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Comparisons of Crystalline Silicotitanate and Resorcinol Formaldehyde Media for Cesium Removal by In-tank Column Processing

Frank G. Smith, Si Young Lee, William D. King*, and Daniel J. McCabe
Savannah River National Laboratory, Westinghouse Savannah River Company, Aiken,
SC 29808

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ABSTRACT

Chemical and thermal performance of crystalline silicotitanate (CST) and resorcinol-formaldehyde (RF) ion exchange media were predicted for column configurations designed for installation in high level waste tanks and intended for cesium removal from radioactive waste supernates. Modeling predictions for the processing of a known Savannah River Site tank waste composition were generated. In a two column configuration under presumed nominal operating conditions (432 gallon packed bed, 10 gpm liquid flow, 25 °C, 45 nCi/g average breakthrough limit) with lead/lag column rotation between processing cycles, approximately two cycles were predicted to treat 1,000,000 gallons of radioactive waste with CST as compared to three cycles predicted for RF. However, this processing mode was shown to be highly unfavorable for RF due to the fact that the lead column is unnecessarily exposed to large radiation doses during movement of the cesium mass transfer zone into the lag column. Thermal modeling calculations indicated that maximum temperatures within stagnant, packed CST and RF columns containing the highest anticipated cesium loading and no active cooling will reach 128 and 78 °C, respectively, within 6 days. Active cooling maintains the cesium-saturated CST and RF columns below 88 and 41 °C, respectively, under stagnant flow conditions.

* author to whom all correspondence should be addressed
email: william02.king@srnl.doe.gov

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INTRODUCTION AND BACKGROUND

In-tank ion exchange columns are being considered for the removal of cesium from radioactive waste solutions at the Savannah River Site (SRS) in Aiken, SC. The waste was generated as a legacy by-product of decades of nuclear material processing operations and is currently stored in large, underground carbon steel tanks (750,000-1,000,000 gallons). The waste is caustic with high concentrations of various sodium salts and lower concentrations of radionuclides which result in a significant radiation field. Removal of specific radionuclides, including cesium, is required prior to disposal of the bulk waste in an immobilized solid form. In-tank column processing of the waste is being considered as a method to accelerate closure of selected waste tanks.

Two cation exchange media are being considered for cesium removal by in-tank column processing: a granular form of crystalline silicotitanate and a spherical form of resorcinol-formaldehyde resin. Crystalline silicotitanate (CST) is an inorganic material composed of sodium, silicon, titanium, and niobium oxides [1] which is highly selective for cesium ions and is not considered to be elutable or reusable. CST is available in an engineered, granular form known as IONSIV IE-911 [2]. The active ion exchange sites contain exchangeable sodium ions located in 8Å tunnels within the solid-state structure [3]. It has been shown in laboratory and performance demonstration tests to be highly effective for cesium removal from waste supernates isolated from storage tanks at Hanford [4],

SRS [5], and Melton Valley [6]. Cesium loaded CST (IE-911) has also been shown to be suitable for vitrification into a final waste form after grinding of the media [6]. The granular form of CST was the baseline material considered during the initial design and development of in-tank column configurations for SRS applications [7].

Spherical resorcinol-formaldehyde (RF) was developed and selected as the baseline material for cesium removal in the Hanford River Protection Project Waste Treatment Plant [8]. This is an organic material composed of resorcinol (1,3-dihydroxy-benzene) units linked by methylene ($-\text{CH}_2-$) bridges [9]. Though granular forms of RF have been studied for cesium removal applications for many years [10], a highly spherical form with excellent hydraulic and handling characteristics was only recently developed for this application [11]. RF is an elutable ion exchange material, and cesium elution can be accomplished by conversion of the sodium resin form that exists under waste loading conditions to the acid resin form by contact with 0.5 M nitric acid. As a result, each waste processing cycle with RF does not involve replacement of loaded resin with fresh material. Instead, the cesium is eluted from the resin and the column is regenerated by conversion back to the sodium form. The concentrated cesium product is an acidic liquid phase rather than a solid phase, which is the case with CST. Spherical RF has been utilized for cesium removal with two actual Hanford tank waste supernate samples [12, 13]. RF resin is susceptible to radiolytic [14, 15] and chemical [16] degradation which impacts performance in each successive processing cycle. These properties limit the effective lifetime of RF columns in waste processing operations.

All evaluations and comparisons of the two ion exchange media were conducted for a column design developed during in-tank column evaluations conducted several years ago

which assumed that the ion exchange media used for cesium removal was IE-911 [7]. The column diameter and height are limited by the size of available or allowable SRS tank top risers and the tank depth. The baseline column design includes a 28 inch ID cylinder with a 6 inch centerline inner cooling tube and four external cooling jackets prepared from 3.5 inch half-pipe tubes symmetrically distributed around the periphery of the column and oriented along the vertical column height.

MODELING

Chemical and thermal modeling efforts were conducted for the baseline in-tank column design and the engineered forms of CST and RF media. Granular CST is produced by UOP, LLC (Des Plaines, IL) as a material known as IONSIV IE-911. Spherical RF is available through Microbeads AS (Smedsmokorset, Norway). The assumed average particle diameter and bed density for IE-911 were 344 μm and 1.0 g CST media/mL bed volume, respectively. The assumed average particle diameter and bed density for sodium-form spherical RF were 457 μm and 0.2563 g RF media/mL bed volume, respectively. The baseline column design used for the calculations involved a 28-in outside diameter stainless steel column with a 6-in schedule 40 stainless steel pipe imbedded at its center to give an annular packed bed with a 27.375 inch outside diameter and a 6.625 inch inside diameter. The packed bed height was assumed to be 15 feet tall, giving a total bed volume of 432 gallons. The external column wall is composed of 5/16 inch stainless steel. Four 3.5 inch schedule 40 half-pipe stainless steel cooling tubes are equally distributed around the perimeter of the outer column wall extending up the vertical column height. Coolant water at 25 °C is supplied at 12.5 gpm to the centerline

cooling tube through an internal 1.5 inch coolant supply pipe located at its center. Coolant water flow is supplied to the outer jackets at 6.25 gpm.

Equilibrium cesium loading for each media were calculated for the waste composition of interest (Table 1) using isotherm models developed for each material and column performance was predicted using a liquid chromatography code. CST isotherms were generated using the ZAM code developed at Texas A&M. RF isotherms were generated using the TMIXP code recently developed at the Savannah River National Laboratory. Instantaneous cesium breakthrough profiles were generated using the VERSE LC code developed at Purdue University. Bucket average lag column cesium breakthrough profiles were calculated by spreadsheet based on the cumulative cesium breakthrough and the solution volume processed. Detailed descriptions of the methodology used were reported by Aleman and Hamm [17, 18].

Thermal analysis was conducted for a two-dimensional, horizontal slice of the column using the commercial Computational Fluid Dynamics code known as FluentTM. Heat transfer effects in the vertical direction associated with end effects were ignored (i.e. the columns were modeled as infinitely long). The column parameters used for thermal analysis of the CST and RF columns are provided in Table 2. In each case the maximum cesium loading (calculated with the isotherm models for a loading temperature of 25 °C) was assumed with initial internal column and external air temperatures of 35 °C. Heat transfer within the column was assumed to occur by conduction heat transfer with no natural convection associated with fluid movement within the bed. Heat transfer from the column wall boundary to the external air (which in this case would be the tank headspace) was assumed to occur by natural convection. For cases with active cooling,

heat transfer at the interfaces between the column walls and the cooling system were assumed to involve forced convection with 25 °C flowing water in the cooling system.

RESULTS AND DISCUSSION

Granular CST (IE-911) and spherical RF resin are both highly selective for cesium removal under caustic conditions in the presence of high concentrations of sodium competitor ions. The SRS tank waste composition utilized for CST and RF performance comparisons is provided in Table 1. This waste composition results in the highest Cs-137 loading levels expected for both ion exchange media of all waste samples planned for processing by in-tank column processing. Predicted cesium equilibrium loading levels for CST and RF with this waste type at 25 °C were 224 and 133 Ci Cs-137/L packed bed [19]. Differences in cesium loading levels are due to differences in selectivity between the various competing ions (primarily Group 1 cations, though strontium can impact cesium loading for CST in some cases) and density differences (1.0 g/mL for CST; 0.3 g/mL for sodium-form RF), since the total cesium capacities of the media are similar on a mass basis (0.6 mmol/g CST and 0.7 mmol/g RF).

Lag column effluent bucket average cesium breakthrough profiles were calculated for both ion exchange media assuming that waste processing was conducted through two columns in series with lead/lag rotation between processing cycles [19]. Nominal operating conditions of 25 °C, 432 gallon packed bed (15 foot tall column), and 10 gpm flow rate were assumed for column loading operations using the waste feed composition provided in Table 1. Cesium breakthrough profiles for CST and RF columns are provided in Figure 1 for the first 100,000 gallons of waste processed (maximum waste

tank volume). For each ion exchange media, the first processing cycle involved greater volume processed than in subsequent cycles since fresh ion exchange media was in the lead column for this cycle. Nearly steady-state performance was observed in subsequent cycles for each media. Steady-state volumes processed for CST and RF columns after the completion of the first cycle were 390,000 and 257,000 gallons, respectively, which corresponds to 903 and 595 packed bed volumes ($1 \text{ BV} = 432 \text{ gallons}$) of supernate processed (based on Na-form volume for RF resin). Clearly both media have high capacity and selectivity for cesium under these conditions.

Liquid phase cesium concentrations down the length of the lead/lag column train at cycle completion (45 nCi/g bucket average breakthrough) are provided for each media in Figure 2. This plot indicates the efficiency of cesium loading and the sharpness of the cesium mass transfer zone for each material under these processing conditions. The cesium mass transfer zone extends across a distance slightly greater than one column length (15 feet) for CST and is largely contained in the lag column. The lead column is nearly saturated with cesium ions. In contrast, the cesium mass transfer zone length for RF is significantly shorter and is contained within approximately one quarter of one column length. This is a consequence of the larger effective porosity and loading kinetics of packed RF beds. Loading kinetics differences for the two media are further exemplified by separate modeling efforts at 20 gpm (not shown) which show negligible impacts of doubling the flow rate for RF on the supernate volume processed while a 14% decrease in volume processed was observed for CST.

RF resin is known to undergo chemical degradation resulting from exposure to dissolved oxygen in waste feed solutions and radiolytic degradation resulting from the radiation

field associated with cesium loaded ion exchange columns. Chemical cycle testing using air-saturated simulants indicates that the maximum expected chemical degradation during cesium loading operations is near 0.6% cesium capacity reduction per cycle [16]. Therefore, chemical degradation has a minor, though not trivial, impact upon cesium removal performance in the first 10-20 chemical cycles. Radiolytic degradation is a more significant concern with regard to RF performance impacts, particular with the processing mode assumed for the cesium performance modeling (Figures 1 and 2). Radiation dose studies indicate that a 100 Mrad dose results in a 14% decrease in RF cesium loading capacity [15]. As shown in Figure 2, processing waste supernate through the lead/lag column train to a 45 nCi/g lag column breakthrough limit results in significant radiation dose to the lead column during the passage of the cesium mass transfer zone through the lag column. Given the sharpness of the RF mass transfer zone (Figure 2), processing waste in this manner is not necessary to maximize utilization of the resin capacity (as is the case for CST).

Simple and conservative estimates of RF radiation exposure for the lead and lag columns under the assumed processing mode (45 nCi/g bucket average lag column breakthrough limit per cycle, lead column elution, lead/lag rotation) have been calculated [20]. Based on the anticipated equilibrium cesium loadings for this feed, the RF radiation dose rate is 4.4 Mrad/day. It was assumed that all of the radiation dose was absorbed, although a fraction (perhaps 25%) will likely escape from the column. RF exposure times were estimated for 10 and 20 gpm flow rates based on the total predicted waste volume processed per cycle. Full resin capacity was assumed until the 100 Mrad dose limit was reached for either column, at which point the resin was considered spent. Fractional

cesium loadings in each cycle and the effects of gradual and (assumed) linear loading were accounted for by adjustments to the total dose rates. A correction of 0.75 was applied for the lead column since column processing was continued approximately twice as long as was needed for saturation of the lead column. This corresponds to a dose correction of 0.5 for the first half of the processing time during lead column loading and 1.0 during the second half of processing when the lead column is saturated. A correction of 0.25 was applied for the lag column maximum dose since this column received no dose during the first half of processing and a time-averaged dose of 0.5 during the remainder of processing in a given cycle. Fractional cesium loading corrections of 90 and 80% were also applied to the lag columns for 10 and 20 gpm flows, respectively, based on the calculated loading profiles (such as the one shown in Figure 2).

Not surprisingly, this processing mode results in significant RF radiation dose per cycle and resin lifetimes of only 2 and 5 cycles were estimated for flow rates of 10 and 20 gpm, respectively [20]. Processing at a higher flow rate has minimal impact upon the cesium breakthrough profile but decreases the RF radiation exposure time by 50%. Obviously, higher flow rates are preferred for RF resin. Based on these results, alternative processing modes were recommended for RF columns. Operating with a 45 nCi/g breakthrough limit results in significant unnecessary radiation dose to the RF resin and is considered the worst operating mode for this material. Alternative recommended processing strategies included two column series operations with a 50% lead column instantaneous cesium breakthrough limit (intermediate dosage) and single column operation with a 45 nCi/g bucket average processing limit (lowest dosage). Single

column operation is expected to at least double resin process lifetime relative to the baseline case.

Thermal analysis calculations for cesium-saturated CST and RF columns were conducted for various processing cases with emphasis on accident scenarios expected to lead to significant temperature increases within the beds [21]. Packed columns loaded with radioactive cesium generate significant heat associated with radiolytic decay. As long as liquid is continuously processed through the columns, temperature rises within the bed are minimal. Even at a waste feed flow rate of 5 gpm the solution temperature increases expected during processing through 15 foot tall cesium-saturated CST and RF packed beds (baseline in-tank column design without active cooling) are only 1.7 and 0.8 °C, respectively. Two accident scenarios were evaluated by thermal analysis involving no fluid flow through the columns to determine the maximum expected bed temperatures and temperature distributions versus time for each media. The modeling parameters and assumptions are provided in Table 2. The modeling parameters are conservative for various reasons including:

- maximum heat loads were based on 25 °C cesium isotherms while the thermal analysis assumes initial column temperatures of 35 °C (lower cesium loadings are expected at 35 °C for each media)
- cesium-saturation is assumed although this is only the case at the end of a loading cycle
- convective heat transfer mechanisms in the packed beds are ignored which would assist heat transfer out of the columns

- air is the assumed fluid external to the columns although the columns will be at least partially immersed in salt solution most of the time
- only natural convection is considered for heat transfer from the columns to the air, although some forced air flow in the tank headspace is expected.

The first thermal analysis case was considered bounding with regard to the maximum achievable temperatures within the columns since it involved simultaneous occurrence of the following conditions: maximum column heat load, no column liquid immersion, loss of process fluid flow, and loss of the active cooling system. The maximum and minimum column temperatures versus time for CST and RF columns under these conditions are provided in Figure 3. The maximum steady-state temperatures of CST and RF-packed beds under these conditions were calculated to be 156 and 88 °C, respectively. However, the CST/salt solution mixture is expected to boil near 130 °C. As expected, based on the heat loads associated with the cesium-saturated columns and the relative porosities, thermal conductivities, and heat capacities of the packed beds (Table 2), RF columns heat up more slowly than CST columns and have much lower steady-state temperatures. RF columns are not expected to reach boiling even under these bounding conditions. However, material temperature limits for CST and RF media are believed to be near 65 °C, and both media are at risk for damage leading to decreased cesium loading if this processing scenario were to occur unmitigated. Media damage may occur within 48 hours for CST and 96 hours for RF. Given the significantly higher temperatures predicted for CST columns, subsequent thermal modeling efforts focused on this media.

Temperature distribution profiles within packed CST columns with and without active cooling are provided in Figure 4. Without active cooling the maximum temperature is located at the innermost portion of the packed bed. With active coolant flow through the inner centerline pipe and the outer cooling jackets significantly lower temperatures are observed and the location of the temperature maximum shifts to the near the column center (lowest distribution in Figure 4). The maximum steady-state temperature for the CST column with active coolant flow is 63 °C, which is just below the aforementioned media temperature limits. The maximum steady state temperature for RF with active coolant flow (not shown) is 41 °C. Temperature distributions were also calculated for other column design options with only centerline cooling and only jacket cooling. Clearly, the most effective portion of the coolant system is the centerline pipe, as would be expected given that it is the center of the columns that tend to heat up (as shown for the “no active cooling” case). The maximum steady-state temperatures predicted for centerline only and jacket only cooling options are 80 and 114 °C, respectively. If it is determined that the project temperature limit for CST media is greater than 65 °C or that the timescale for column heat-up is acceptable a design simplification may be possible involving the removal of the outer jacket portion of the coolant system. This design simplification is also possible should RF be the preferred media.

CONCLUSIONS

Good performance with regard to cesium removal from SRS waste supernates was predicted for both CST and RF engineered media. More waste can be processed per treatment cycle under the baseline conditions with CST than RF, but CST is non-reusable and requires lead column replacement after each cycle. RF resin is elutable and reusable,

though it is subject to chemical and radiolytic damage leading to reduced capacity with each processing cycle. Rapid cesium loading kinetics with RF resin allows for considerable processing flexibility and the ability to minimize chemical and radiolytic exposure per cycle. Risks from thermal excursions within the columns under accident scenarios are greater with CST primarily due to the higher cesium loading. In the worst case bounding situation without active cooling, cesium-saturated CST columns can reach the solution boiling point. The baseline cooling system for the in-tank column design provides adequate cooling capacity to maintain the CST columns within process limits and modeling efforts indicate that a design simplification may be possible involving removal of the outer cooling jackets. The physical, chemical, and performance properties of both media are quite good. Technology selection for in-tank column processing will likely be weighted toward downstream impacts and dealing with secondary waste streams generated.

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Table 1. Chemical Composition of the Savannah River Site Tank Waste Supernate Sample.

Species	Molarity
Na^{+1}	6.0
NO_3^{-1}	2.26
NO_2^{-1}	0.74
OH^{-1}	1.97
AlO_2^{-1}	0.35
CO_3^{-2}	0.11
SO_4^{-2}	0.15
PO_4^{-3}	0.01
Cl^{-1}	0.03
F^{-1}	0.03
K^{+1}	0.01
Cs^{+1}	1.08×10^{-4}
Cs-137 (pCi/mL)	2.22 E8
Sr^{+1}	3.02×10^{-6}
Rb^{+1}	1.18×10^{-5}

Table 2. Thermal Modeling Parameters for Cesium-Saturated CST and RF Packed Columns Immersed in Waste Supernate.

Parameter	CST	RF
heat load (W/m^3)	1273	661
total bed porosity (%)	57	80
IX media-salt solution thermal conductivity (W/mK)	0.413	0.599
IX media-salt solution specific heat ($\text{J/kg}\cdot\text{K}$)	2517	3140
initial column temperature ($^{\circ}\text{C}$)	35	
coolant water temperature ($^{\circ}\text{C}$)	25 for active cooling cases	
coolant water flow rate (gpm)	6.25 for side jackets 12.5 for centerline pipe 0.0 for cases with no active cooling	
ambient temperature ($^{\circ}\text{C}$)	35	
heat transfer coefficient ($\text{W/m}^2\cdot\text{sec}$)	238 for centerline pipe wall 620 for outer column wall at jacket 1.5 for outer column wall	
heat transfer mechanism within bed	conduction	
heat transfer mechanism at column wall boundary	natural convection	
heat transfer mechanism at bed cooling system interfaces	forced convection for cases with active cooling	

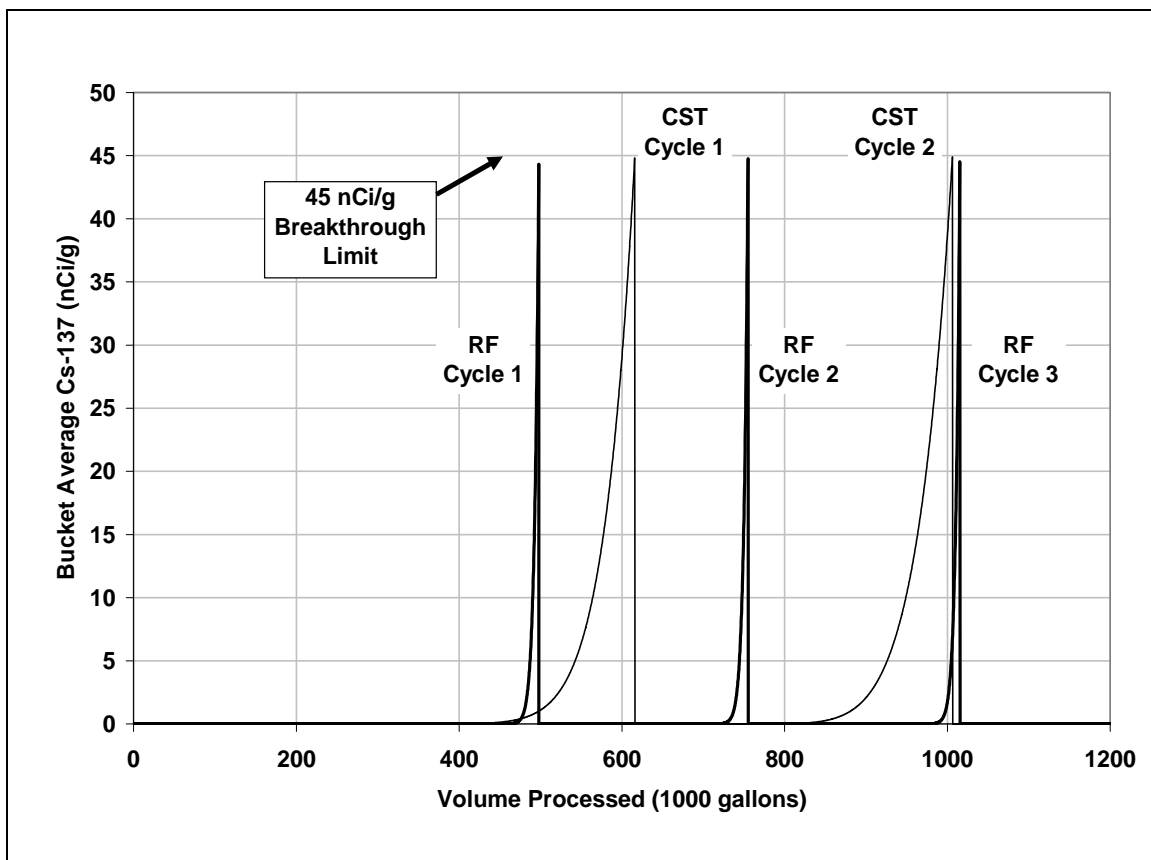


Figure 1. Cs-137 lag column effluent bucket average breakthrough profiles for crystalline silicotitanate and resorcinol formaldehyde columns at 25 °C and 15 gpm flow.

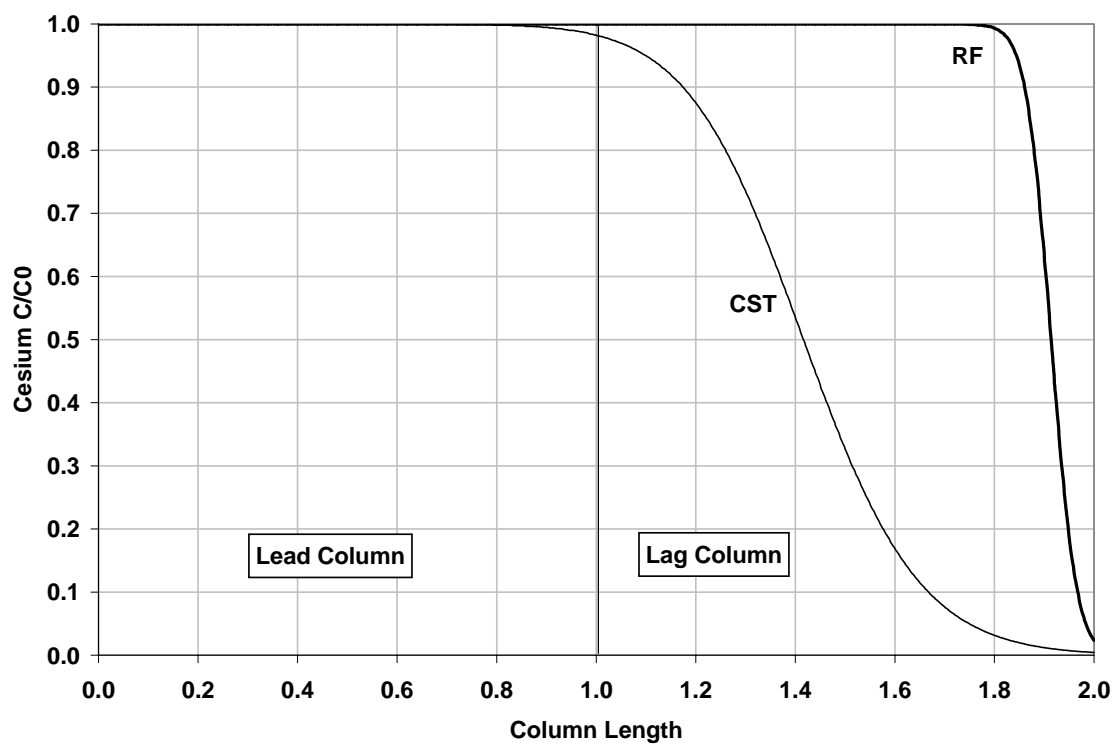


Figure 2. Liquid phase cesium concentration profiles down the length of the column train at the conclusion of the cesium loading cycle for CST and RF columns.

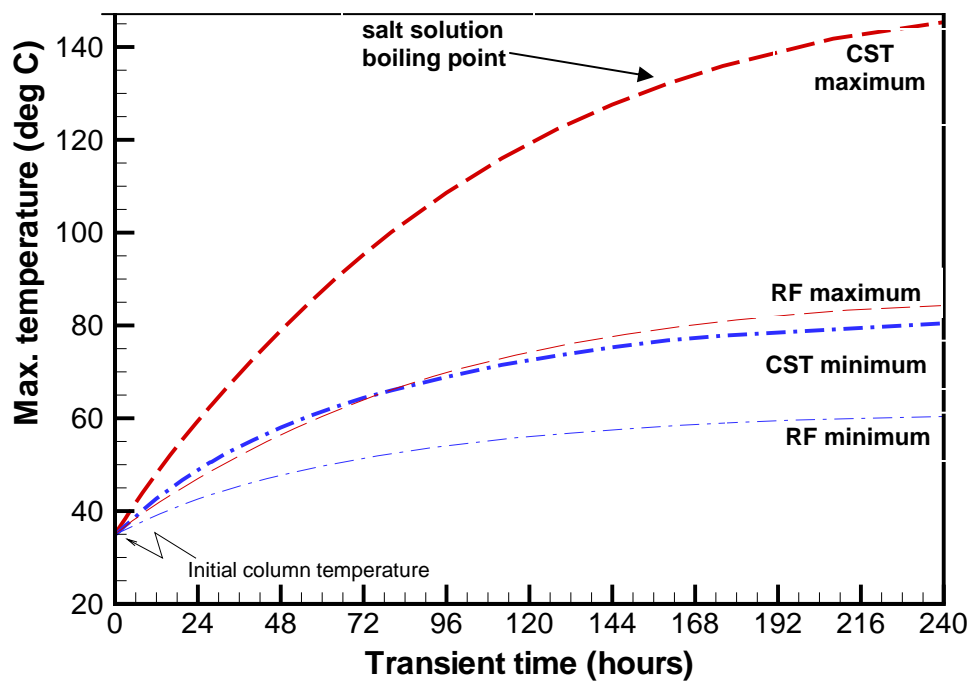


Figure 3. Maximum and minimum temperatures versus time for CST and RF columns under bounding conditions (highest Cs-137 loading, stagnant liquid flow, no active cooling).

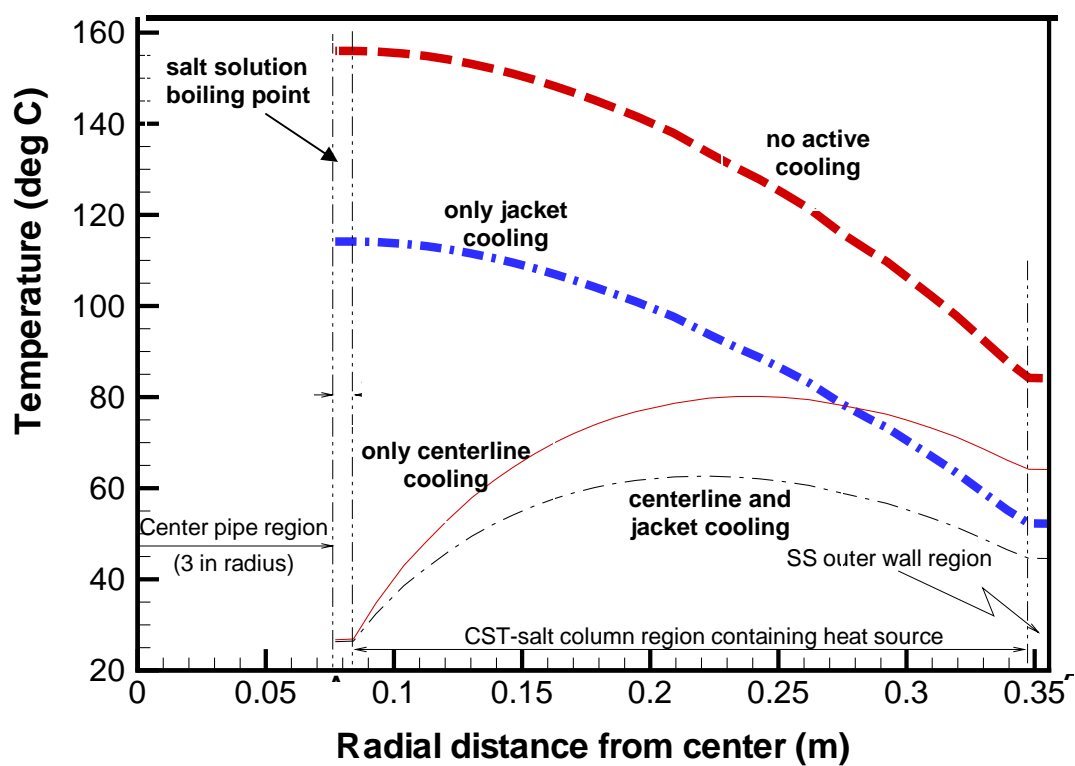


Figure 4. Steady-state temperature profiles for various accident and operating scenarios with the CST column at the maximum Cs-137 loading.