residual insoluble solids build up and relatively shallow suction depths may hinder further dissolution; mixing devices will likely be required at this point to remove the heel from the tank. In general, salt dissolution kinetics were found to only be a minor factor in the overall rate in which saltcake is dissolved from a tank; plant operational steps (e.g. sampling, water addition, supernate removal, jet/downcomer mining) were found to have a larger impact on project schedules [11].

Salt dissolution campaigns in the last decade were performed by MDG/DASR exclusively until the SCD process was developed. TABLE V summarizes the results of these campaigns [15]. Operational difficulties proved to be a much larger factor in the success of these campaigns than dissolution kinetics. Gas Release Mode was implemented for Tank 41 and Tank 25, primarily to support interstitial liquid removal.

**TABLE V. Salt Dissolution Campaigns using MDG, 2005-2015 [15]**

<table>
<thead>
<tr>
<th>Campaign</th>
<th>Method</th>
<th>Saltcake Dissolved</th>
<th>Water:Saltcake Volume Ratio</th>
<th>Dissolution Rate</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tank 37</td>
<td>2005 MDG</td>
<td>731</td>
<td>2.52</td>
<td>0.287</td>
<td></td>
</tr>
<tr>
<td>Tank 41</td>
<td>2005 DASR/MD</td>
<td>478</td>
<td>1.93</td>
<td>0.0317</td>
<td>Batch 1 dissolution water sat in Tank 41 for almost 2 years prior to transfer out</td>
</tr>
<tr>
<td></td>
<td>2005 Batches 2 and 3</td>
<td>MDG</td>
<td>458</td>
<td>2.27</td>
<td>Batches 2 and 3 had transfer interruptions, which more than doubled the intended sit time</td>
</tr>
<tr>
<td></td>
<td>2008 MDG</td>
<td>624</td>
<td>1.49</td>
<td>0.329</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2011 MDG/MD</td>
<td>306</td>
<td>3.88</td>
<td>0.0677</td>
<td>Beneficial reuse program</td>
</tr>
<tr>
<td>Tank 25 cumulative</td>
<td>2008 MDG with Recirculation</td>
<td>2,285</td>
<td>1.56</td>
<td>0.112</td>
<td>Delays occurred due to problems with the transfer pump and difficulty flushing the IAL</td>
</tr>
<tr>
<td></td>
<td>2008 DASR</td>
<td>306</td>
<td>3.19</td>
<td>1.59</td>
<td>Interstitial draining was performed prior to water addition</td>
</tr>
<tr>
<td></td>
<td>2009 MDG with Recirculation</td>
<td>784</td>
<td>1.88</td>
<td>0.134</td>
<td>Line flush occurred during Batch 2</td>
</tr>
<tr>
<td></td>
<td>2009 MDG with Recirculation</td>
<td>837</td>
<td>1.19</td>
<td>0.459</td>
<td>Batch 3 likely counts dissolution from Batch 2; values are not representative</td>
</tr>
<tr>
<td></td>
<td>2010 MDG with Recirculation</td>
<td>664</td>
<td>1.50</td>
<td>0.254</td>
<td></td>
</tr>
</tbody>
</table>
These calculated low dissolution rates are due to process delays caused by equipment or operational problems, and are not an accurate reflection of actual dissolution rates.

Batch 1 water addition volume was not completely removed, and more water was added between the batches, contributing to dissolution. Batches 1 and 2 were combined to present more accurate values.


Salt dissolution requires a significant amount of water or dilute supernate: as seen in TABLE V, approximately twice the volume of liquid is needed to dissolve a given volume of saltcake. Flammability controls restrict direct supernate transfers from one tank to another for dissolution. Addition of fresh water is typically used, but this can add thousands of cubic meters of liquid volume into the system, which puts a strain on Tank Farm capacity and evaporator operation.

The Defense Waste Processing Facility (DWPF) receives sludge from the Tank Farms for vitrification. As a result of the process, a dilute waste Recycle stream is returned to the Tank Farm. This is done in small batches limited by the size of the Recycle Collection Tank, which has a capacity of approximately 30 m³ (8,000 gallons). This material typically is sent to the Tank Farms and eventually processed through an evaporator. The nature of this process lends itself perfectly for integration with salt dissolution because of the limited batch size and because it does not add volume into the system.

In 2011, Tank 41 began salt dissolution using DWPF Recycle. This campaign did not remove interstitial liquid; the intent was to use MDG with a transfer pump to recirculate liquid through the saltcake between Recycle receipts to facilitate contact with the saltcake. However, the pump failed and recirculation was not performed, so fresh Recycle mostly accumulated in the tank above a layer of saturated salt solution. The campaign was not very efficient; the water to saltcake ratio was relatively high, and the specific gravity was less than 1.2 [10]. Flowsheet modeling predicted salt dissolution performance similar to water, so the low specific gravity and high dissolution ratio are likely due to the lack of recirculation within the tank. Because this campaign did not add additional liquid to the system, low efficiency was less of a concern. DWPF Recycle is planned for use in future salt dissolution campaigns, where possible (some locations, such as Tank 10 and Tank 37, have transfer restrictions and cannot receive DWPF Recycle as currently configured).

Semi-Continuous Dissolution (SCD) – Tank 37 (2010) and Tank 10 (2013)

Tank 37 was used to demonstrate the effectiveness of SCD for salt dissolution, as well as recover space for continued evaporator operation. Salt dissolution recovered over 848 m³ (224,000 gal) of tank space in 26 days of operation. The process was very efficient, and achieved a dissolution rate that is as fast as any previous campaign other than in Tank 22 where steam recirculation was used. Modeling predicted a specific gravity of 1.54 at 90% efficiency; the average recorded during transfers was 1.59.

Tank 10, after sitting essentially untouched for nearly 30 years, resumed salt dissolution in 2013 using SCD. Tank 10 differed from Tank 37 in several ways:

- Tank 10 was a dry, inactive tank with no free supernate at the start of salt dissolution
- Tank 10 required no flammability controls for salt dissolution
- Tank 10 did not have a jet mined into the saltcake – it used a transfer pump that was installed above the saltcake and moved down as dissolution progressed
- Although both Tank 10 and Tank 37 had previously undergone salt dissolution, Tank 37 had accumulated new saltcake; Tank 10 dissolved old saltcake where a significant amount of the high-solubility salts had already been removed
During dissolution in Tank 10, a saltcake mound was discovered that was above the elevation of the majority of the bulk saltcake; the liquid level was raised to cover this mound, which resulted in a thicker liquid layer over much of the tank. Tank 10 was estimated to have a specific gravity of 1.38 at 90% efficiency [16]; the observed average specific gravity puts it slightly under 90%. The dissolution rate in Tank 10 was likely affected by the time required to cover the saltcake mound, but is still one of the better dissolution rates seen in modern dissolution campaigns. Even with the differences in characteristics and operations between the campaigns in Tank 37 and Tank 10, the water to dissolved saltcake ratio is the same.

<table>
<thead>
<tr>
<th>Campaign</th>
<th>Average Specific Gravity</th>
<th>Saltcake Dissolved</th>
<th>Water:Saltcake Volume Ratio</th>
<th>Dissolution Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tank 37[^1]</td>
<td>2010</td>
<td>1.59</td>
<td>848</td>
<td>2.08</td>
</tr>
<tr>
<td>Tank 10[^2]</td>
<td>2013</td>
<td>1.35</td>
<td>182</td>
<td>2.08</td>
</tr>
</tbody>
</table>

The success of SCD is partially due to the physical attributes – creating a plume of dilute water near the saltcake, while removing concentrated salt solution. However, the larger benefit is that once the system is running (flows established), there are few operational steps to perform and therefore fewer opportunities for delay.

**FUTURE OF SALT DISSOLUTION AND SALT TANK CLOSURE**

This section highlights future plans for saltcake removal from waste tanks, in addition to obstacles that may need to be overcome or improvements that are not yet implemented.

- Tank 37 is in preparation for another salt dissolution campaign using SCD that will remove 1,400 m³ (371,000 gallons), equivalent to a depth of 2.5 m (100 in.), of saltcake to recover space for use as a drop tank in the evaporator system. Periodic dissolution campaigns in Tank 37 and the other drop tanks will be necessary for much of the remainder of the life of the Tank Farms to maintain space for evaporator operation. SCD will likely be used in tanks that cannot receive DWPF Recycle.
- SCD will continue to be used to dissolve saltcake from Tank 10 to support future salt batches and move the tank towards closure. It is likely that Tank 10 salt dissolution will conclude in Tank 10 with some amount of insoluble salt and sludge on the bottom of the tank; this will likely require installation of mixing devices to complete waste removal prior to closure. No tank has undergone saltcake removal through closure under the current Safety Analysis; criticality safety will have to be ensured for the mixture of low solubility salts and sludge. The heel will have to be sampled and a cleaning strategy developed. Many of the “old-style” salt tanks will execute salt removal and closure based on successes and lessons learned from Tank 10.
- Tank 41 will continue dissolution using DWPF Recycle. Tank 41, in addition to other Type III salt tanks, will serve as a hub tank once the saltcake is removed.
- Strategies for waste removal from salt tanks with a significant sludge layer will have to be developed. There are six saltcake tanks that contain a sludge layer of more than 35 m³ each. Three of these tanks have sludge on top of the saltcake layer. At least some portion of waste removal from these tanks will be a combination of saltcake and sludge removal using mixing devices.
• Salt dissolution via tank-to-tank transfer can be beneficial in the reuse of dilute supernate for dissolution. Current flammability limitations make dissolution in this fashion cost prohibitive. Future plans are to refine the Flammability Program and salt dissolution strategies to facilitate this process.

CONCLUSION

SRS has more than 40 years of experience dissolving saltcake. Laboratory testing, in-tank experience, and modeling have optimized the process in that time; the following parameters are important to consider when evaluating the performance of a salt dissolution campaign:

• Dissolution Efficiency – Amount of saltcake dissolved vs amount of water added. This can be monitored by a combination of solution specific gravity and the volumetric ratio of water to saltcake.
• Dissolution Rate – Saltcake dissolved (removed) over time. The actual rate of dissolution of salt as driven by chemistry and mass transport is important, and is typically relatively quick unless stratification occurs. The more useful parameter is the rate of saltcake removed from the tank, accounting for all operations from the addition of water to removal of the dissolved salt.
• Tank Farm Capacity Impacts – Net effect to freeboard in the Tank Farms. In general, saltcake occupies half the volume of the salt solution that results from dissolution. Dissolved salt solution is processed for disposal, but it must be stored while a Salt Batch is assembled, adjusted, and qualified for feed. Using fresh water has an initial net negative effect on capacity until the solution can be processed. Using existing dilute supernate (i.e. DWPF Recycle) increases capacity since the supernate used is already occupying space in the Tank Farms.
• Reduction of Interferences – Interruptions, process upsets, transfer conflicts, and equipment failures can all add significant time to a salt dissolution project. Many interferences occur during transition from operational step to another; operating in a continuous fashion (such as with SCD) can reduce the opportunities for delays to occur.

The following attributes are important to improving bulk salt dissolution operations:

• Maintain the liquid level just above the top of the saltcake. Benefit: promote contact and reduce short circuiting, which will increase efficiency and salt dissolution rate.
• Where possible, use existing dilute supernate to dissolve salt, such as the reuse of DWPF Recycle in Tank 41. Benefit: Minimize the amount of additional volume added to overall Liquid Waste System.
• If existing supernate cannot be used, the SCD should be considered. Benefit: The SCD process allows for very efficient salt dissolution, while avoiding the need to GRM controls. In addition, SCD has the potential to reduce project delays due to the continuous nature of the process.
REFERENCES


7. D. V. Hicks, Tank 10 Salt Dissolution Completion in Support of Salt Batch 7, SRR-LWE-2013-00099, Rev. 0, Savannah River Site, June 2013.


10. SRS Liquid Waste NTank Sample Database.


