



UNITED STATES  
NUCLEAR REGULATORY COMMISSION  
WASHINGTON, D.C. 20555-0001

September 6, 2016

MEMORANDUM TO: Richard Chang, Acting Chief  
Low-Level Waste Branch  
Division of Decommissioning, Uranium Recovery,  
and Waste Programs  
Office of Nuclear Material Safety  
and Safeguards

THROUGH: Christopher McKenney, Chief */RA/*  
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FROM: Cynthia Barr, Senior Systems Performance Analyst */RA/*  
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Office of Nuclear Material Safety  
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SUBJECT: TECHNICAL REVIEW: U.S. DEPARTMENT OF ENERGY  
DOCUMENTATION RELATED TO TANKS 16H AND 12H  
GROUTING OPERATIONS WITH EMPHASES ON  
SPECIFICATIONS, TESTING, RECOMMENDATIONS AND  
PLACEMENT PROCEDURES (PROJECT NO. PRO0734)

The U.S. Nuclear Regulatory Commission (NRC) has performed a technical review of several documents prepared by the U.S. Department of Energy (DOE) that provide information on grouting and closure of Tanks 16H in 2015 and 12H in early 2016. The focus of NRC's technical review is grout formulations and specifications, testing, recommendations and placement procedures. NRC also revisits findings from previous technical review reports related to Tanks 18F and 19F grouted in 2012; as well as Tanks 5F and 6F grouted in 2013 (Agencywide Documents Access and Management System (ADAMS) Accession No. ML13269A365 and ADAMS Accession No. ML14342A784). This technical review can be tied to several monitoring factors listed in NRC's combined F-Area and H-Area Tank Farm monitoring plan entitled "U.S. Nuclear Regulatory Commission Plan for Monitoring Disposal Actions Taken by the U.S. Department of Energy at the Savannah River Site F-Area and H-Area Tank Farm Facilities in Accordance with the National Defense Authorization Act for Fiscal Year 2005" [available using ADAMS Accession No. ML15238A761] issued in October 2015. The Monitoring Plan discusses NRC's approach to fulfilling its responsibilities under the National Defense Authorization Act for Fiscal Year 2005 to monitor DOE disposal actions to assess compliance with the Performance Objectives in 10 CFR Part 61, Subpart C, for DOE wastes (and

associated disposal facilities) found to be incidental to reprocessing. NRC's Monitoring Plan lists the technical areas, which are the focus of NRC's monitoring activities. This technical review supports NRC's Monitoring (of) Factors 3.2 "Groundwater Conditioning via Reducing Grout," 3.3, "Shrinkage and Cracking," and 3.4, "Grout Performance" listed in the NRC's Monitoring Plan, which are important to F-Area and H-Area Tank Farm Facility engineered barrier performance, as discussed in more detail in the evaluation section below.

The NRC staff concludes that performance requirements for the tank grout formulation recommended and tested for Tank 16H and 12H closure are generally consistent with initial bulk chemical and hydraulic properties assumed in DOE's H-Area Tank Farm Facility Performance Assessment (PA) (SRR-CWDA-2010-00128). However, DOE assumes but has not provided sufficient information and testing to support its exclusion of shrinkage gaps, cracks, and other preferential flow pathways through the grout monolith from the reference case in DOE's PA. These conclusions were also true for Tank 18F, 19F, 5F and 6F and DOE's F-Area Tank Farm Facility PA.

The NRC staff expects DOE to provide additional information related to the extent and performance impact of tank grout shrinkage to have reasonable assurance that the performance objectives specified in Subpart C of Part 61 of Title 10 of the Code of Federal Regulations (10 CFR Part 61, Subpart C) are met. As stated above, DOE assumes in the PAs for F- and H-Area that the grout does not shrink, crack or fracture in the base or reference case. Rather, the grout is assumed to degrade slowly with a subsequent increase in hydraulic conductivity of the grout matrix over time. This assumption is risk-significant because conceptually DOE assumes that the entire grout matrix is available to condition infiltrating groundwater to relatively low Eh (e.g., initially  $-470$  mV) and high pH, which is necessary to maintain the low solubility of key radionuclides. For the tank grout to condition infiltrating water to relatively low Eh and high pH, water must flow through and interact with the grout. In contrast, if flow is concentrated along fast pathways through the tank grout (e.g., gaps between the tank wall/internal tank components and tank grout, or shrinkage gaps, cracks and fractures in the grout), flow rates through the grout may be significantly faster and the extent of interaction between infiltrating groundwater and tank grout may be significantly less than assumed in DOE's PAs, thereby hastening the time to transition to risk-significant solubility for certain key radionuclides. During its review of Tank 16H grouting video (SRR-CWDA-2015-00170), NRC staff also observed large-aperture cracks in grout that developed shortly after the grout had been placed in the Tank 16H annulus. NRC staff expects DOE to provide additional information on the mechanisms for crack formation, including thermal cracking, for all grout monoliths, including those in Tanks 16H and 12H. NRC staff will continue to evaluate the potential for shrinkage- and cracking-induced preferential flow through the tank grout under MF 3.3, "Shrinkage and Cracking" (ADAMS Accession No. ML15238A761), as well as DOE's assumptions regarding flow through the tank grout that influences the extent of groundwater conditioning in MF 3.2 "Groundwater Conditioning via Reducing Grout".

During its review of Tank 16H grouting video (SRR-CWDA-2015-00170), NRC staff observed potential bleed water segregation of tank grout during placement that could enhance shrinkage along the periphery (i.e., at the wall) of the tank and result in inhomogeneous material properties affecting water percolation patterns through the monolith. The NRC staff continues to monitor the potential for segregation of grout bleed water and consequent impacts on future water flow through the grout monolith and waste release under Monitoring Factor 3.4, "Grout Performance" because of its importance to the demonstration that the long-term closure of both F-Area Tank

Farm and H-Area Tank Farm tanks will meet 10 CFR Part 61, Subpart C, performance objectives for protection of the general population from releases of radioactivity, and protection of individuals from inadvertent intrusion. Tank 12H grouting video has not yet been requested by NRC, because DOE has only recently completed grouting of this tank. Therefore, NRC staff reaches only preliminary conclusions on Tank 12H grouting in this technical review report.

The NRC staff will also continue to monitor void volumes in the waste tanks to the extent that information is available (Monitoring Factor 3.4, "Grout Performance"); the importance of alkali-silica reactivity on cementitious material degradation (Monitoring Factor 3.3, "Shrinkage and Cracking"); and the impacts on the (i) pH buffering capacity of tank grout and (ii) timing of release of key radionuclides that will derive from its Portland cement containing up to 5 wt percent limestone (Monitoring Factor 3.4, "Grout Performance"). It is NRC staff's position that this information would enhance DOE's demonstration that the performance objectives listed in 10 CFR Part 61, Subpart C are met.

Other conclusions unique to Tanks 16H and 12H grouting include the following:

- DOE should take reasonable measures to ensure a sufficient number of cement trucks are in rotation to optimize grout distribution throughout the tank and minimize mounding.
- More flowable clean cap grout used to fill remaining void space at the top of the Tank 16H primary and annulus may have significantly different hydraulic properties compared to the rest of the bulk fill grout placed in the primary and annulus of Tank 16H. DOE should address the potential for either a capillary or permeability barrier to form due to the varying hydraulic conductivity of the clean cap and bulk fill grout used in Tank 16H.
- The results of the grout drop test report (RPT-5539-EG-0016) suggest the potential for segregation and bleed water production in the annulus of Tank 12H during initial grouting operations if grout was dropped into standing water. DOE should provide additional information regarding the quantity and performance impact of the presence standing water in Tank 12H during grouting.
- Lehigh Grade 120 slag used in the Tank 12H grout mix starting on the second day of grouting is expected to provide superior chemical performance compared to Holcim Grade 100 slag due to the higher activity index and increased reduction capacity. The compressive strength of the Grade 120 slag is also expected to increase due to a combination of small particle size and increased reactivity. DOE should evaluate differences in hydraulic conductivity between the Grade 100 and Grade 120 slag used to fill Tank 12H and any resulting performance impact.

R. Chang

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In this report, there is no significant change to the NRC staff overall conclusions from the F- and H-Tank Farm TERs regarding compliance of DOE disposal actions with the 10 CFR Part 61 performance objectives.

Enclosure:

Technical Review of Documents Related  
to Tanks 16H and 12H Grout Formulations,  
Testing, Procedures, and Operations at the  
H-Area Tank Farm at the Savannah River Site

In this report, there is no change to the NRC staff overall conclusions from the F- and H-Tank Farm TERs regarding compliance of DOE disposal actions with the 10 CFR Part 61 performance objectives.

Enclosure:

Technical Review of Documents Related to Tanks 16H and 12H Grout Formulations, Testing, Procedures, and Operations at the H-Area Tank Farm at the Savannah River Site

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**ADAMS Accession No. ML16231A444**

<b>OFFICE</b>	NMSS	NMSS	NMSS	NMSS	NMSS
<b>NAME</b>	CBarr	TMoon	JShaffner	CMcKenney	CBarr
<b>DATE</b>	8/19/2016	8/22/2016	8/24/2016	9/2/2016	9/7/2016

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## **Technical Review of Documents Related to Tanks 16H and 12H Grout Formulations, Testing, Procedures, and Operations at the H-Area Tank Farm at Savannah River Site**

Date: August 19, 2016

Reviewers:

Cynthia Barr, U.S. Nuclear Regulatory Commission (NRC)  
Cynthia Dinwiddie, Southwest Research Institute®

General Grout Documents:

1. C-SPP-F-00055. Ganguly, A. "Furnishing and Delivery of Tank Closure Grout." Revision 4. Aiken, South Carolina: Savannah River Remediation, LLC. December 20, 2012.
2. C-SPP-F-00057. McCord, J.B. "Furnishing and Delivery of Cooling Coil Grout Dry Feeds." Revision 2. Aiken, South Carolina: Savannah River Remediation, LLC. July 2014.
3. RPT-5539-EG-0016 (SRRA051386-2-A). Diener, G. "Savannah River Remediation Tank Closure Grout Assessment Final Report" (Grout Drop Test Report). Revision 0. Barnwell, South Carolina: EnergySolutions. December 16, 2014.
4. SDDR No. 13182. "Section 3.2.3.3 States the Slag Cement Must Meet ASTM C989, Grade 100. This Slag is No Longer Available in the Southeast, U.S.A. Change Specification to Allow for the Use of ASTM 989, Grade 120 Slag. (Supplier Deviation Disposition Request)." Augusta, Georgia: Argos Ready Mix, LLC. March 30, 2015.
5. SRNL-STI-2012-00546. Cozzi, A.D. and B.R. Pickenheim. "Impact of Standing Bleed Water on Saltstone Placement." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. September 2012a.
6. SRNL-STI-2012-00576. Cozzi, A.D. and B.R. Pickenheim. "Impact of Standing Water on Saltstone Placement II - Hydraulic Conductivity Data." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. October 2012b.
7. SRNL-STI-2012-00578. Langton, C.A., et al. "Relationship Between Flowability and Tank Closure Grout Quality." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. October 2012.
8. USQ-HTF-2015-00300. Voegtlen, R.O. "Supplier Deviation Disposition Request (SDDR) Number 13182–Deviation from Specification C-SPP-F-00055, Revision 4 (Technical Review Package)." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. September 10, 2015.

9. VSL-15R3740-1. Gong, W. et al. "Investigation of Alternate Ground Granulated Blast Furnace Slag for the Saltstone Facility (Final Report)." Revision 0. Washington, DC: Vitreous State Laboratory, The Catholic University of America. August 26, 2015.
10. WSRC-STI-2008-00172. Harbour, J.R., et al. "Closure of HLW Tanks—Formulation for a Cooling Coil Grout." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. April 2008.
11. WSRC-STI-2008-00298. Hansen, E.K. et al. "Closure of HLW Tanks—Phase 2, Full Scale Cooling Coils Grout Fill Demonstrations." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. June 2008.

Tank 18F and 19F Documents:

12. SRNL-STI-2011-00551. Stefanko, D.B. and C.A. Langton. "Tanks 18 and 19-F Structural Flowable Grout Fill Material Evaluation and Recommendations." Revision 1. Aiken, South Carolina: Savannah River National Laboratory. April 2013.
13. SRNL-STI-2011-00564. Stefanko, D.B. and C.A. Langton. "Tank 18 and 19-F Tier 1A Equipment Fill Mock Up Test Summary." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. September 2011.
14. SRNL-STI-2011-00592. Stefanko, D.B. and C.A. Langton. "Tanks 18 and 19-F Equipment Grout Fill Material Evaluation and Recommendations." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. November 2011.
15. SRNL-STI-2011-00749. "Tank 18F and 19F Tank Fill Grout Scale Up Test Summary." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. December 2011.
16. SRR-CES-2012-00031. "Summary Report of the Equipment Grout Mock-up Test." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. April 13, 2012.

Tank 5F and 6F Documents:

17. 2013-NCR-15-WFC-0006. "F Tank Farm Grout - Tank 6 (Non-Conformance Report)." Aiken, South Carolina: Savannah River Remediation. October 2, 2014; USQ-FTF-2013-00317. "New Data – Use-As-Is Disposition of the Non-Conformance Report (NCR) 2013-NCR-15-WFC-0006 'F Tank Farm Grout – Tank 6.' Non-Conformance Tank 6 Grout Water Content Higher than Allowed per C-SPP-F-00055, Rev. 4 'Furnishing and Delivery of Tank Closure Grout.'" Revision 0. Aiken, South Carolina: Savannah River Remediation. October 3, 2014; and tank grout batch tickets for September 19, 2013.
18. SRR-CWDA-2014-00015. Cantrell, J.R. "Tank 5 and 6 Grouting Project Lessons Learned." Revision 0. Aiken, South Carolina: Savannah River Remediation. February 6, 2014.

19. SRR-LWE-2013-00214. Chandler, T.L. "Engineering Path Forward – Tanks 5 & 6: Record of Additional Grouting Actions. Revision 0. Aiken, South Carolina: Savannah River Remediation. February 10, 2014.
20. Work Order No. 01199254-65. "Tanks 5–6 Pump Standing Water in Risers." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. December 10, 2013.

Tank 16H Documents:

21. C-SPP-Z-00012. Patel, R. "Vault 4 Clean Cap Grout (Procurement Specification)." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. March 20, 2014.
22. SRR-CWDA-2013-00091. "Industrial Wastewater Closure Module for Liquid Waste Tank 16H, H-Area Tank Farm, Savannah River Site." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. April 2015.
23. SRR-CWDA-2014-00011. Smith, F.M. "Evaluation of Vendor Supplied Clean Cap Material for Saltstone Disposal Units." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. March 2014.
24. SRR-CWDA-2015-00096. Layton, M. "Unreviewed Waste Management Question Evaluation – Use Alternative Tank 16 Fill Grout (Per Specification C-SPP-Z-00012)." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. September 2015.
25. SRR-CWDA-2015-00100. "Evaluation of the Use of an Alternative Tank 16 Fill Grout (Per Specification C-SPP-Z-00012) (Interoffice Memorandum to G.C. Arthur from M.H. Layton)." Revision 2. Aiken, South Carolina: Savannah River Remediation, LLC. September 2015.
26. SRR-CWDA-2015-00159. "Tank 16 Final Configuration Report for H-Tank Farm at the Savannah River Site." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. May 2016.
27. SRR-CWDA-2015-00160. "Evaluation of the Performance Assessment Impact of using an Alternative Fill Grout in the H-Area Tank Farm (Interoffice Memorandum to G.C. Arthur from M.H. Layton)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. January 4, 2016.
28. SRR-LWE-2014-00013. Walters, C.D. "Tank 16H Grout Strategy." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. January 6, 2015.
29. SRR-LWE-2014-000150. Ostler, M. "Tank 16-H Closure Assurance Plan." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. March 2015.

30. SRR-LWP-2014-00049. Sareen, H. "SRR Subcontractor Surveillance Plan Furnishing and Delivery of Tank Closure Grout (TK 16) (P.O. No. SRRA-064184-1)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. June 1, 2015.
31. SRR-TCR-2015-00024. "Tank 16 Grouting Lessons Learned (Interoffice Memorandum from B. Davis to L. Blackford and J. Williams)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. January 27, 2016.
32. Work Order No. 01324150-64. Fail, J.A. "TK Clos & Reg Cn to Perform Grout Prep/Grout Placement TK 16." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. August 22, 2014.

Tank 12H Documents:

33. LWO-LWQ-2016-00001. Thompson, J.W. "SRR Subcontractor Surveillance Plan Furnishing and Delivery of Tank Closure Grout (TK 12) (P.O. No. SRRA-064184-1)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. January 13, 2016.
34. SRR-CWDA-2014-00086. "Industrial Wastewater Closure Module for Liquid Waste Tank 12H, H-Area Tank Farm, Savannah River Site." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. May 2015.
35. SRR-CWDA-2015-00057. "Evaluation of the Use of Grade 120 Slag Cement in Tank Closure Grout versus Performance Assessment Assumptions (Interoffice Memorandum to G.C. Arthur from M.H. Layton)." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. August 27, 2015.
36. SRR-CWDA-2015-00088. Layton, M.H. "Unreviewed Waste Management Question Evaluation – Use of Grade 120 Slag in Tank Closure Grout." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. September 2015.
37. SRR-LWE-2012-00030. "Tank 12 Cooling Coil Flushing Strategy (Interoffice Memorandum to M.D. Buxton from S.J. Worthy and J.R. Tihey)." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. November 28, 2012.
38. SRR-LWE-2014-00147. Chandler, T. "Tank 12H Grout Strategy." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. August 31, 2015.
39. SRR-LWE-2014-00161. Walters, C.D. "Tank 12 Internal Equipment Evaluation." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. February 12, 2015.
40. SRR-LWE-2015-00032. Ostler, M. "Tank 12-H Closure Assurance Plan." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. April 2015.

41. SRR-LWE-2015-00048. Griffin, A.L. "Path Forward for Tank 12 Annulus Liquid Removal (Interoffice Memorandum to E. Patten et al. from A.L. Griffin and G.C. Arthur)." Aiken, South Carolina: Savannah River Remediation, LLC. Revision 0. July 7, 2015.
42. Work Order No. 01337683-33. Patton, G.W. "Placement of Bulk Fill Grout: Tank 12." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. April 29, 2015.

## **NRC Technical Reviews**

Summaries of the primary documents related to Savannah River Site (SRS) Tank Farm grouting listed above are provided in Appendix A. Technical reviews of the grout-related documents listed above are the basis for NRC's evaluation of SRS Tanks 18F, 19F, 5F, 6F, 16H, and 12H grouting and final configurations discussed below.

## **Evaluation**

### **Tank Grout Formulation, Testing, Placement and Performance**

Many of NRC staff's concerns about the waste tank grout formulation that resulted from the original technical review of Tank 18F and 19F grouting and subsequent review of Tank 5F and 6F grouting operations remain at the time of this writing. This technical review, which is focused on Tank 16H and 12H grouting operations, summarizes remaining NRC staff recommendations from prior technical review reports and accounts for new information or changes to DOE's approaches for Tank 16H and 12H and new non-conformances. To fill the primaries and annuli of Tanks 16H and 12H, DOE selected the same tank grout that had been used previously to fill Tanks 5F, 6F, 18F, and 19F (C-SPP-F-00055, Attachment 5.5). The following discussion addresses grout flowability and mounding, specifications and testing, bleed water segregation, grout drop testing, shrinkage, groundwater in-leakage, non-conformances, alkali-silica reactivity (ASR), and thermal considerations for bulk fill tank and annulus grout.

#### *Grout Flowability and Mounding*

During a June 12, 2012, onsite observation visit, NRC staff inquired about the extent of grout mounding below the center riser, which was the only riser used to place grout into Tanks 18F and 19F, and the ability of DOE to completely fill waste tanks with grout at their periphery as a result of the mounding (ADAMS Accession No. ML12191A210). Although significant mounding was observed by NRC early during grouting of Tanks 18F and 19F, DOE did not indicate nor document any significant issues with filling the tanks. Void volume estimates were provided to NRC in the Final Configuration Report inputs (SRR-LWE-2012-00217), and that indicated approximately 3 percent less volume of grout was used compared to what was estimated to fill the tanks. For Type IV tanks, DOE places grout through a center riser, although when questioned by NRC, DOE indicated that it could create additional entry points into the tank, if access to the interior of the tanks for grouting was needed due to mounding (ADAMS Accession No. ML13269A365). Other important attributes of Type IV tanks are (1) the absence of cooling coils used in other tanks to cool the waste, and (2) the domed roofs of the tank vaults. The domed roofs of the tank vaults make it easier to fill the tank from the center riser. Cooling coils act as obstructions and thereby make it more difficult to clean waste from the bottom of the tanks as well as grout the tanks. Over four miles of cooling coils are present in SRS Tank Farm tanks with cooling coils (Type I, II, III, and IIIA tanks contain cooling coils). To completely fill tanks containing cooling coils with tank grout, DOE enhanced tank grout flowability by specifying a higher range of desirable slump flow values (C-SPP-F-00055, Revision 4, Attachment 5.5), which were achieved solely through the use of admixtures (high-range water reducer). Acceptable slump flow is obtained at the batch plant and 8.0 gal (30.3 L) of water is withheld to allow further slump adjustments through potential water additions after grout is delivered to the site (per ASTM C94). During the October 2014 teleconference about Tanks 5F and 6F grouting operations (ADAMS Accession No. ML14330A037), NRC inquired about the process DOE uses

to reach the desired slump through use of water additions and admixtures. DOE clarified how it provides immediate feedback to the Argos batch plant regarding slump flow test results at the beginning of the day when the trucks reach the site to achieve the desirable slump through addition of admixtures at the batch plant without the need to add additional water at the site. For Type I Tanks 5F and 6F, which were grouted in 2013, DOE used four risers as grout entry points (Risers 1, 3, 5, and 8) and noted no significant issues with filling void space at the top of the tank due to mounding in the Final Configuration Report with less than 2 percent deviation between estimated and actual bulk fill grout volumes (SRR-CWDA-2014-00020).

In 2015, DOE began grouting Tank 16H located in the H-Area Tank Farm Facility. During Tank 16H grouting operations, problematic mounding of tank grout occurred beneath three of four grout-placement risers. Prior to the July 28–29, 2015, onsite observation visit, tank grout was last placed in the tank primary on July 21, 2015. Triple-degree temperatures were recorded nearby in Augusta, Georgia, on both July 20 and 21, 2015. Grout placement was temporarily halted on July 21, 2015 due to excessive mounding. DOE indicated that on that date, grout had mounded up to nearly the top of the tank beneath Risers 2, 6 and 8, with only inches to spare (ADAMS Accession No. ML15239A612). In contrast, approximately 2 ft (0.6 m) of space remained between the grout surface and the top of the tank below Riser 3 (ADAMS Accession No. ML15239A612). DOE attributed mounding in Tank 16H to high ambient summer temperatures that increased the set rate of fresh grout (ADAMS Accession No. ML15239A612), but later acknowledged the potential role that non-optimal grout delivery rates may have played (ADAMS Accession No. ML16167A237). To date, DOE has made no effort to establish a causative relationship or correlate ambient temperatures or grout placement rates with the Tank 16H mounding phenomenon (ADAMS Accession No. ML16167A237), which if undertaken would improve understanding of contributing factors. DOE does not monitor in-tank temperatures (ADAMS Accession No. ML16167A237), which are expected to be dominated by the heat of hydration during grouting operations. While the tanks are located underground and are insulated from surface temperature fluctuations, DOE indicates that ventilation of the tanks introduces ambient air into the tanks and could influence in-tank temperatures during grout hydration.

Because solidified mounds of grout beneath Tank 16H risers could block flow of fresh tank grout into the primary and leave open-air void volumes<sup>1</sup> near the tank top, DOE decided to stop work to provide time to take corrective actions to mitigate the mounding issues in late July 2015. DOE evaluated various alternatives, including use of alternative formulations, use of alternative risers, and use of directional tremies, which could be used to deliver grout at an angle from Riser 3 (ADAMS Accession No. ML15239A612). During the July 28–29, 2015, onsite observation visit, DOE indicated that grout could be delivered to the Tank 16H primary through the 3' 6" Riser (ADAMS Accession No. ML15239A612); however, review of inspection video collected on July 29, 2015, indicated that grout at that location had also nearly reached the top of the tank beneath the riser (SRR-CWDA-2015-00170). In the end, DOE decided to use a more flowable grout formulation to complete grouting of the Tank 16H primary and annulus. Clean cap grout that had been developed for use in Saltstone Disposal Facility (SDF) was selected as the more flowable grout. Clean cap grout is an aggregate-free grout used to cap waste monoliths at the SDF (C-SPP-Z-00012). Pozzolanic and cementitious material components include slag cement (45 weight percent), Class F fly ash (45 weight percent), and

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<sup>1</sup> DOE estimates that the tank was 94 percent full at the time when DOE temporarily stopped grouting operations to prevent plugging the grout access points into Tank 16H (SRR-CWDA-2015-00109).

Portland Type I/II cement (10 weight percent). Water is the only other ingredient (C-SPP-Z-00012). NRC awaits information from DOE to describe the meaning of the term “TEMPER,” which was also listed as a “material” (ingredient) on one clean cap grout batch ticket for Tank 16H (042239).

During review of Tank 16H grouting video (SRR-CWDA-2015-00170), NRC staff observed that many cement trucks discharged tank grout at an overall rate of less than 1 cubic yard per minute, apparently due to temporary shut-downs or what DOE calls *slow rolling*. Slow rolling is the intentional intermittent discharge of grout executed to keep fresh grout flowing and thereby avoid plugging the slick line when cement trucks are slow to arrive due to unanticipated vehicular or traffic issues or to having an insufficient number of trucks in rotation (ADAMS Accession No. ML16111B174). SRNL-STI-2012-00578 indicated that increasing the grout delivery rate by using a higher capacity concrete/grout pump would result in better grout distribution throughout waste tanks, but according to DOE, no subsequent change to grout pump capacity was implemented, nor are there plans to do so in the future (ADAMS Accession No. ML16167A237). SRNL-STI-2012-00578 did not address mounding associated with slow rolling. The Tank 16H grout strategy indicated that having 8 to 10 cement trucks in rotation was ideal (SRR-LWE-2014-00013), whereas the Tank 12H grout strategy later clarified that a grout delivery rate of 8 to 10 trucks *per hour* (SRR-LWE-2014-00147) was ideal. Eight to 10 trucks per hour converts to 56 to 70 cubic yards per hour (assuming discharge of 7 cubic yards of grout per truck), in contrast with Section 3.6.1.2 of the procurement specification, which requires a sustained average delivery of 74 cubic yards per hour during an 8-hr work day (C-SPP-F-00055, Revision 4). The Tank 16H grout strategy stated that “Lessons learned from previous tank closures identified the benefit of a minimal time gap between grout trucks during select lifts in the annulus and the primary tank.” DOE was unclear, however, on what “select lifts” were being referred to<sup>2</sup>. Despite recommendations in SRR-LWE-2014-00013 and SRR-LWE-2014-00147, grout delivery contracts to date have not specified a grout delivery frequency or minimum number of cement trucks in rotation. When questioned by NRC staff about the feasibility of establishing contractual obligations related to the supply of grout trucks in rotation, DOE indicated that such contractual obligations would lead to significantly higher costs, as batch plant demand can be high and DOE has to compete with other customers for grout trucks (ADAMS Accession No. ML16167A237). Instead, DOE contractors work with the batch plant to schedule tank grouting during weeks when the plant can supply more cement trucks to the tank closure effort (ADAMS Accession No. ML16167A237). DOE also noted that while issues with a less than optimal number of trucks in rotation occurred during Tank 12H grouting operations, no significant mounding issues occurred for Tank 12H (ADAMS Accession No. ML16167A237), implying that grout delivery rates are not the sole factor contributing to the phenomena experienced during Tank 16H grouting in the summer of 2015.

At the July 28–29, 2015, onsite observation visit, DOE discussed steps it could take to prevent early tank grout set up and ensure its flowability. These included (i) use of cooling water to keep grout temperatures lower, (ii) slick line (length) adjustments, and (iii) halting grouting operations when ambient temperature exceeds a threshold value. However, during a recent teleconference, DOE indicated that they have not yet placed constraints on grouting operations related to high ambient temperatures beyond rescheduling operations, such as occurred in August 2015, to protect workers from heat effects (ADAMS Accession No. ML16167A237).

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<sup>2</sup> NRC notes that relatively high grout discharge rates during annulus ventilation duct grouting would help ensure the ventilation duct is fully grouted particularly when grouting from outside the duct, as was the case for Tank 16H.

NRC also concurs that, as stated in SRNL-STI-2012-00578, mounding beneath risers can be partially mitigated and grout distribution improved if a sufficient number of cement trucks are in rotation. Allowing grout to flow for only a few minutes followed by a temporary shutdown (slow rolling) results in the premature halt near the discharge point of flowing grout lobes<sup>3</sup> and thereby constructs elevated grout mounds. In contrast, continuous discharge of an entire grout batch enables grout to continuously flow until a barrier (e.g., the tank wall) is reached. If each cement truck were to discharge continuously, there would be less grout buildup around the discharge zone and grout would be more evenly distributed throughout the waste tank. Mounding is exacerbated when grout discharge from a single cement truck starts, stops and restarts. While elevated ambient temperatures may reduce the set time of grout, DOE has not shown that summer temperatures were the leading cause of problematic mounding in Tank 16H.

*Tank Grout Specifications and Testing*

Tank type differences impact the grout placement approach and, potentially, grout performance. Tanks 5F, 6F, 12H and 16H contain many more internal tank obstructions (e.g., cooling coils) than did Tanks 18F and 19F, and have flat, not domed, roofs. The grout specification for Tanks 5F, 6F, 12H, and 16H (C-SPP-F-00055, Revision 4) differed from that of Tanks 18F and 19F (C-SPP-F-00055, Revision 2) only in that a greater slump flow range was specified to enhance grout flowability in tanks containing carbon steel cooling coils. DOE achieved higher slump flow by increasing the dose of high-range water-reducer ADVA Cast 575 (W.R. Grace & Co., Cambridge, Massachusetts) to 40 or 41.25 fluid oz per cubic yard (ADAMS Accession No. ML13267A452; SRR-CWDA-2013-00026, Attachments 3 and 4); however, 40 fluid oz/cubic yard is the maximum amount allowed by the tank grout specification (C-SPP-F-00055, Revision 4, Attachment 5.5). NRC staff reexamined batch tickets provided by DOE for Tanks 18F, 19F, 5F, 6F, and 16H to better understand how admixture dosages have varied during the various tank grouting operations (Table 1). During Tank 16H grouting operations, the dose of ADVA Cast 575 used per batch slightly exceeded the amount specified in C-SPP-F-00055 (Revision 4, Attachment 5.5). Hydration stabilizer RECOVER (W.R. Grace & Co., Cambridge, Massachusetts) dosages also varied, but viscosity modifier EXP 958 (W.R. Grace & Co., Cambridge, Massachusetts) dosages have remained constant at the maximum value allowed (Table 1). It is worthwhile to note that while the use of high-range water-reducer ADVA 575 has increased to achieve greater flowability, the viscosity modifying admixture (VMA), EXP 958 dosage has not changed although VMAs are used to counter-balance the use of high-range water-reducers, which at higher quantities can lead to excessive bleed water segregation.

**Table 1. Evolution of Admixture Dosages Used to Batch Tank Grout.**

Admixture	Dose in Fluid Ounces per 8-cubic-yard Batch					
	Procurement Specification	Tank 18	Tank 19	Tank 5	Tank 6	Tank 16
ADVA 575	80–320	160	160	320	320	330
RECOVER	As Required	30	30	50–60	50–60	30–60
EXP 958	Up to 330	330	330	330	330	330

The Tank 16H Final Configuration report summarized results of tank grout bleed-water testing; although most of the tank grout batches met the zero-bleed requirement (C-SPP-F-00055,

<sup>3</sup>A grout flow lobe is a single fan- or channel-shaped mass of grout that forms on a grout mound as a result of changing discharge or changing direction of flow

Revision 4), some did not (SRR-CWDA-2015-00159). A supplier deviation disposition request (SDDR No. 13307) addressed the two highest bleed water test results, which were 3.3 and 8.9 percent (SRR-CWDA-2015-00159). Following DOE's evaluation, the SDDR was dispositioned as "use as is" (SRR-CWDA-2015-00159). NRC staff will request that DOE provide documentation associated with SDDR No. 13307 for review, and will continue to monitor the extent of bleed water segregation in tank grouting operations. DOE also provided NRC with five accepted grout batch tickets, including two batch tickets for clean cap grout (discussed below), and five rejected grout batch tickets (SRR-CWDA-2016-00031). Reasons given for rejecting grout batches were slump flow test results that were too low or too high, poor mixing, and segregation (SRR-CWDA-2016-00031).

The aforementioned mounding of tank grout beneath three grout-placement risers in Tank 16H resulted in the unanticipated decision to complete Lifts 5 and 6 primary tank and annulus grouting operations with more flowable clean cap grout (C-SPP-Z-00012), which is an aggregate-free grout commonly used for leveling and filling headspace to cap the top of saltstone monoliths at the SDF (SRR-CWDA-2015-00109; SRR-CWDA-2015-00170). Clean cap grout has desirable fresh grout properties such as high flowability and appropriate reductive capacity (SRR-CWDA-2013-00091; SRR-CWDA-2015-00109). Unlike tank grout, however, clean cap grout is a low-bleed formulation (SRR-CWDA-2015-00109), not a zero- or virtually zero-bleed formulation.

Tank grout was designed to provide the following desirable physical and chemical properties: (i) high compressive strength (greater than 2000 psi) and (ii) high degradation resistance to provide stability; and (iii) high pH (e.g., initially assumed to be 11.1 in the HTF PA and grout specification is >12.1), (iv) low hydraulic conductivity (e.g., initially assumed to be  $2.1 \times 10^{-9}$  cm/s in the HTF PA), (v) relatively low porosity (assumed to be 0.21 in the HTF PA), (vi) low effective diffusion coefficient (e.g., initially assumed to be  $5 \times 10^{-8}$  cm<sup>2</sup>/s in the HTF PA) and (vii) low Eh (e.g., initially assumed to be -0.47 V in the HTF PA) to limit migration of contaminants after operational closure (SRR-CWDA-2013-00091; SRR-CWDA-2015-00160).

The HTF PA stated that "the entire tank is assumed to be filled with [tank] grout; therefore structural failure (i.e., collapse) is not considered." The HTF PA assumes that tank grout has adequate compressive strength [i.e., minimum of 2000 psi (138 bars) at 28 days post-placement, per HTF PA Table 3.2-9 (SRR-CWDA-2010-00128)], to withstand the overburden load on each tank<sup>4</sup>, thereby providing stability upon closure and a physical barrier that will discourage intruders. To confirm that this minimum strength was achieved for tank grout placed into Tank 16H, 28-day testing of 272 grout specimens collected at the point of delivery was performed by DOE (SRR-CWDA-2015-00159). The Tank 16H final configuration report discussed an associated deviation from the grout specification that requires test cylinders

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<sup>4</sup> Although DOE indicates that the compressive strength of the tank grout is adequate to withstand the overburden load on each tank, it is not clear to NRC that the tank grout, which is not expected to be fully bonded to the tank and vault, would initially be relied on to accept the load of overlying surface materials, including an engineered cover system to be placed over the tank farms. The reinforced concrete vault will initially be relied on to withstand the overburden load on each tank until such time that the vault fails. When discussing site stability during the July 2015 onsite observation (ADAMS Accession No. ML15239A628), NRC similarly noted that a bounding structural analysis might consider the mass of the tank grout without the associated stiffness of a solid, grout filled monolith, because the tank grout is not expected to create a solid monolith with the tank/vault given the potential for shrinkage and cracking.

be maintained for 28 days in a controlled humidity and temperature environment. The deviation occurred when an equipment failure resulted in a 7-hour window during which temperatures exceeded the specification by up to 2 °C (3.6 °F) (SRR-LWE-2015-00085). Nevertheless, all tested tank grout cylinders had compressive strengths greater than the design 28-day compressive strength of 2000 psi (138 bars) and the average 28-day compressive strength of Tank 16H tank grout was 2,788 psi (192 bars). NRC does not consider the equipment failure and resulting temperature deviation a significant issue with respect to the compressive strength testing.

Because placement of clean cap grout in the Tank 16H primary and annulus was not anticipated during the planning stage, there was no requirement to collect specimens or conduct ASTM C39 compressive strength testing of clean cap grout placed into Tank 16H. However, slump flow data were obtained, according to batch tickets #042239 and #042345, for clean cap grout batches discharged into the primary and annulus, consistent with the procurement specification's (C-SPP-Z-00012) requirement of a field acceptance test for ASTM C1611 slump flow to be in the range of 26–38 in (66–97 cm). Later, even though the Tank 12H subcontractor surveillance plan (LWO-LWQ-2016-00001) anticipated potential placement of clean cap grout into Tank 12H, DOE established no requirements for ASTM C39 compressive strength testing of it. Whereas high-quality tank grout has high compressive strength [ $>2000$  psi ( $>138$  bar)] to provide waste tank stability, and low permeability ( $2.1 \times 10^{-9}$  cm/s) and relatively low porosity (21 percent) to limit percolation of water through the grout matrix (SRR-CWDA-2010-00128), clean cap grout prepared with a low water-to-premix ratio has adequate compressive strengths (SRNL-STI-2012-00558; see also SRR-CWDA-2015-00160), slightly higher mean hydraulic conductivity values ( $2.2 \times 10^{-9}$  cm/s) (SRR-CWDA-2015-00160), but much higher porosity values (~50 to 60 percent) (SRNL-STI-2012-00558; PNNL-20706) by 28-days post-placement, such that its use in Tank 16H may yet enhance the water percolation rate through the grout matrix to the contamination zone. SRR-CWDA-2015-00100 documented DOE's evaluation of the impact of switching from placement of tank grout to clean cap grout in the midst of placing final grout Lifts 5 and 6 into the Tank 16H primary and annulus (SRR-CWDA-2015-00096). DOE concluded that because use of clean cap grout would minimize remaining void space at the top of the tank and be limited to less than 10 percent of the original tank volume, tank stability and the inadvertent intruder barrier provided by the two-component grout monolith would be maintained (SRR-CWDA-2015-00100). During the May 17, 2016, teleconference with NRC (ADAMS Accession No. ML16167A237), DOE clarified that most of the grout placed into Lift 6 in the Tank 16H annulus was tank grout (i.e., 25 or 26 out of 31 truckloads or 81 to 84 percent of the Lift 6 volume). The different characteristic hydraulic properties of tank grout and clean cap grout suggests that a hydrologic barrier (such as a capillary or permeability barrier) may develop at the interface between underlying tank grout and overlying clean cap grout, but potential barriers such as these and their implications on performance have not been addressed by DOE (SRR-CWDA-2015-00100; ADAMS Accession No. ML16167A237).

As of April 2015, DOE was preparing to switch from use of Grade 100 ground granulated blast furnace slag cement in the tank grout formulation (C-SPP-F-00055) to Grade 120 (SRR-LWE-2015-00032). The switch to use of Grade 120 occurred on the second day of Tank 12H grouting in January 2016<sup>5</sup>. In contrast with DOE's position that the hydraulic conductivity of tank

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<sup>5</sup> Prior to the issuance of this TRR, DOE clarified via email on August 15, 2016, that Grouting of Tank 12 was initiated on January 19, 2016, with Lift 1 in the primary (see slide #31 of the OOV presentation material, SRR-CWDA-2016-00009 [ADAMS Accession No. ML16111B232]). Lift 1 was completed the next day, January 20, 2016.

grout will not be impacted by the change in slag grade (SRR-CWDA-2015-00057), differences in particle size and other factors might produce a grout with a different hydraulic conductivity. Had DOE used the same Grade 120 slag-based grout throughout the entire tank, a more homogeneous grout monolith would have developed. NRC will follow-up with DOE with respect to any performance impact associated with use of two different grout formulations of potentially varying hydraulic conductivity.

Finally, NRC concludes that switching from Holcim Grade 100 to Lehigh Grade 120 slag is likely beneficial with respect to the chemical performance of grout placed in Tank 12H due to the higher activity index and reduction capacity. The compressive strength is also expected to increase due to a combination of small particle size and enhanced reactivity of the higher grade slag. Assuming grout performance and testing requirements are met, tank grout comprised in part of Grade 120 slag likely will meet PA assumptions and closure of Tank 12H and those to be closed in the future will likely be carried out in compliance with performance objectives.

### *Grout Segregation*

Prior to the February 2–3, 2016, onsite observation visit, NRC staff reviewed a representative sample of the complete set of video footage that DOE provided of Tank 16H bulk tank and annulus grouting (SRR-CWDA-2015-00170). Grout less prone to bleed water segregation<sup>6</sup> appeared to be placed near the active riser, probably due to locally high elevations associated with mounding. However, NRC consistently observed rapidly migrating dark water exuding (i.e., bleeding) from slowly flowing, light-colored grout lobes as they move away from the discharge zones of Tanks 18F, 19F, 5F, 6F, and now 16H. NRC note the potential for bleed water to segregate from the grout mix during grout flow and distribution throughout the tank, whereby potentially higher water to cement ratio grout is delivered to outlying portions of the tank far from the discharge riser. Dark water emerges from the free surfaces of freshly flowing light-colored grout lobes: from their front edges, side edges, and top surfaces. Numerous examples of bleed water segregation were documented by video cameras in the Tank 16H primary and in the annulus (SRR-CWDA-2015-00170). Camera operators watching a 42-in (107-cm) monitor in the command center (ADAMS Accession No. ML15239A612) seemed interested in this phenomenon based on cameras that focused in on such occurrences and on aqueous ponds forming in low points at tank edges far from the active riser (SRR-CWDA-2015-00170).

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On both days 20 trucks were delivered totaling approximately 64,000 gallons of grout for Lift 1. Lift 1 was initiated utilizing grout made with Grade 100 slag and was switched to Grade 120 slag with the eighth truck delivered on January 20, 2016. Therefore, 27 trucks containing grout made with Grade 100 slag were placed in Tank 12H resulting in approximately 43,00 gallons of grout made utilizing Grade 100 slag. Starting with the eighth truck on January 20, 2016, the remainder of the grouting was done utilizing Grade 120 slag.

<sup>6</sup> The term “bleed water segregation” describes the movement of excess water to the surface of fresh grout (e.g., Wainwright and Ait-Aider, 1995; Olorunsago, 1998; Josserand et al., 2006). Bleeding is visual evidence of the gravitational settlement of heavy aggregate that comprises the granular skeleton of the grout matrix; the consolidation process displaces mix water upward and outward. Bleeding is observed when the bleed rate exceeds the evaporation rate, and the evaporation rate in the high-humidity in-tank environment is likely low. Bleeding is regulated by the particle size distribution of the cement(s) in the grout mix and, in particular, by the quantity and reactivity of the cement(s) (Wainwright and Ait-Aider, 1995). Excessive bleed water production, which has been observed by NRC in video of grouting operations conducted in Tanks 18F, 19F, 5F, 6F, and 16H, could result in significant in-tank heterogeneities (i.e., variable water-to-cementitious materials ratio, compressive strength, and hydraulic conductivity, among other factors).

Based on these observations, NRC staff find that excess water was exuding from the bulk mass of flowing tank grout when it was being distributed throughout Tank 16H, and that this exudate increased the overall volume of water that collected in pools at the tank wall beyond the amount introduced as slick line and tremie lubricant. Mounded grout will hydrate in a relatively dry microclimate, whereas grout submerged under standing water at the tank perimeter will hydrate in a saturated microclimate; because of this, grout properties are unlikely to be uniform (ADAMS Accession No. ML13127A291). Tank grout that hydrates and hardens in a subaqueous environment might be of different quality relative to that forming subaerially, although it is not entirely clear which environment will produce higher-quality, better-performing grout as discussed in more detail below.

When NRC staff reviewed portions of the Tank 5F and 6F grouting video with DOE staff during a March 26–27, 2014, onsite observation visit, DOE staff had said that water pooling in the tank was either chromated water from flushing of failed cooling coils or Slick Willie (Enviro-Systems, Smyrna, Georgia) aqueous pump-priming agent [i.e., a powder added in what amounts to 9 cubic feet (0.25 cubic meters) of water to form a solution] (ADAMS Accession No. ML14342A784). During a teleconference on October 29, 2014, DOE quantified the total volumes of pump-priming solution added to Tanks 5F and 6F. According to DOE's calculations, Tank 5F received approximately 1,050 gal (3,975 L) of aqueous Slick Willie over a 17 partial-day grouting period, while Tank 6F received approximately 875 gal (3,312 L) over a 16 partial-day grouting period; therefore the aqueous solution was added to the waste tanks at an average rate of ~60 gal/day (227 L/day) (ADAMS Accession No. ML14330A037; SRR-CWDA-2014-00029). Slick Willie was added to Tanks 5F and 6F separately from the tank grout that was discharged into each tank, and the two substances were not actively mixed together. Data published by Enviro-Systems about the physical properties of cementitious materials that set up after being thoroughly mixed with Slick Willie are not directly relevant. Thus, NRC cannot rely on such information to support DOE's conclusion that the aqueous Slick Willie solution was incorporated into the grout monolith in a manner that would not negatively impact later percolation of water through the grout monolith (ADAMS Accession No. ML14342A784). Slick Willie was also previously disposed of inside Tanks 18F and 19F, according to DOE (ADAMS Accession No. ML16167A237). As a follow-up action to the May 17, 2016, teleconference, DOE agreed to provide an estimate of the total volume of Slick Willie added to Tanks 18F and 19F.

In the Tank 5F and 6F lessons learned document (SRR-CWDA-2014-00015), Lesson #11 stated that: "Near the end of bulk tank filling, liquid was observed near the bottom of a few risers." DOE had prepared Work Order No. 01199254-65 specifically for such a case in order to remove excess liquid from risers near the end of grouting activities. At the March 26–27, 2014 onsite observation visit, DOE stated, however, that this work order was unnecessary because there was no free water to be removed (ADAMS Accession No. ML14106A573). Ultimately, SRR-CWDA-2014-00015 recommended using less Slick Willie in the future [i.e., <60 gal/day (<227 L)] to minimize potential for unincorporated liquid to be encased in grout inside the tank. In response to that recommendation, DOE indicated that they would simply no longer dispose of Slick Willie in future tanks, beginning with Tank 16H (ADAMS Accession No. ML15239A612; ADAMS Accession No. ML14330A037). Slick Willie pump-priming agent, which is to remain in use at the grout pump, will instead be disposed of by other means (ADAMS Accession No. ML14342A784). During the July 28–29, 2015, onsite observation visit, DOE approximated that from 5 to 7 gal (18.9 to 26.5 L) of water per day (quantity was dependent on the length of the line) was used to lubricate the Tank 16H slick lines and tremies at the beginning of the day and was then discharged into the primary. This occurred daily during 25 days of grouting, summing

to a total of ~175 gal (~660 L) of water added to the primary (ADAMS Accession No. ML15239A612; ADAMS Accession No. ML16167A237). On this basis, approximately an order of magnitude less lubrication water was discharged into Tank 16H than had been discharged into each of Tanks 5F and 6F in the form of Slick Willie solution (ADAMS Accession No. ML16167A237). Likewise, it took 6 days to grout the annulus, so up to 42 gal (160 L) of lubrication water was discharged into it (ADAMS Accession No. ML16167A237). Re-lubrication of slick lines and tremies later in the day is not a typical occurrence (ADAMS Accession No. ML16167A237). NRC staff will continue to evaluate the sources of water that contributed to the development of significant ponds within low spots inside this and other tanks. DOE should continue to minimize or eliminate excess water introduction to waste tanks or provide additional support that the excess water does not negatively impact performance. For example, DOE could provide additional information on bleed water evaporation rates, and could provide additional information that excess water initially present or introduced to Tanks 18F, 19F, 5F, 6F, 16H, and 12H did not reduce the integrity of the various grout monoliths to less than what is assumed in the PA.

### *Grout Drop/Placement Testing*

To date, DOE has delivered grout to the tanks using a Thom-Katt (TK) 70 pump, slick lines, and tremies<sup>7</sup> located no greater than 5 ft (1.5 m) above the grout surface (see Figure 1). For example, Work Order Nos. 01199252-30 and 01199254-18 called for grout to be placed into Tanks 5F and 6F through multiple risers with a maximum drop height of 5 ft (1.5 m). DOE used <5-ft (<1.5 m) drop height to minimize bleed water segregation. During the March 26–27, 2014, onsite observation visit, DOE indicated that their grout team recommended additional testing take place before using a drop height >5-ft (>1.5 m) because the tank grout with higher slump placed into tanks with cooling coils had a greater potential for bleed water segregation (ADAMS Accession No. ML14342A784). The grout drop test report (RPT-5539-EG-0016), detailed in Appendix A, documented the results of three tests conducted to evaluate the potential for bleed water segregation to occur with increased drop height or due to the presence of aqueous pools. The results of three grout drop tests are summarized next, along with DOE's related decisions.

During Test 1, grout was discharged by tremie into a dry mold from a drop height of 5 ft (1.5 m); therefore, this grout drop test case was most similar to current tank grouting operations when and where aqueous pools are absent. A slight decrease in aggregate at the top of a sample taken from the 9 ft (2.7 m) radius position was observed in a sample removed from the monolith of Test 1 (RPT-5539-EG-0016, Page 44 and Figure 32, Sample T1-9-O-1). NRC review of the video of this grout drop test revealed what looked like poorly mixed aggregate rather than well-mixed grout initially discharging to the test pool, although this observation was not discussed in the report.

During Test 2, grout was discharged by tremie into a 4-in (10-cm) standing pool of water from a drop height of 5 ft (1.5 m) and at a relatively low discharge rate of 0.8 cubic yards (0.61 cubic meters) per minute. When grout is discharged from a tremie into a ponded aqueous environment, bleed water segregation may be further enhanced and exacerbated (cf. SRNL-

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<sup>7</sup> A tremie is a long flexible pipe/hose that is inserted into the tank (through one of the tank risers) to guide the placement of the tank closure grout and limit the free fall of the grout (to less than five feet). When the grout level reaches the bottom of the tremie, the tremie is dropped into the tank and a new tremie is placed approximately five feet above the grout (or the maximum allowed free fall height).

STI-2012-546; SRNL-STI-2012-576). RPT-5539-EG-0016 described significant segregation of aggregate from fines, with aggregate remaining near the drop point and also stratifying at low levels due to gravitational effects, while fines and water were rapidly swept out to the tank perimeter (see Pages 20 and 44, Figure 33, Samples T2-5-O-1 and T2-9-O-1). The role that grout discharge rate has in bleed water segregation was not directly addressed by this test, but DOE contractors indicated that while higher discharge rates could lead to greater segregation, DOE did not think that the discharge rate would have a significant impact on the reported results (ADAMS Accession No. ML16167A237). It is also important to note that DOE contractors stated that the grout drop tests (RPT-5539-EG-0016) are not expected to be representative of real tank systems (ADAMS Accession No. ML16167A237)--placing grout into 4 in (10 cm) of pooled water is not typical of grouting operations at the tank farm facilities. DOE emphasized the different mixing energies that are associated with pouring grout directly into standing water versus that of the more typical grout flow lobes as they slide into standing water in low-lying areas of the tanks (ADAMS Accession No. ML16167A237). Likewise, the bench-scale saltstone studies of Cozzi and Pickenheim (SRNL-STI-2012-546; SRNL-STI-2012-576)<sup>8</sup>, summarized in Appendix A, also do not mimic a typical tank grout discharge scenario in any way, and therefore those results are also not directly applicable to tank grouting operations for more reasons than grout formulation alone.



**Figure 1. Grout Drop Test Molds with Boom Pumper Truck and Tremie (Photograph Adapted from RPT-5539-EG-0016).**

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<sup>8</sup> The Cozzi and Pickenheim studies were undertaken to better understand the potential impacts on physicochemical saltstone properties associated with the presence of excess water and indicate positive performance impacts of excess water. As discussed in the Appendix A summary of Cozzi and Pickenheim, DOE tested samples with a water to cement ratio of either 0.6 or 0.64. The water to cement ratios may not represent conditions in the field due to incorporation of excess flush water into saltstone.

Prior to commencing grouting operations in the Tank 12H primary, 3,500 gal (13,250 L) of water remaining in the tank was largely evaporated over the course of approximately 1 year, but residual pools present on the floor of the tank when grouting began were mapped and those areas were avoided during initial grouting of Tank 12H (ADAMS Accession No. ML16111B174). During the February 2016 onsite observation, NRC also noted that DOE had temporarily skipped grouting Lifts 2 and 3 in the Tank 12H annulus. DOE explained that the delay was due to accumulation of water in the annulus from groundwater in-leakage. Based on Test 2 results, DOE indicated in their Tank 12H grout strategy document that they would avoid placing grout directly into wet areas of the tank because doing so could enhance bleed water segregation (SRR-LWE-2014-00147; ADAMS Accession No. ML16111B174). Similarly, Tank 16H video footage dated June 2, 2015, showed standing water in the primary before grouting operations commenced. During a recent teleconference, DOE indicated that sources of water in tanks and annuli prior to grouting may include Slick Willie pump priming agent, bleed water segregation, chromated cooling coil flush water, condensation associated with the ventilation system, and groundwater in-leakage (ADAMS Accession No. ML16167A237).

During Test 3B, grout was dropped in freefall from a height of 42 ft (12.8 m) into a dry mold. The discharge rate used during this test (1.27 cubic yards per minute) was consistent with rates of from 1.0 to 1.4 cubic yards per minute used during actual waste tank grouting under continuous discharge conditions. Bleed water segregation was not visually apparent in the samples collected from this monolith. For Test 3B, grout quality also appeared to be more homogeneous, perhaps due to grout being placed into the containment mold over a wider discharge zone via diffuse freefall. Based on these results, DOE prepared the Tank 12H grout strategy document to allow grout placement absent use of a tremie (SRR-LWE-2014-00147); however, DOE later confirmed that a tremie was used during Tank 12H grouting operations to control grout placement and minimize grout drop height (ADAMS Accession No. ML16167A237). Furthermore, DOE indicated it plans to continue using tremies during future grouting operations (ADAMS Accession No. ML16167A237).

RPT-5539-EG-0016 provides evidence that compressive strength and hydraulic conductivity are initially dependent on distance from the discharge zone, typically exhibiting higher quality properties near the impact point and lower quality properties further away (RPT-5539-EG-0016, see Figures 24–28, 30), although results for Tests 1 and 3B at later times showed an improvement in properties over time (e.g., RPT-5539-EG-0016, see Figures 30 and 31). NRC raised potential technical issues associated with excessive bleed water segregation in ADAMS Accession No. ML13127A291 that are supported by information presented in RPT-5539-EG-0016, specifically, that “Grout matrix porosity and permeability may increase radially due to the shedding of segregated water to zones near [the] tank perimeter.” However, the performance impact of bleed water segregation of SRS tank grouts away from the discharge zone has yet to be determined, and studies conducted by Cozzi and Pickenheim (SRNL-STI-2012-546; SRNL-STI-2012-576) suggest some benefit of excess water with respect to grout performance, although the applicability of the test results to SRS tank grouting is unclear.

Although grouting into pools of standing water is clearly not recommended for tank grouting, the potential differences in the physicochemical properties of reducing grouts associated with grouting in more modest amounts of standing bleed water versus exposure to dry air has also been studied by Cozzi and Pickenheim (SRNL-STI-2012-00546; SRNL-STI-2012-00576) albeit for different grout formulations than used in SRS tanks. For more information, see the reference summaries in Appendix A. During this study, saltstone grout specimens that were exposed to

ambient air during hydration exhibited hydraulic conductivities that were three orders of magnitude greater than those of specimens hydrated in a high humidity environment, perhaps due to development of shrinkage microcracks. Moreover, and perhaps partly because of their relatively high hydraulic conductivities, specimens that became hydrated and potentially oxidized while exposed to ambient air exhibited the greatest tendency to leach incorporated constituents. For specimens hydrated in sealed, high-humidity containers, neither the water-to-premix ratio nor excess standing water had appreciable effects on hydraulic conductivity magnitude and leachability. Saltstone grout specimens that were developed by pouring grout into 10 percent excess salt solution exhibited a 4 to 5 percent increase in density from top to bottom, due to enhanced bleed water segregation and settling in an environment containing excess water. Likewise, seven of eight samples tested exhibited density-dependent effects on hydraulic conductivity, but the effects were not significant enough to effect grout quality. Overall, this study demonstrated that premature drying of reducing grouts during early hydration is detrimental to their quality with respect to isolating waste. Therefore, maintaining a moist environment inside tanks and vaults is critical to development of high-quality grout properties, including the relatively low values of hydraulic conductivity assumed in the tank farm PAs.

As stated above, during the May 17, 2016 teleconference, DOE indicated that the results of the grout drop test report are not thought to be representative of grouting operations at the Tank Farm. Moreover, DOE indicated that while some bleed water segregation should be expected, the grout does not mix with the excess water and that the quality of the grout should not be considered lower quality (ADAMS Accession No. ML16167A237)<sup>9</sup>. Nonetheless, NRC concludes that DOE has not ruled out the potential for a dependence of grout quality on distance from the drop point and that the presence of ponded water during grouting operations may serve to enhance the natural bleed water segregation that appears to be inherent to the tank grout mix<sup>10</sup>. Therefore, DOE should continue to make an effort to remove copious amounts of excess ponded water from waste tanks and annuli before and during grouting operations, whenever aqueous ponds are present, to increase the likelihood that higher-quality grout that meets the assumptions of the HTF and FTF PAs is placed into waste tanks and annuli. While it appears clear that aqueous ponds should be avoided, it is unclear what quantity of excess water will lead to undesirable grout properties. Thus, NRC will continue to monitor DOE grouting operations and evaluate testing designed to better understand the performance impact of excess water in SRS tanks during grouting.

With respect to use of Grade 120 slag, NRC expects that switching from coarser-grained Grade 100 slag cement to finer-grained Grade 120 slag cement may reduce bleed water segregation (SCA, 2002; Topçu and Elgün, 2004) because Grade 120 slag is more reactive than Grade 100, and can thereby induce faster kinetics and more rapid hydration reactions (ADAMS Accession No. ML16167A237). Staff will continue to evaluate the extent of bleed

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<sup>9</sup> It is worth noting that Center for Nuclear Waste Regulatory Analysis (CNWRA) staff, who tested intermediate-scale, reducing grout physical analog monoliths, observed permeability variations ranging over five to seven orders of magnitude due to the presence of cracks and shrinkage gaps (Dinwiddie et al., 2012; Table 4-1), whereas DOE maintains that they expect permeability of tank grout to vary over no more than one order of magnitude (ADAMS Accession No. ML16167A237).

<sup>10</sup> Based on video observation of tank grouting (SRR-CWDA-2015-00170), bleed water segregation may be endemic to the tank grout mix (C-SPP-F-00055) (e.g., see earlier discussion and summary of Tank 18F video observations contained in ADAMS Accession No. ML13127A291).

water production in Tank 12H and other tanks to be grouted in the future to verify NRC expectations.

### *Grout Shrinkage*

Grout expands during the heating process associated with hydration and then shrinks during cooling, leaving annular shrinkage gaps between the bulk grout mass and other paraphernalia inside tanks and tank annuli, including gaps at walls, around carbon steel cooling coils, equipment, and between adjacent grout flow lobes. Tank grout and clean cap grout are not shrinkage compensating grout mixes. DOE is not planning to develop a shrinkage compensating grout formula due to the low priority they place on this issue and inadequate funding levels (ADAMS Accession No. ML14342A784) and it deleted use of shrinkage-compensating admixtures from Revision 3 of the tank grout procurement specification (C-SPP-F-00055). NRC staff view work to develop shrinkage-compensating grout formulas as potentially important to the adequate closure of tanks. The NRC staff concurs with Stefanko and Langton's (2013) recommendations in SRNL-STI-2011-00551 for testing of shrinkage-compensating admixtures and implementation of measures to help mitigate tank grout shrinkage. DOE should consider giving higher priority to development of a shrinkage compensating grout formula and to its subsequent testing.

In SRNL-STI-2011-00551, Stefanko and Langton (2013) described how H.N. Guerrero designed instrumented shrinkage characterization test forms and a test protocol for measuring dimensional changes of the tank grout as a function of temperature, time, and relative humidity. These shrinkage tests were postponed by Tank Closure Project personnel, but DOE is now reengaged in performing ongoing ASTM C157 shrinkage testing of tank and clean cap grouts by testing both fully submerged (subaqueous) grout samples and fully subaerial grout samples hydrated in nearly 100 percent relative humidity conditions (ADAMS Accession No. ML16111B174; ADAMS Accession No. ML16167A237). The conditions under which some of the ASTM C157 shrinkage test(s) were performed may not be directly relevant to subaerially mounded tank grout hydrating inside waste tanks, yet others may be representative of conditions affecting grout that hydrates inside waste tanks beneath pools of excess water. Shrinkage of subaerial grout may increase significantly as relative humidity decreases below 100 percent. The extent of shrinkage occurring inside waste tanks may be greater than implied by DOE's shrinkage testing results due to tank ventilation ongoing during grouting operations that would reduce relative humidity by some unknown amount. DOE estimated that ventilation rates in the Tank 16H primary and annulus were 24,000 cubic feet per hour and 11,000 cubic feet per hour, respectively, during grouting (ADAMS Accession No. ML16167A237). DOE is testing shrinkage of clean cap grouts prepared with two water-to-premix ratios (0.45 and 0.5) and Grade 120 slag (the clean cap grout placed into Tank 16H was formulated with a water-to-premix ratio of 0.5 and Grade 100 slag) (ADAMS Accession No. ML16167A237). Therefore, the applicability of eventual test results is unclear. The shrinkage testing reports will not be completed until December 2016 (tank grout) and June 2017 (clean cap grout) at the earliest (ADAMS Accession No. ML16167A237).

If percolating water flow occurs primarily through cracks or gaps in the tank grout, it may come into contact with a much smaller fraction of the tank grout than it would if traveling through the matrix, leading to rapid, localized depletion of the reductive and buffering capacity of the tank grout along preferential fast flow paths. Because DOE assumes that the solubility of certain key

radionuclides is dependent on the chemistry of the percolating water, with lower solubility expected for water adequately conditioned by its interaction with tank grout, the extent of interaction between percolating water and tank grout is risk-significant. NRC staff will continue to evaluate the potential for annular and shrinkage gaps to form around internal tank fixtures, at tank walls, and between individual grout flow lobes, which may lead to bypass of the reducing grout matrix in preference for faster flow pathways, resulting in less water conditioning.

The impact of bypassing flow through the tank grout is being studied by NRC's contactors at the Center for Nuclear Waste Regulatory Analyses (CNWRA). CNWRA has performed water conditioning experiments using a synthetic SRS groundwater interacting with (i) an early generation reducing grout and (ii) tank grout prepared with Grade 100 slag cement (i.e., according to C-SPP-F-00055). Dynamic flow tests using early generation reducing grout specimens indicated that the pH of synthetic SRS groundwater increases to a value above 10 pH units almost immediately after contacting the grout (Walter and Necsoiu, 2015). Static tests of nominally 0.06-in<sup>3</sup> (1-cm<sup>3</sup>) specimens of tank grout with a grout-to-water-mass ratio of 0.7 resulted in a steady Eh of -77 mV after 5 days with dissolved oxygen concentrations <8 ug/L. Two subsequent static tests using the same grout specimens resulted in steady Eh values of -10 to -20 mV after 3 to 4 days. Although the static tests attempted to maximize interaction between the grout and water through use of small cubes of tank grout, the low, initial Eh values of -470 mV assumed in DOE's PA were not achieved in these tests. CNWRA staff more recently prepared tank grout specimens with Grade 120 slag cement and plans to test the grout prepared with the Grade 120 slag in future water conditioning experiments.

The distinction between slow matrix flow through reducing tank grout versus rapid bypass flow through shrinkage gaps and cracks is important to performance. NRC staff acknowledges the difference in impact to performance of distributed grout shrinkage within a Type I waste tank containing ~6.9 km (~4.3 mi) of cooling coils (SRR-CWDA-2010-00128) or a Type II waste tank containing ~9.0 km (~5.6 mi) of cooling coils (SRR-CWDA-2015-00159) and the potentially more focused grout shrinkage expected to occur in a tank without cooling coils. Shrinkage away from carbon steel cooling coils may serve to somewhat increase gap-localized interaction between groundwater and tank grout and result in greater water conditioning compared to a case where focused and larger-scale shrinkage away from the tank wall dominates all water flow as may be the case for Type IV tanks. If grout shrinkage were to occur around cooling coils as well as at grout flow lobe interfaces and monolith edges at the tank or annulus wall and at structural columns/equipment, infiltrating water could be directed through the interior of the tank via many more distributed preferential pathways, potentially conditioning infiltrating water more than in the absence of cooling coil annular shrinkage gaps. Nevertheless, NRC staff expects DOE to provide information and test results sufficient to evaluate conceptual models reflecting preferential flow through tank grout monoliths, whether that flow occurs around the cooling coils, at the interface of individual grout flow lobes, along the length of large-scale, vertically oriented equipment, or along tank and annulus walls, if preferential flow through such pathways cannot be ruled out.

#### *Groundwater In-Leakage in Tank 12H*

Groundwater in-leakage into SRS tank systems has been documented in tank inspection and other DOE reports. During the February 2016 onsite observation visit, NRC inquired about the delay in placement of Lifts 2 and 3 in the annulus of Tank 12H. DOE indicated that placement of these grout lifts was delayed due to the presence of approximately 6 in (15 cm) of

groundwater that had accumulated in the annulus over a 5-day period (ADAMS Accession No. ML16111B174), beginning immediately after the temporary ventilation system, which forced unheated air through the annulus, was shut off (ADAMS Accession No. ML16167A237). Approximately 9 in (23 cm) of groundwater was present in the annulus during the onsite observation visit (ADAMS Accession No. ML16111B174). Observations indicated that groundwater accumulated faster in the annulus when air was “pulled” under negative pressure rather than “pushed” through the annulus using positive pressure (ADAMS Accession No. ML16167A237 and SRR-LWE-2015-00048). DOE estimated that ~1000 gal (3,785 L) of groundwater was pumped out of the Tank 12H annulus (ADAMS Accession No. ML16167A237). DOE staff indicated during the February onsite observation that they expected water levels in the annulus could be reduced to no more than 2 in (5 cm) by pumped removal. If there was 2 in (5 cm) of standing water left remaining in the annulus when grouting began, bleed water segregation likely would have been enhanced (cf. RPT-5539-EG-0016, Test 2), which may or may not have had significant adverse impacts on hydraulic conductivity of the tank grout (cf. SRNL-STI-2012-00576). DOE was also prepared to remove accumulated water from the annulus into decant totes after annulus grouting had begun. During the May 17, 2016, teleconference, NRC inquired whether DOE had placed constraints on grouting operations related to accumulation of water in the tanks. DOE indicated that it uses expert judgement based on when grouting is performed (ADAMS Accession No. ML16167A237). DOE indicated that the water level in Tank 12H was measured using a steel tape. That is, workers dropped a measuring tape with a weight attached into the water, and used a camera to read the water level from the tape (ADAMS Accession No. ML16167A237)<sup>11</sup>. DOE also has data on pumped water volumes removed from Tank 12H. NRC staff will follow-up with DOE on the amount of groundwater in-leakage that was pumped out of the primary or from risers. DOE staff indicated that water was pumped out of every Tank 12H riser. Dehumidification with heating was employed in the tank primary (ADAMS Accession No. ML16167A237). During the May 17, 2016, teleconference, DOE indicated that it is working with SC DHEC to enable original, operational ventilation systems to remain in place during future grouting operations (ADAMS Accession No. ML16167A237) to better manage excess moisture.

DOE indicated that no water had accumulated in the Tank 16H annulus at the start of grouting. DOE indicated that water observed in the primary at the start of grouting was related to cooling coil flushing (ADAMS Accession No. ML16167A237). Unlike Tank 12H, only the bottom of Tank 16H is typically located below the water table.

#### *Follow-up Items on Tanks 5 and 6*

During an October 29, 2014, teleconference between NRC and DOE, batch ticket #039403 associated with a grout load placed into the Tank 6F primary on August 28, 2013, was discussed (see ADAMS Accession No. ML14342A784). At that time, a remark on the batch ticket suggested that this grout batch had been rejected, even while it had been placed into the tank in its entirety. However, SRR-CWDA-2016-00031 indicates that the grout batch represented by batch ticket #039403 was unremarkable and not rejected, and that the prior grout batch associated with ticket #039402 had been rejected after up to 4 cubic yards of out-of-specification grout had been discharged into the tank through Riser 5. A construction

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<sup>11</sup> During tank operations, probes monitor water levels inside the tank. During tank isolation, however, the probes are removed from service and a video camera is utilized to monitor water levels thereafter (ADAMS Accession No. ML16167A237).

discipline engineer did not approve of the runny appearance of the grout observed in the hopper (ADAMS Accession No. ML16111B174), halted the discharge, and rejected the entire grout batch even though up to half of the batch had already been placed (SRR-CWDA-2016-00031). Grout batch ticket #039402 was recently provided for NRC review as Attachment 1 to SRR-CWDA-2016-00031. Informational “info” slump flow test results were not recorded on this batch ticket, and while the discharge start time was recorded, the discharge stop time and total time *en route* to discharge were not recorded. DOE confirmed that the construction discipline engineer monitored the grout added to a TK 70 trailer-mounted concrete pump to provide a qualitative check on the grout quality and had the ability to reject a batch at any time (ADAMS Accession No. ML14342A784). As recommended by NRC staff in the Tank 5F and 6F technical review report (ADAMS Accession No. ML14342A784), DOE further investigated the situation and clarified during a February 2–3, 2016, onsite observation visit that this rejected grout batch lacked its full measure of aggregate (ADAMS Accession No. ML16111B174), despite the batch ticket indicating otherwise. Argos, the batch plant operator, and its personnel who observe preparation of each grout batch, were unable to identify a problem with the computer system used to batch the tank grout ingredients in their specified quantities (ADAMS Accession No. ML16111B174). DOE was unaware of similar problems occurring at the Argos batch plant previously, or of any similarly out-of-specification grout discharged into waste tanks during prior grouting operations (ADAMS Accession No. ML16111B174). In partial response to Action Item 5 resulting from the July 2015 onsite observation visit (SRR-CWDA-2015-00170), DOE determined that no changes in procedure were needed because the construction discipline engineer’s quality control check had worked as intended. Based on this event, DOE has provided additional training to workers regarding conditions to watch for during the visual quality control check (ADAMS Accession No. ML16111B174).

Finally, NRC staff previously addressed a non-conformance whereby all grout placed into Tank 6F on September 19, 2013, contained excess water of approximately 2 to 3 percent [i.e., 1–2 gal (3.8–7.6 L) per cubic yard more than specified by C-SPP-F-00055, Attachment 5.5] (ADAMS Accession No. ML14342A784). Recent review of associated video footage indicated that this grout was placed in the tank via Riser 5 (SRR-CWDA-2014-00029). A total of 25 to 30 truckloads of relatively high water content grout was placed through a single riser over the course of a day. There is a potential for a higher permeability zone associated with a larger water-to-cementitious materials ratio to have formed in this area that may increase flow at this location relative to other areas. NRC staff concludes that DOE’s procedures may not prevent significant quantities of lower quality grout from being discharged into waste tanks of visual cues are too subtle to observe a difference in grout properties, as was the case when a day’s worth of grout with excess water was placed in Tank 6F. However, the procedure may identify grout that is grossly out of specification, if that grout is observed to behave or appear significantly different than other truckloads of grout discharged to the tanks or annuli as was the case when a partial truckload of grout with insufficient aggregate was placed in Tank 6F.

### *Alkali–Silica Reactivity*

Alkali–Silica Reaction (ASR) is a slow process whereby the siloxane groups in siliceous minerals in the coarse aggregate (e.g., granite pea gravel in tank grout) are attacked by hydroxyl ions in highly alkaline pore solutions, resulting in formation of an alkali–silica gel (ADAMS Accession No. ML112241029; Qu et al., 2015; Sadek et al., 2016). Alkali–silica gel increases in volume (swells) with water imbibition and causes expansion, which may result in spalling, cracking and deterioration of stiffness and strength (Qu et al., 2015; Sadek et al.,

2016). Reactive siliceous minerals include chert, quartzite, opal, and strained quartz crystals (ADAMS Accession No. ML112241029).

At the 28<sup>th</sup> Regulatory Information Conference, new research programs on “Alkali–Silica Reaction Degradation in Nuclear Concrete Structures,” being executed by (i) the National Institute of Standards and Technology (NIST; Sadek et al., 2016), (ii) Electric Power Research Institute (EPRI) and DOE (Qu et al., 2015; Guimaraes et al., 2016; Sellier et al., 2016; ORNL/LTR-2015/407), (iii) Institute for Radiological Protection and Nuclear Safety (IRSN; Marquié, 2016), and (iv) Organisation for Economic Co-operation and Development (OECD) and the Nuclear Energy Agency’s (NEA) Committee on the Safety of Nuclear Installations (CSNI; Orbovic, 2016), were described. Many standard tests for ASR susceptibility are of limited use for common, moderately reactive aggregates (Guimaraes et al., 2016). There are no non-destructive evaluation tests that can reliably (i) identify ASR or (ii) determine the extent of ASR in a structure (Qu, 2015), but EPRI has developed six large-scale specimens that exhibit varying degrees of ASR for use in development of non-destructive evaluation methods (Guimaraes et al., 2016), and IRSN has just begun a 10-year effort in this arena (Marquié, 2016). Development of ultrasonic and acoustic emission<sup>12</sup> non-destructive evaluation methods are underway (Qu et al., 2015; Guimaraes et al., 2016; Marquié, 2016). NIST is developing strategies for identifying and quantifying reactive phases in aggregates (Sadek et al., 2016). Related ASR research, including numerical process modeling of the evolution of degradation and structural response (e.g., Sellier et al., 2016), is ramping up, but there were few results to report, with the exception of those obtained by Qu et al. (2015). ASR degradation numerical modeling workshops to analyze and interpret preliminary and final results are anticipated in 2016 and 2017 (Sellier et al., 2016).

In the technical review report concerning Tank 18F and 19F grouting operations (ADAMS Accession No. ML13269A365), NRC staff evaluated tests that DOE conducted for ASR susceptibility. NRC staff is concerned that DOE’s criterion for acceptance of vendor-supplied granite aggregate relies on short-term tests (ASTM C1260) that are unlikely (i) to predict ASR susceptibility over the long performance period and (ii) to demonstrate compliance with the performance objective specified at 10 CFR 61.41. NRC staff continues to recommend that DOE consider conducting tests to evaluate the potential for ASR degradation to occur to tank grout, given anticipated water ingress and environmental conditions (i.e., temperature and humidity variations), pore water alkalinity, and reactive aggregate volume fraction, and the potential effects of ASR on long-term performance of the engineered barrier system. Staff will continue to evaluate this technical issue during future monitoring activities.

### *Thermal Data Evaluation*

During a teleconference on October 29, 2014, the NRC requested from DOE any available information regarding adiabatic temperature rise in tank grout (ADAMS Accession No. ML14330A037). DOE stated that they may instrument one or more tanks with thermocouples prior to grouting activities to obtain field-relevant temperature data (ADAMS Accession No. ML14330A037). DOE also indicated that the thermal data collected during the Tank 18F and 19F Grout Scale-Up Test indicate that the semi-adiabatic temperature rise was 23 °C (41 °F),

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<sup>12</sup> Attachment 2 contains information on work commissioned by NRC at the Southwest Research Institute on the feasibility of use of acoustic emission monitoring to better understand the potential for and extent of crack formation in large, underground grout monoliths used for waste isolation at Savannah River Site.

which met the thermal objective for grout that can be mass placed (SRNL-STI-2011-00749). However, as stated in a NRC staff technical review report for Tanks 18F and 19F grouting operations (ADAMS Accession No. ML13269A365), it is not clear that the temperature profile and evolution measured in the test form are representative of temperatures and temperature gradients that would be attained during grouting of large waste tanks. NRC staff notes that environmental monitoring of hydrating tank grout, particularly in regard to its thermal evolution, would yield valuable data that is relevant to grout porosity, hydration products, and the potential for thermal cracking to occur within the various tank types. During the July 28–29, 2015, onsite observation visit, however, DOE informed NRC staff that it had no plans to install thermocouple strings or similar sensors in a waste tank to measure spatiotemporal temperature variations and gradients attained during grout hydration (ADAMS Accession No. ML15239A612). NRC's recommendations concerning thermal characteristics of the tank grout remain the same as those for Tanks 18F, 19F, 5F, and 6F. Specifically, DOE should:

- Conduct a more detailed thermal analysis that considers tank-specific grout pouring sequences and geometries to determine the potential for thermal cracking of the tank grout.
- Measure the adiabatic temperature rise and thermal properties of the grout once placed in the tank, because temperatures/temperature gradients attained may influence porosity, hydration products, and the potential for thermal cracking.

NRC staff will continue to evaluate this technical issue during future monitoring activities.

#### Annulus and Ventilation Duct Grouting

The East and West risers were used to grout the Tank 16H annulus. For improved visibility of annulus grouting operations, the NRC staff previously recommended that DOE consider placing video cameras in *all* tank annuli risers, if available, or else occasionally reposition cameras during grouting operations if cameras could not be placed in all available risers simultaneously (ADAMS Accession No. ML14342A784). However, video cameras were located only in the East and West Risers of Tank 16H, in contrast to the strategy employed during Tank 5F and 6F annulus grouting when cameras were only installed in the North and South Risers, approximately 90 degrees from the nearest discharge riser in Tank 5F and 6F (the East and West Risers were used to place grout in Tank 5F and 6F). During a March 26–27, 2014, onsite observation visit, DOE indicated that the visibility of the Tank 5F and 6F annuli was approximately 50 percent (ADAMS Accession No. ML14342A784); visibility of the Tank 16H annulus likely was similar. During the February 2–3, 2016, onsite observation visit, DOE stated that Tank 12H annulus cameras were located in the East and West Risers and one may be placed in the South Riser, but that none could be placed in the North Riser (ADAMS Accession No. ML16111B174).

The Tank 16H and 12H closure modules (SRR-CWDA-2013-00091 and SRR-CWDA-2014-00086) suggested that grouting the annulus ventilation duct might require a grout more flowable than tank grout; however, neither grout strategy document (SRR-LWE-2014-00013 or SRR-LWE-2014-00147) addressed the issue. Later, DOE reiterated the potential use of a more flowable, but otherwise undefined grout during the February 2–3, 2016, onsite observation visit (ADAMS Accession No. ML16111B174). During a recent teleconference, however, DOE

indicated that only tank grout was used to fill the Tank 16H duct, including its vertical section (ADAMS Accession No. ML16167A237).

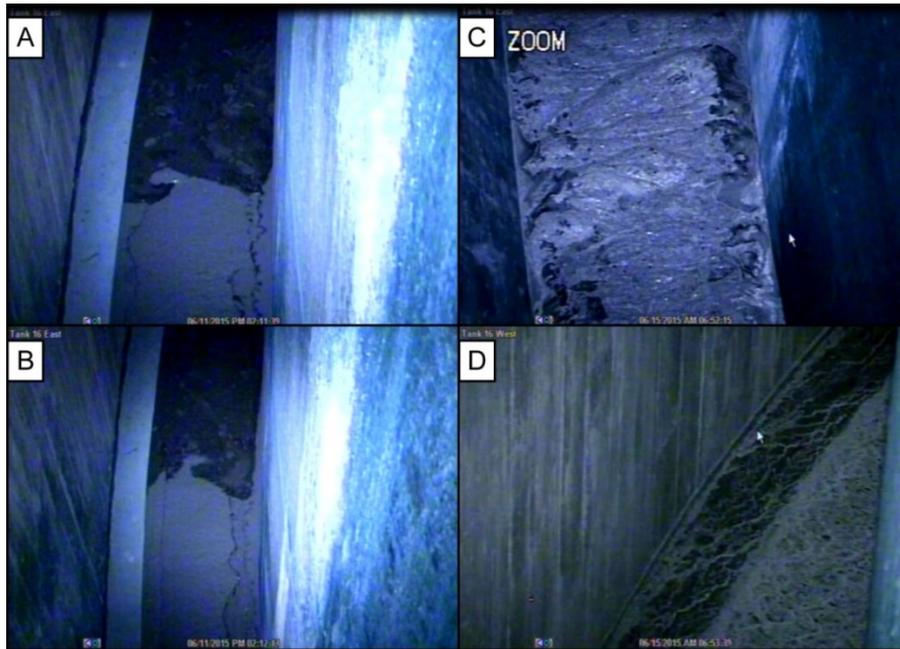
During the July 28–29, 2015, onsite observation visit, DOE staff described Tank 16H annulus grouting operations of the 12-to-18 in (31-to-46 cm)-diameter horizontal ventilation duct, the top of which is 18 in (46 cm) above the annulus floor (ADAMS Accession No. ML15239A612; SRR-CWDA-2013-00091, Figure 7.3-3; ADAMS Accession No. ML16167A237). The diameter of the horizontal ductwork tapers from 18 in (46 cm) at its junction with the vertical inlet and exhaust ductwork to 12 in (31 cm) on the opposite side of the annulus (ADAMS Accession No. ML16167A237; Tank 16H Ventilation Diagram W163389). In contrast to structural-support-related grouting procedures used when grouting Tanks 5F, 6F, and 12H, pre-grouting of the Tank 16H annulus pan to the base of the smallest horizontal duct, where it is at most 6 in (15 cm) above the pan, was not undertaken. Also in contrast, the horizontal ductwork was filled on June 11, 2015, with grout via open registers during bulk filling operations, and direct filling of the horizontal ductwork through its vertical inlet and exhaust ventilation ports was not performed (ADAMS Accession No. ML15239A612; SRR-CWDA-2015-00095). DOE noted that the degraded condition of the Tank 16H ductwork, including broken, open seams, rust holes, and punched holes from removal of sample coupons, led to the decision to grout it from the outside, using its 16 rectangular registers, sized 14 in by 6 in (36 cm by 15 cm) and spaced 17 ft (5 m) apart, as well as other failure-point openings to deliver grout into the duct from both above and below (ADAMS Accession No. ML16167A237). A 10-to-15-ft (3.0-to-4.6-m) arc length of the annulus contained no horizontal duct (ADAMS Accession No. ML16167A237). From the vantage point of the two cameras that viewed the annulus, there were 8 or 9 registers that could not be observed (ADAMS Accession No. ML16167A237). During the May 17, 2016, teleconference, DOE indicated that if the horizontal ductwork is substantially intact, they will always fill waste tank ventilation ducts from inside via the vertical inlet and exhaust ducts as grout entry points (ADAMS Accession No. ML16167A237); such was not the case for Tank 16H.

Also during the July 28–29, 2015, onsite observation visit, DOE indicated that residual waste in the Tank 16H ductwork (e.g., U-ESR-H-00113, Attachment 25—Tank Annulus Waste Heights) could block the flow of grout into the duct (ADAMS Accession No. ML16111B174). DOE visually confirmed entry of grout into some registers in the field of view of the East Riser camera, but could not evaluate volumetric grout uptake by the duct because its volume was negligible compared to that of the bulk annulus fill (ADAMS Accession No. ML15239A612; ADAMS Accession No. ML16167A237). DOE provided NRC with video acquired on June 11, 2015, of initial grout inflow into the ventilation duct (SRR-CWDA-2015-00170), but no video was available from the West Riser camera during initial grouting operations due to the inopportune direction the camera was pointed when grout began flowing into nearby registers. The continuity of grout placement is particularly important when contaminated ducts are being filled. Uninterrupted flow of grout is essential to ensure that permanent porosity does not develop inside ductwork. Attachment 1 provides a description of NRC staff observations of the Tank 16H horizontal ductwork grouting process. During the May 17, 2016, teleconference, NRC asked DOE to provide additional supporting information to justify its confidence that the horizontal ventilation duct in Tank 16H had been filled. DOE indicated that the large number of openings in the duct (see Tank 16H Ventilation Diagram W163389) and the direct video observations in the Command Center of grout entering some of the registers in the duct were sufficient justification to provide confidence that the duct had been completely filled on June 11, 2015 (ADAMS Accession No. ML16167A237). While DOE's approach to verifying placement of grout in the ventilation duct is reasonable, DOE should attempt to place and position cameras in

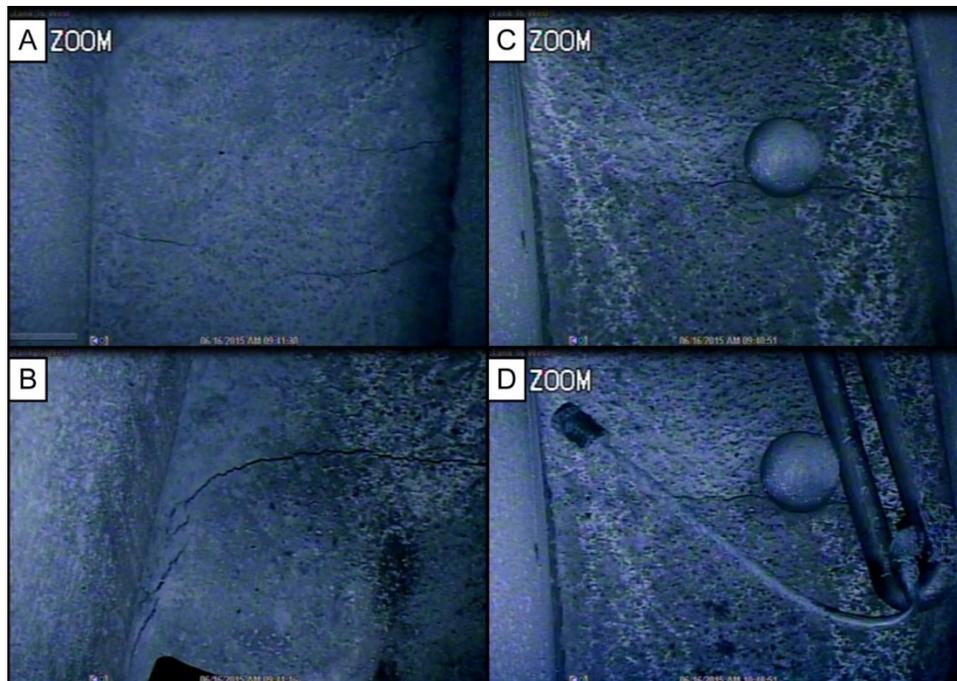
such a manner to maximize visualization of grout entry and exit from duct registers. Improved visualization will increase the evidentiary support for void volume in the ventilation ducts having been filled and will enhance DOE's ability to develop lessons learned related to grout placement strategies that will increase the likelihood that ducts are fully grouted and do not contain risk-significant void space.

With one exception, all tanks grouted to date had their vertical inlet and exhaust sections of their ventilation ducts filled nearly simultaneously with the placement of grout in the annuli outside each duct to ensure that structural integrity of each duct was maintained. For Tank 16H, however, DOE did not exercise the same abundance of caution. The Tank 16H procedure simply assumed that there was no danger of the duct being crushed by pressure applied by the external grout fill in the annulus (ADAMS Accession No. ML16167A237). When questioned, DOE indicated that staff observed no evidence of duct collapse in Tank 16H, yet DOE indicated that it returned to the typical, cautious grouting approach for alternating between grouting the annulus and the vertical duct in Tank 12H (ADAMS Accession No. ML16167A237).

During the July 28–29, 2015, onsite observation tour, NRC staff noted discoloration in the annulus grout, or mottling that may have indicated bleed water segregation (e.g., Figure 2). The camera operator indicated that there were shadows around the edges of flow lobes, but not necessarily cracks; however, he noted that surficial microcracks were sometimes observed on the grout surface (ADAMS Accession No. ML15239A612). During review of Tank 16H annulus-inspection video acquired on the mornings of June 15 and 16, 2015 (SRR-CWDA-2015-00170), NRC staff noted the presence of large-aperture cracks in grout that had been placed beneath the West Riser on June 11, 2015. During the inspections, camera operators seemed interested in viewing the wide-aperture cracks. Many were oriented radially (Figure 3), but one system of cracks was *en echelon* parallel to the inner wall and then rotated to radial (Figure 3B). Cracks were noted on either side of a tremie that had been dropped onto the grout surface on June 11, 2015 (Figure 3A and 3B). Another crack was located beneath and to the side of a pig released on June 11, 2015 (Figure 3C and 3D). During the May 17, 2016, teleconference, DOE indicated that more cracks were observed later, at higher levels in the annulus near the top of the tank, which they attributed to ventilation-induced drying (ADAMS Accession No. ML16167A237). DOE staff indicated that cracks were observed to have developed beneath pour points approximately six times during grouting operations, which they attributed to differential topography associated with mounding. Cracks were also observed in grout in the primary, which DOE attributed to drying (ADAMS Accession No. ML16167A237).



**Figure 2. A and B. Onlapping Black Bleed Water and Tank Grout Flowing Beneath. Date of Video: June 11, 2016. C. and D. Mottling Evidence of Black Bleed Water after Tank Grout Dried. Date of Video: June 15, 2016.**



**Figure 3. Cracks Formed in Tank Grout in the Tank 16H Annulus Beneath the West Riser. A. Cracks Formed North of the Discarded Tremie. B. *En Echelon* Cracks Formed South of the Discarded Tremie. C. Crack Formed Beneath Discarded Pig Further South of the Discarded Tremie. D. Jet Dropped Above Crack at Pig. Date of Video: June 16, 2016.**

During the February 2–3, 2016, onsite observation visit, DOE noted that placement of Lifts 2 and 3 in the Tank 12H annulus had been delayed due to the presence of accumulated groundwater in the annulus. While DOE pumped out the majority of standing water and waited for the remainder to evaporate, Tank 12H grouting proceeded directly from placement of Lift 1 to Lift 4 in the primary (ADAMS Accession No. ML16111B174).

The NRC staff will continue to evaluate technical issues associated with grouting the annuli, ventilation ducts, and annulus risers of waste tanks during future monitoring activities.

### Equipment Grouting

SRNL-STI-2011-00592 provides the grout formulation (T1A-62.5FA) used to grout equipment remaining in Tanks 16H and 12H. The equipment grout formulation called for use of Grade 100 ground granulated blast furnace slag cement, but DOE planned to use Grade 120 to grout Tank 12H equipment<sup>13</sup>. DOE indicates that there are no regulatory requirements for the properties of equipment fill grout, so there are no quality control test requirements associated with production of this grout (SRR-LWE-2014-000150 and SRR-LWE-2015-00032). The equipment fill grout is mixed by SRR Construction and work is controlled via work package (SRR-LWE-2014-000150 and SRR-LWE-2015-00032). During the February 2–3, 2016, onsite observation visit, DOE staff described equipment grouting operations (ADAMS Accession No. ML16111B174; see also SRR-CWDA-2015-00095). Equipment grout was prepared onsite in small batches. SRR personnel measured the dry ingredients by weight, pre-mixed them, and then combined the premix with water per the formulation. The mixture was allowed to hydrate using a low shear mixer, and then a high shear mixer was engaged to finish mixing the equipment grout and thin it, after which there was a short timespan before it would begin to set up. Equipment grout was metered as it was placed into small openings in each piece of equipment using gravity driven flow through a hose and funnel. Tank equipment had high point vents that collected overflow and indicated that equipment filling was complete (SRR-LWE-2014-00013). The volume of grout accepted by each piece of equipment was recorded. During the July 28–29, 2015, onsite observation visit, DOE indicated that the mounded tank grout placed into Tank 16H was only 1 to 2 ft (0.3 to 0.6m) from the top of the tank, and that contractors had begun to perform equipment grouting, which began on July 28, 2015, and was completed on July 30, 2015. In particular, DOE indicated that inspection ports 42 and 59 were being grouted during the visit and tour (ADAMS Accession No. ML15239A612).

While the equipment grouting tests performed by DOE focused primarily on the Advanced Design Mixer Pump (ADMP), these tests are also relevant to grouting equipment in Tanks 16H and 12H. The ADMP represents a bounding case for grouting internal tank fixtures because of its large size and complex flow path. Tank 16H has similar equipment, including a transfer jet, and a transfer pump (SRR-LWE-2014-00013). In the technical review report concerning Tank 18F and 19F grouting operations (ADAMS Accession No. ML13269A365), staff expressed concern that the ADMP mock-up test described in SRNL-STI-2011-00564 provided insufficient data to support a conclusion that the ADMP void spaces could be completely filled with the tested equipment grouts to eliminate a potential vertical flow path through Tank 18F. However, the scaled-up version of this mock-up test (SRR-CES-2012-00031) provided sufficient data to

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<sup>13</sup> NRC has not yet reviewed documentation to confirm that Grade 120 slag was used to grout the Tank 12H equipment.

support that conclusion because equipment grout flowability was shown to be maintained for the required time and the test showed that the equipment grout could fill and vent through the same 1-in (2.5-cm) diameter opening, successfully filling the mock-up equipment.

The Tank 16H final configuration report (SRR-CWDA-2015-00159) stated that the largest pieces of equipment in the tank (two rotary jet assemblies and a transfer pump) were filled with 80 percent of the estimated grout volume to 20 percent *more than* the estimated grout volume. The NRC staff will continue to monitor equipment grouting, equipment grout shrinkage, and testing of the recommended equipment grout fill formulation for future tanks.

### Cooling Coil Flushing and Grouting

Tanks 16H and 12H have 44 and 36 chromate water, carbon steel, 2-in (5-cm)-diameter cooling coils, respectively, inside their primary tanks (SRR-CWDA-2013-00091; SRR-LWE-2014-00147; SRR-CWDA-2015-00159). In Tank 16H, five coils that exhibited guillotine failure were flushed during cleaning, prior to grouting operations. Failed cooling coils were grouted after a 2-ft (0.6-m) layer of grout was placed in the primary to provide structural support (ADAMS Accession No. ML16167A237). This external level of tank grout was the minimum needed to provide support to vertical coils while maximizing the potential for guillotined or severed coils to vent during grouting (SRR-LWE-2014-00013; SRR-LWE-2014-00147). For Tank 16H, failed coil grouting occurred on June 10, 2015, after bulk fill had been placed in the primary over a three-day period (SRR-CWDA-2015-00170, Attachment 1). Failed coils were grouted from each inlet and outlet. Details about failed coil grouting operations for Tank 12H will be available for review in the Tank 12H Final Configuration report, when it is complete.

For intact cooling coils, triple rinsing of coils as part of tank closure was recommended in the Tanks 5F and 6F grouting lessons learned document (SRR-CWDA-2014-00015). However, the practice and recommendation were abandoned for Tanks 16H and 12H as DOE thought a single water flush was sufficient (ADAMS Accession No. ML16167A237). For Tanks 16H and 12H, intact cooling coils were flushed once in advance to remove chromate water before grouting operations began and again in conjunction with grouting the coils to minimize air entrainment (ADAMS Accession No. ML16167A237).

WSRC-STI-2008-00172 provides the cooling coil grout formulation (90 wt% Masterflow 816 and 10 wt% Grade 100 ground granulated blast furnace slag cement) used to grout cooling coils in Tank 16H. Masterflow 816 is marketed by BASF Corporation as a cement-based, aggregate-free, fluid, non-shrink, non-bleeding, high-strength cable grout with extended working time. For Tank 12H, DOE planned to use Grade 120 slag in place of Grade 100 slag to produce the cooling coil grout<sup>14</sup>. There are no requirements for the properties of cooling coil grout, so there were no quality control or test requirements associated with its production (SRR-LWE-2014-000150 and SRR-LWE-2015-00032). WSRC-STI-2008-00172 indicates that the cooling coil grout is required to have a reductive capacity at least as great as the tank grout, if not greater. Because the cooling coil grout formulation was selected before the formulation for tank grout, NRC staff verified that tank grout had a weight percent (wt%) of blast furnace slag cement (i.e., 6 wt%) that is less than that of cooling coil grout (7.5 wt%) (SRNL-STI-2011-00551).

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<sup>14</sup> NRC has not yet reviewed documentation confirming that Grade 120 slag was used to grout the Tank 12H cooling coils.

During the July 28–29, 2015 and February 2–3, 2016, onsite observation visits, DOE staff described Tank 16H and 12H cooling coil grouting operations (SRR-CWDA-2015-00095 and ADAMS Accession No. ML16111B174). WSRC-STI-2008-00298 recommended that DOE employ a mixing system that could blend the quantity of material required to fill one or more cooling coils. The total volumes of cooling coils range from 75 to 116 gal (284 to 439 L) (ADAMS Accession No. ML16111B174; SRR-CWDA-2015-00159). Therefore, 150 gal (568 L) batches of cooling coil grout (C-SPP-F-00057) were prepared. Cooling coil grout was mixed and placed by SRR Construction and controlled via work package (SRR-LWE-2014-000150 and SRR-LWE-2015-00032). Dry ingredients were premixed at a vendor facility and delivered to the site in a Super Sack® (BAG Corp, Richardson, Texas) (ADAMS Accession No. ML15239A612). SRR personnel then batched a fixed amount of water with the contents of the Super Sack and mixed it in a skid-mounted grout mixer. A hand pump was used to control pressure and flow to meter the grout into the cooling coils (ADAMS Accession No. ML15239A612). A totalizer at the flow meter provided the quantity of grout added to the coils in real time. WSRC-STI-2008-00298 called for filling intact cooling coils with water prior to grout placement to remove air, prevent air entrainment, and help ensure that a liquid-to-liquid interface is maintained during cooling coil grouting. Intact cooling coils were thus reflashed with water (disposed of in another waste tank) and then grouted from the coil inlet. When a solid stream of grout was visually detected at the coil outlet, a minimum surplus of 10 gal (38 L) of grout was introduced to the coil to ensure complete filling (ADAMS Accession No. ML15239A612; SRR-CWDA-2015-00159). DOE confirmed that the water-to-grout interface was maintained throughout the process of grouting Tank 16H cooling coils. The flushwater/grout interface volume was collected at the outlet in totes, solidified, and disposed of separately (ADAMS Accession No. ML15239A612; SRR-CWDA-2015-00159).

WSRC-STI-2008-00298 demonstrated that internally grouted piping surrounded by an insulating material underwent significant temperature rise during hydration. During the March 26–27, 2014, onsite observation visit, NRC staff asked DOE how it controlled the temperature of cooling coil grout (ADAMS Accession No. ML14106A573). NRC staff also raised the issue of the potential for cooling coil grout to boil during hydration due to the insulation provided by external tank grout in the previous technical review report on grouting operations (ADAMS Accession No. ML14342A784). During the July 28–29, 2015, onsite observation visit, DOE indicated that the mounded tank grout placed into Tank 16H was at that time only 1 to 2 ft (0.3 to 0.6 m) from the top of the tank (the primary was 94 percent complete), and that contractors were only then preparing to perform equipment and cooling coil grouting (ADAMS Accession No. ML15239A612). Thus, intact cooling coils were almost entirely encased in tank grout prior to being internally grouted with coil grout during the period from August 13–28, 2015 (SRR-CWDA-2015-00170, Attachment 1). At the May 17, 2016, teleconference, NRC expressed concern with the timing of intact cooling coil grouting, which was performed only after the primary had been nearly filled, because of the potential for coil grout to boil when insulated by external tank grout. DOE indicated that workers, who routinely perform jobs in valve houses related to installation, removal, and maintenance of grout hoses, have not observed high temperatures that would suggest excessive temperatures in the coils (ADAMS Accession No. ML16167A237). Workers who have monitored sensors that would indicate excessive heat production have seen no evidence of such when disconnecting hoses (ADAMS Accession No. ML16167A237). DOE should continue to consider heat transfer requirements such that cooling coil grout does not exceed its boiling temperature after placement into a highly insulated system that is also producing its own heat of hydration (WSRC-STI-2008-00298).

Following unsuccessful grouting of five intact cooling coils in Tanks 5F and 6F, DOE subsequently implemented changes to the coil grouting process (e.g., addition of screens to capture debris and use of a larger diameter line-cleaning device) to improve the chances of success during future grouting operations (ADAMS Accession No. ML14342A784). During the February 2–3, 2016, onsite observation visit and the May 17, 2016, teleconference, DOE discussed the results of Tank 16H cooling coil grouting (ADAMS Accession No. ML16111B174 and ADAMS Accession No. ML16167A237). Tank 16H had five cooling coils that exhibited guillotine failure, and each was successfully grouted from inlet and outlet. DOE indicated that one of these coils was fractured low (ADAMS Accession No. ML15239A612), suggesting that its outlet may have been encased in the stabilizing grout layer when it was grouted, and thus obscured. One failed (considered failed due to blockage) coil (Cooling Coil 12) was grouted from both the inlet and outlet and altogether took only 11 gal<sup>15</sup> (42 L) of grout (SRR-CWDA-2015-00159). Out of 39 intact cooling coils, DOE successfully grouted 37. Two other intact coils were not completely filled due to grouting delays that allowed some grout to harden inside the coils before they were completely filled (SRR-LWE-2015-00085). Cooling Coil 17 took 81 of 116 gal (307 of 439 L), so it was approximately 70 percent filled (SRR-CWDA-2015-00159). Cooling Coil 22 took 35 to 40 of 113 gal (132 to 151 of 428 L), so it was approximately 31 to 35 percent filled (SRR-CWDA-2015-00159). Likewise, details concerning Tank 12H cooling coil grouting will be available for review in the Tank 12H Final Configuration report when it is complete. NRC staff reviewed the reasons why grouting of three cooling coils in Tank 16H was unsuccessful, and will continue to monitor the steps that DOE takes in the future to prevent in-process grouting delays that enables premature hardening of grout in coils before they are fully filled. DOE should consider making a backup grout addition line readily available that can be used if needed. NRC staff will continue to monitor DOE's actions to prevent plugging of the cooling coil grout addition line.

Recommendations in the Tanks 5F and 6F lessons learned document (SRR-CWDA-2014-00015) that may reduce radiation doses to workers associated with the flushing and grouting of cooling coils included (i) minimization of the time between coil flushing and subsequent grouting operations, and (ii) planning ahead for high radiation/contamination potential by establishing temporary shielding and additional contamination controls. During the May 17, 2016, teleconference, DOE also indicated that for worker protection, it would be best to flush coils just once, immediately prior to grouting (ADAMS Accession No. ML16167A237).

NRC staff will continue to monitor cooling coil grouting, cooling coil grout shrinkage, how DOE minimizes air entrainment and controls the temperature of freshly placed grout under locally insulated conditions, and any future testing of the cooling coil grout formulation and operations that is undertaken.

### Final Configuration

The final configuration of Tank 16H and deviations from the closure module (SRR-CWDA-2013-00091), are described in SRR-CWDA-2015-00159, but uncertainties exist in the fill volumes reported. The actual reducing grout volume placed was within 97.7 percent of the estimate for Tank 16H. The volume of reducing grout placed in the annulus of Tank 16H was 1.5 percent greater than anticipated, while the volume of tank grout placed into the tank and

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<sup>15</sup> As a point of reference, the internal volume of the cooling coils is approximately 75 to 116 gallons (ADAMS Accession No. ML15239A612); SRR-CWDA-2015-00159.

annulus risers were 9 percent greater than anticipated. In the final configuration report, DOE indicated that the actual reducing grout volumes reported were based on the total number of grout batches (i.e., truckloads) discharged into the tank and annulus, assuming 8 cubic yards (6.1 cubic meters) per load of tank grout and 7 cubic yards (5.4 cubic meters) per load of clean cap grout (SRR-CWDA-2015-00159). Because DOE does not take into account the exact amount of each tank grout batch that is used for testing, the volume estimates are uncertain. DOE previously stated that riser volume estimates should not be considered highly accurate because these estimates are based on the total time it takes to completely discharge one truckload of tank grout and the time it takes to fill a riser (ADAMS Accession No. ML14106A573). With regard to equipment grouting, the final configuration report provides comparison of the theoretical fill volume and the actual volume of buckets used to deliver the equipment grout. ASTM C39 compressive strength testing indicated that the strength of the emplaced tank grout exceeds the compressive strength assumed in the HTF PA (SRR-CWDA-2010-00128); however, clean cap grout placed into Tank 16H was not tested. The average 28-day compressive strength of tank grout placed into Tank 16H was 2,788 psi (192 bars), well above the value of 2,000 psi (138 bars) described in the closure module (SRR-CWDA-2013-00091). To represent the potential strength of Tank 16H clean cap grout, which was not tested directly, DOE provided statistics obtained from 500+-day compressive strength measurements of four grout cylinders that were cast from Z-Area Vault 4 grout batches. These compressive strengths ranged from 5,560 to 6,880 psi (383 to 474 bars) with an average of 6,202 psi (428 bars), much greater than the nominal 2,000 psi (138 bars) assumed by the HTF PA (SRR-CWDA-2015-00159; SRR-CWDA-2010-00128).

Details concerning the Tank 12H final configuration will be reviewed by NRC staff when the Tank 12H Final Configuration report is complete. DOE should consider methods for improving grout volume estimates for the tanks, annuli, risers, and equipment to help ensure void space is fully grouted and better understand the nature of any remaining void space. DOE should consider archiving clean cap grout samples for 28-day compressive strength testing if it is used ever again to complete filling a waste tank.

#### Quality Assurance

The DOE quality assurance plan for Tank 16H, as described in SRR-LWE-2014-000150, is clear and, if implemented properly, should have ensured that Tank 16H was closed according to plan, while meeting all regulatory process and documentation requirements. The NRC staff will continue to evaluate whether the DOE quality assurance plan is being implemented effectively during future onsite observation visits and technical reviews.

#### Teleconference or Meeting

During a teleconference on May 17, 2016, DOE and NRC discussed NRC staff questions related to Tanks 16H and 12H grouting operations, including follow-up action items from the February 2016 onsite observation visit pertaining to grouting operations. Much of the meeting summary is discussed in the preceding evaluation. For additional details regarding the teleconference, please refer to the meeting summary (ADAMS Accession No. ML16167A237).

### **Follow-Up Actions**

NRC staff will continue to monitor DOE's bulk fill, equipment, and cooling coil grout formulations under Monitoring Factors 3.2 "Groundwater Conditioning via Reducing Grout," 3.3, "Shrinkage and Cracking," and 3.4, "Grout Performance" listed in NRC staff's Tank Farm Monitoring Plan (ADAMS Accession No. ML15238A761) while focusing on the technical issues listed in this technical review report and on any new technical issues that arise. A comprehensive list of follow-up action items which includes items prepared following the May 17, 2016, teleconference, as well as new items identified in completing this technical review report is found below in Attachment 3.

### **Open Issues**

No open issues resulted from this technical review. However, insufficient information is provided to address the likelihood for preferential flow pathways to form through grout monoliths due to shrinkage, cracking, and void space. NRC staff will continue to follow-up on this technical issue under 3.2 "Groundwater Conditioning via Reducing Grout," and Monitoring Factor 3.3, "Shrinkage and Cracking" (See ADAMS Accession No. ML15238A761).

### **Conclusions**

Due to the similarities in the grout formulation and approach to grouting Type I Tanks 5F, 6F, and 12H, Type II Tank 16H, and Type IV Tanks 18F and 19F, many of the conclusions resulting from the NRC staff's previous reviews of documentation related to Tanks 5F, 6F, 18F and 19F remain relevant to the review of Tanks 16H and 12H grouting operations. Relevant major and minor conclusions from the Tanks 5F, 6F, 18F and 19F reviews are repeated below along with new conclusions from the Tanks 16H and 12H reviews.

#### **Major Conclusions for Tanks 18F and 19F**

- The NRC staff concludes that performance requirements for grout formulations recommended and tested for Tanks 18F and 19F closure are generally consistent with bulk, initial chemical and hydraulic properties assumed in DOE's FTF Performance Assessment (SRS-REG-2007-00002, Rev. 1). However, the NRC staff also concludes that DOE has not provided sufficient information and testing to exclude preferential flow through the tank grout monolith from its reference case. Primarily, the NRC staff expects DOE to provide additional information related to the extent and performance impact of shrinkage to have reasonable assurance that the performance objectives specified in Subpart C of Part 61 of Title 10 of the Code of Federal Regulations (10 CFR Part 61, Subpart C) will be met.
- Further, during the review of tank grouting video, NRC staff has observed potential segregation of tank grout that could enhance the extent of shrinkage along the periphery of the Type IV tanks (i.e., along the tank walls). The NRC staff will continue to evaluate the potential for shrinkage and cracking induced preferential flow through the tank grout under Monitoring Factor 3.3, "Shrinkage and Cracking" (See ADAMS Accession No. ML12212A192). NRC also continues to monitor the potential for segregation of emplaced grout and its impacts on flow through the grout monolith and waste release under Monitoring Factor 3.4, "Grout Performance".

### Minor Conclusions for Tanks 18F and 19F

The NRC staff will also continue to monitor void volumes in the emplaced grout to the extent information is available (Monitoring Factor 3.4, "Grout Performance"), the importance of alkali-silica reactivity on cementitious material degradation (Monitoring Factor 3.3, "Shrinkage and Cracking") and the impact of limestone additions to the grout mix on pH buffering of water contacting the emplaced grout (Monitoring Factor 3.4, "Grout Performance"). NRC staff also expects DOE to provide additional information on the potential for thermal cracking of the grout monolith for Tanks 18F and 19F.

### Major Conclusions for Tanks 5F and 6F

Major and minor conclusions from the Technical Review Report for Tanks 18F and 19F grouting were repeated in the Tanks 5F and 6F Technical Review Report. Additional conclusions (or additional detail regarding a previous conclusion) were also listed.

### Additional Conclusions for Tanks 5F and 6F

- NRC staff observed grout with higher flowability in the Tanks 5F and 6F grouting operation videos compared to that placed into Tanks 18F and 19F due to the higher slump specified for use in tanks with cooling coils.
- NRC staff observed via video potential instances of bleed water segregation (e.g., mottling of grout that may be due to incomplete mixing or segregation, bright watery sheen at the leading edge of the fresh grout flow lobe, strong color differentials). While NRC staff acknowledges the potential for these observations to be due to the Slick Willie pump priming agent, chromated water, or due to shadows caused by lighting angles, making that determination is subjective and the priming agent or water may have a potential impact on hydraulic properties and grout quality.
- DOE should minimize or eliminate excess water introduction to waste tanks, and provide evidence that introduction of excess water (e.g., in the form of Slick Willie) into Tanks 5F and 6F (and 18F and 19F) did not reduce the integrity of the tank grout to less than what is assumed in the FTF PA (SRS-REG-2007-00002, Revision 1).
- DOE should take reasonable measures to prevent future placement of out-of-specification grout because inhomogeneity in the grout will affect flow in the monolith due to higher permeability zones receiving higher flow rates relative to surrounding zones.
- DOE should consider giving higher priority to development and testing of a shrinkage compensating tank grout formulation.
- Given that only approximately 50 percent of the tank annuli were visible in videos documenting annulus grouting, DOE should consider placing video cameras in all riser locations within tank annuli during grouting operations or else occasionally reposition video cameras into different available risers to improve visibility.

- Two of the failed cooling coils were only partially filled because DOE had not adequately cleaned the line prior to the fill, which allowed grout residue to plug the line. NRC staff notes that the lessons learned report (SRR-CWDA-2014-00015) provides several suggestions to prevent cooling coil grout addition line pluggage (e.g. increasing flush frequency, increasing flush water velocity, installing screens to prevent solids from plugging the line, increasing the pig diameter, and pre-charging the line with water). NRC staff will continue to monitor DOE's actions to prevent plugging of the cooling coil grout addition line.
- Field-collected temperature data from actual waste tanks would provide valuable information regarding grout integrity given the potential for thermal cracking of large, hydrating grout monoliths.

### Conclusions for Tanks 16H and 12H

- DOE should take reasonable measures to ensure a sufficient number of cement trucks are in rotation to optimize grout distribution throughout the tank and minimize mounding.
- DOE should take measures to continuously fill cooling coils with grout to ensure complete filling and to avoid creating grout blockages within intact coils that could have otherwise been fully filled (SRR-CWDA-2015-00159). Complete filling of cooling coils is needed to eliminate in-tank void space and preferential flow paths. DOE should continue to document related lessons learned and implement a path forward that will mitigate future occurrences.
- DOE should consider heat transfer requirements such that highly insulated cooling coil grout (i.e., in coils surrounded externally by tank grout) does not exceed its boiling temperature shortly after placement (WSRC-STI-2008-00298).
- NRC staff observed via Tank 16H grouting video instances of bleed water segregation. Non-uniformly distributed excess water in the tank and annulus may have a potential impact on hydraulic properties and grout quality. DOE should remove excess ponded water from the tank before, during, and near the end of grouting operations, whenever aqueous ponds are present, to ensure adequate quality grout is placed into tanks and annuli. Alternatively, DOE could provide additional information to support a determination that the quantities of water present in the tanks during grouting do not adversely impact grout performance.
- More flowable clean cap grout used to fill remaining void space at the top of the Tank 16H primary and annulus may have significantly different hydraulic properties compared to the rest of the bulk fill grout placed in the primary and annulus of Tank 16H. DOE should address the potential for either a capillary or permeability barrier to form due to the varying hydraulic conductivity of the clean cap and bulk fill grout used in Tank 16H.
- The results of the grout drop test report (RPT-5539-EG-0016) suggest the potential for segregation and bleed water production in the annulus of Tank 12H during initial grouting operations if grout was dropped into standing water. DOE should provide additional information regarding the quantity and performance impact of the presence standing water in Tank 12H during grouting.
- Lehigh Grade 120 slag used in the Tank 12H grout mix starting on the second day of grouting is expected to provide superior chemical performance compared to Holcim Grade 100 slag due to the higher activity index and increased reduction capacity. The compressive strength of the Grade 120 slag is also expected to increase due to a combination of small particle size and increased reactivity. DOE should evaluate differences in hydraulic conductivity between the Grade 100 and Grade 120 slag used to fill Tank 12H and any resulting performance impact.

## Appendix A

### Review Status of General Grout Documents:

C-SPP-F-00055. Ganguly, A. "Furnishing and Delivery of Tank Closure Grout." Revision 4. Aiken, South Carolina: Savannah River Remediation, LLC. December 20, 2012.

This document provided procurement specifications for furnishing and delivery of a low-paste, No. 8 stone (LP#8) reducing tank grout (see Attachment 5.5). This specification called (i) for use of slag cement that met the requirements of ASTM C989 Grade 100, and (ii) for its ASTM C1611 slump flow to be in the range of 26–30 in (66–76 cm). NRC staff reviewed Revision 2 and Revision 4 of this document in the technical review reports "U.S. DOE Documentation Related to Tanks 18 and 19 Final Configurations with an Emphasis on Grouting from Recommendations and Testing, to Final Specifications and Procedures" (ADAMS Accession No. ML13269A365) and "U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures" (ADAMS Accession No. ML14342A784). The reader is referred to NRC staff's prior technical reviews for summaries of the reference. Based on the outcome of supplier deviation disposition request SDDR No. 13182, it is anticipated that a future Revision 5 of this procurement specification will implement the following slag-related changes:

- a) REPLACE "Grade 100" with "Grade 100 or Grade 120" at the end of Sections 3.2.3.3.A, 3.2.3.3.B and 3.2.3.3.C (SDDR No. 13182).
- b) ADD this sentence to the end of Section 3.2.3.3.A: "The recommended material sources for the Grade 120 Slag Cement shall be Lehigh Grade 120 (preferred) or Lafarge Grade 120 (optional)" (SDDR No. 13182).
- c) REPLACE Slag Cement Material Grade "100" by "100/120" in the Cementitious Material column of Attachment 5.5 (SDDR No. 13182).

C-SPP-F-00057. McCord, J.B. "Furnishing and Delivery of Cooling Coil Grout Dry Feeds." Revision 2. Aiken, South Carolina: Savannah River Remediation, LLC. July 2014.

This document provides the procurement specifications for furnishing and delivery of cooling coil grout dry feeds, which include components Masterflow 816<sup>®</sup> grout and ASTM C989 *Grade 100* slag cement provided either individually or together in a 9:1 ratio inside of SuperSaks designed to minimize moisture intrusion. Batched quantities may vary within  $\pm 2$  percent of 1709.25 lbs (775 kg) for Masterflow 816 and 190 lbs (86 kg) for slag cement. The specification calls for documentation that demonstrates the grout components meet specification requirements. Any material substitutions relative to this specification, such as use of Grade 120 slag cement in lieu of Grade 100, would necessitate a Supplier Deviation Disposition Request (SDDR) and approval of SRR, as would any change in material supplier.

RPT-5539-EG-0016 (SRRA051386-2-A). Diener, G. "Savannah River Remediation Tank Closure Grout Assessment Final Report (Grout Drop Test Report)." Revision 0. Barnwell, South Carolina: EnergySolutions. December 16, 2014.

To assess potential changes to grouting operations being considered by SRR, this grout drop test report documented intermediate-scale field experiments conducted to examine physical

effects of discharging grout via three different scenarios. The field experiments discharged 7 cubic yards (i.e., one truckload) of SRS tank grout [26–30 in (66–76 cm) slump] into three 20-ft (6-m)-diameter molds via each of these scenarios: (i) from a 42 ft (12.8 m) drop height using a tremie with free end positioned 5 ft (1.5 m) above an unyielding surface, (ii) from a 42 ft (12.8 m) drop height using a tremie with free end positioned approximately 5 ft (1.5 m) above a 4-in (10-cm)-deep pool of water, and (iii) from a 42 ft (12.8 m) drop height without a tremie, allowing tank grout to freefall onto an unyielding surface. Note that a tremie is a long hose connected to the grout distribution pipe that is used by DOE to reduce the drop height of grout into the tank. The first two tests used two 37-ft (11.3-m)-long tremies equivalent to those used for tank closure operations. Each experiment was visually recorded by video camera, which were provided to NRC staff for review.

The baseline for tank grout discharge (represented by Test 1) is placement into a tank or annulus via a tremie with free end located within 5 ft (1.5 m) above a dry tank floor, dry grout surface or fresh grout surface. Test 2, which discharged grout into standing water via a tremie, represented non-removal of cooling coil flushwater prior to grouting operations. Tests 3A and 3B, which discharged grout from a 42-ft (12.8-m) drop height without a tremie were of potential interest as a worker dose-limiting option. A fourth test that would have examined physical effects of allowing tank grout to freefall from a drop height of 42 ft (12.8 m) into a 4-in (10-cm)-deep pool of water was not undertaken due to unanticipated failure of a containment mold during the attempted completion of the first grout freefall test (Test 3A).

Grout discharge rates were not constant during the grout drop tests and were inconsistent with the intended discharge range of 1.17 to 1.23 cubic yards per minute. Grout was discharged most rapidly during baseline Test 1 (1.52 cubic yards per minute), most slowly during Test 2 (0.80 cubic yards per minute), and at intermediate rates during Tests 3A and 3B (0.88 and 1.27 cubic yards per minute).

The report indicates that there was no visual evidence of bleed water segregation during Test 1 grout placement; however, the report goes on to provide photographs of cylindrical samples taken from the grout monolith and indicated that these photographs showed a slight decrease in aggregate at the top of T1-9-O-1 (the Test 1 sample collected at the 9 foot radius position). During Test 2, the authors report ample visual evidence of bleed water segregation during grout placement, as well as later based on analysis of photographs of cylindrical samples and optical microscopy assessments of aggregate distribution. During Tests 3A and 3B, the grout stream leaving the nozzle was described as dispersing into small clumps of grout as it fell through the air with heavy splattering against the containment walls after impacting the steel plate<sup>16</sup>. The authors of the report indicated that splattering and splashing were significantly reduced after the grout depth was sufficient to adsorb the impact of the falling grout. The report authors stated that they observed no visual evidence of bleed water segregation during Tests 3A/3B and optical microscopy samples were consistent with this observation.

The physical effects of the grout drop test scenarios were evaluated on core-drilled grout samples, recovered after a minimum of 3 weeks of set time, by conducting measurements of

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<sup>16</sup> Four circular containment areas were prepared by placing an 8 foot x 10 foot steel plate (1" thick) on the ground which was intended to represent the unyielding surface of the tank bottom. A layer of sand was placed around the steel plates to form a level base so that a 20 foot diameter above ground pool could be erected on top of the steel plate.

their compressive strength, saturated hydraulic conductivity, and aggregate distribution after a 28-day or longer set time. Grout cylinders were removed from locations 1, 5, and 9 ft (0.3, 1.5, and 2.7 m) from the center of each monolith to ascertain if grout properties had radial dependencies. Compressive strength exhibited radial dependencies at 28 days for Tests 1 and 2. Test 2, had the lowest compressive strengths after 28 days. In general, compressive strength increased with curing time for Tests 1 and 3 (compressive strength was only measured after a 28 day curing time for Test 2). For Test 1, five of six grout samples tested after a 90-day set time met the compressive strength PA requirement of 2000 psi (138 bars); the other was thought to have failed prematurely due to having a chipped surface. When cylinders from each monolith were tested for hydraulic conductivity at 7 weeks (i.e., ~49 days) post-placement, saturated hydraulic conductivity increased with radius. Test 3B exhibited the highest hydraulic conductivities at every radius. Test 2, which exhibited more significant bleed water segregation compared to other tests, exhibited the lowest hydraulic conductivities at every radius, yet exhibited the largest relative increase in the property between radii of 5 and 9 ft (1.5 and 2.7 m). The authors indicated that calcium-silicate-hydrate formation during continued hydration in high relative humidity was responsible for healing of microcracks that were thought to have been caused due to drilling of the specimens. CSH formation allowed for improved compressive strength and saturated hydraulic conductivity with additional curing time. Test 3B yielded saturated hydraulic conductivities with less radial dependency when measured 18 weeks (i.e., ~126 days) post-placement and by that time, conductivity was decreasing with increasing radius. Optical microscopy samples extracted from Test 1 and 3B monoliths generally indicated uniform distribution of aggregate throughout each sample, except for a slight decrease in aggregate observed at the top of sample T1-9-O-1, collected at a 9 ft (2.7 m) radius. In contrast, core samples T2-5-O-1 and T2-9-O-1 from the Test 2 monolith exhibited distinct segregation of aggregate below from fines above with increasing radius from the central drop point. Approximately the top 2.4 in (6 cm) of material comprising sample T2-9-O-1 was generally devoid of pea gravel and enriched in fines.

The authors concluded that (i) discharging tank grout without a tremie would not impact the properties of the cured grout because both compressive strength and saturated hydraulic conductivity likely would be within the grout specification (e.g., SRNL-RP-2011-00977) and segregation would be unlikely, but that (ii) discharging tank grout into 4 in (10 cm) of ponded water is not recommended because significant segregation would occur and the grout would likely not meet the grout specification requirements for compressive strength. They allowed that shallower ponded water might yield less bleed water segregation and that additional testing would be necessary to support such a finding.

SDDR No. 13182. "Section 3.2.3.3 States the Slag Cement Must Meet ASTM C989, Grade 100. This Slag is No Longer Available in the Southeast, U.S.A. Change Specification to Allow for the Use of ASTM 989, Grade 120 Slag. (Supplier Deviation Disposition Request)." Augusta, Georgia: Argos Ready Mix, LLC. March 30, 2015.

This supplier deviation disposition request (SDDR) documented DOE's disposition of a proposed deviation to the tank grout procurement specification (C-SPP-F-00055, Revision 4) due to a limited supply of Grade 100 ground granulated blast furnace slag cement. Dispositions of SDDRs must be handled as described in procurement specifications. This SDDR specifically requested a deviation that would allow use of Lehigh Hanson (Cape Canaveral) Grade 120 slag cement, or Grade 120 slag from another source in the southeastern United States. The request occurred because production of Holcim, Inc. (Birmingham, Alabama) Grade 100 slag, previously

used at SRS as a component in saltstone, tank grout, and clean cap grout, had ceased (VSL-15R3740-1). Argos, who prepared the SDDR, provided 12 months of Lehigh Hanson's Grade 120 slag ASTM C989 test reports to support the request. SRR agreed to the deviation request, allowing use of LeHigh Hanson Grade 120 slag to replace Holcim, Inc. Grade 100. A future Revision 5 of C-SPP-F-00055 is expected to formally include related changes to the specification for slag cement. Vitreous State Laboratory recommended LeHigh Hanson Grade 120 as the first choice, and Lafarge Grade 120 slag as the second-best choice amongst slag materials tested (VSL-15R3740-1).

SRNL-STI-2012-00546. Cozzi, A.D. and B.R. Pickenheim. "Impact of Standing Bleed Water on Saltstone Placement." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. September 2012a.

This report was the first of two that documented a study of the effects of excess water, during the early hydration process, on physicochemical properties of reducing grout. The presence or absence of aqueous environments during the hydration and hardening of reducing grout may impact properties important to waste isolation. For example, grout that hydrates and hardens in the absence of standing water may undergo local surface oxidation, resulting in increased leachability of redox-sensitive species. In contrast, while standing water may be available for potential incorporation into subsequent grout placements, excess water may degrade grout properties such as homogeneity by exacerbating the effects of bleed water segregation. Eight grout types studied by Cozzi and Pickenheim (2012a,b) were variations on waste-isolating saltstone representing different conditions that may occur during processing or after it has been placed and begins hardening. Two water-to-premix ratios (0.60 and 0.64) were tested. Two grout types were fully exposed to ambient air after 7 days, and the other six were kept in humid environments inside sealed molds. Five of the grouts were poured into empty molds, with one being capped with standing water post-gel; the other three were poured into molds that contained salt solution (i.e., "standing water"). After 28 days, grout specimens representing the eight types were de-molded and leached according to the ANSI/ANS 16.1 standard test. The two grout types exposed to ambient air during hydration exhibited the greatest tendency to leach incorporated constituents, as evidenced by having both the highest leachate electrical conductivity values and the lowest Leachability Indices for potassium, sodium, rhenium, nitrite, and nitrate. The leachability of grout placed in standing water, however, was unaffected. Duplicate grout specimens representing the eight types were de-molded, sliced into four disks, and the density of each disk was analyzed to understand the effects of bleed water segregation on grout homogeneity. Two grout types poured into 10 percent excess salt solution exhibited a 4 to 5 percent increase in density from top to bottom of each specimen, which is indicative of bleed water segregation and settling. The authors concluded that premature drying of saltstone was detrimental to its quality with respect to isolating waste, but that the presence of excess water may exacerbate settling that causes nonhomogeneity.

SRNL-STI-2012-00576. Cozzi, A.D. and B.R. Pickenheim. "Impact of Standing Water on Saltstone Placement II - Hydraulic Conductivity Data." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. October 2012b.

This report was the second of two that documented a study of the effects of excess water, during the early hydration process, on physicochemical properties of reducing grout. The presence or absence of aqueous environments during the hydration and hardening of reducing grout may impact properties important to waste isolation. A relatively dry environment

may be favorable for shrinkage crack formation that would increase hydraulic conductivity, but excess standing water may also result in development of heterogeneous distributions of density and hydraulic conductivity. Eight grout types studied by Cozzi and Pickenheim (2012a,b) were variations on waste-isolating saltstone representing different conditions that may occur during processing or after it has been placed and begins hardening. Two water-to-premix ratios (0.60 and 0.64) were tested. Two grout types were fully exposed to ambient air after 7 days, and the other six were kept in humid environments inside sealed molds. Five of the grouts were poured into empty molds, with one being capped with standing water post-gel; the other three were poured into molds that contained salt solution (i.e., “standing water”). After 28 days, each specimen was sliced in half, and the saturated hydraulic conductivity of each half was measured to determine if the previously observed density gradients (SRNL-STI-2012-00546) could be correlated with hydraulic conductivity gradients. The two grout types exposed to air during hydration had hydraulic conductivities that were three orders of magnitude greater than those of specimens hydrated in a humid environment, perhaps due to development of microcracks. Among the other six grout types, neither the water-to-premix ratio nor excess standing water had appreciable effects on hydraulic conductivity. However, seven of the eight grout types exhibited density-related depth-dependencies of hydraulic conductivity, with higher densities being correlated to lower hydraulic conductivities. However, the authors concluded that although a depth-dependent effect on hydraulic conductivity was detectable, grout quality was not meaningfully degraded by bleedwater segregation and settling given the natural variation in hydraulic conductivity that is expected within saltstone from the influence of variable process conditions and environmental conditions that may occur. Moreover, they concluded that hydration and hardening of reducing grout in a moist environment is critical to development of high-quality properties, such as the PA-assumed low values of hydraulic conductivity.

SRNL-STI-2012-00578. Langton, C.A., et al. “Relationship Between Flowability and Tank Closure Grout Quality.” Revision 0. Aiken, South Carolina: Savannah River National Laboratory. October 2012.

This report evaluated the relationship between tank grout flowability and compressive strength to determine whether the maximum acceptable slump for tank grout could be increased from 28 to 30 in (71 to 76 cm) to improve grout flowability and decrease mounding without negatively impacting grout quality. Langton et al. concluded that the compressive strengths of Tanks 18-F and 19-F quality control test specimens were not a function of slump over the range tested [i.e., from 24 to 28 in (61 to 71 cm)] and that the upper limit for slump measured per ASTM C1611 could be increased by an additional 2 in (5 cm) without negatively affecting grout quality. The authors also recommended that the grout delivery rate be increased by (i) using a higher capacity grout pump and (ii) through more continuous discharge to improve grout distribution throughout tanks, particularly tanks containing significant obstructions such as cooling coils. While this report discussed a potential increase in the grout drop height from  $\leq 5$  ft ( $\leq 1.5$  m) to  $\leq 10$  ft ( $\leq 3$  m) intended to impart greater kinetic energy to the grout and improve its in-tank distribution, the authors indicated that additional testing was necessary to determine whether bleed water segregation, which would negatively impact grout quality, would be enhanced if the grout drop height was increased from 5 to 10 ft (1.5 to 3 m).

USQ-HTF-2015-00300. Voegtlen, R.O. "Supplier Deviation Disposition Request (SDDR) Number 13182–Deviation from Specification C-SPP-F-00055, Revision 4 (Technical Review Package)." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. September 10, 2015.

This technical review package contains a series of related reports, including a Design Authority Technical Review (DATR) (originated April 17, 2015 and signed September 2, 2015), an Unreviewed Safety Question Review (USQS) report (originated April 17, 2015 and signed September 2, 2015), a Consolidated Hazard Analysis Process (CHAP) Screening (originated April 17, 2015 and signed September 10, 2015), a description of the proposed action in an attachment, and a UWMQ Determination. Specification C-SPP-F-00055, Revision 4, Section 3.2.3.3.A requires that ground granulated blast furnace slag cement used to batch tank grout meet the requirements of ASTM C989, Grade 100. SDDR No. 13182, initiated in March 2015, proposed a deviation to allow use of Grade 120 slag cement in lieu of Grade 100 (i.e., the "proposed action"). Because the deviation or proposed action would alter the tank grout formulation, the UWMQ Determination was initiated and an UWMQ Evaluation (UWMQE) was performed. UWMQE-SRR-CWDA-2015-00088, "Use of Grade 120 Slag in Tank Closure Grout (USQ-HTF-2015-00300)" was approved and the deviation was determined acceptable because its implementation would have no adverse impact on the facility or its systems, nor would it compromise PA conclusions.

VSL-15R3740-1. Gong, W. et al. "Investigation of Alternate Ground Granulated Blast Furnace Slag for the Saltstone Facility (Final Report)." Revision 0. Washington, DC: Vitreous State Laboratory, The Catholic University of America. August 26, 2015.

This report describes testing performed for SRR to assess characteristics of grout specimens prepared using alternative slag cements. Granulometry, reductive capacity, viscosity, yield stress, temporal gelation behavior (i.e., "gel time" at which point a grout slurry is no longer pourable), and heat of hydration (through the first 12 days post-placement) test results were obtained from alternative slag cements and from grout specimens prepared with alternative slag cements, including: Holcim, Inc. Grade 100 (control); Lehigh Hanson Grade 120; Lafarge Grade 120; Argos Grade 120; and Essroc Grade 100 (Essroc Grade 100 is misclassified because it meets Grade 120 requirements of ASTM C989). In general, substituting a Grade 120 slag for Grade 100 should result in higher compressive strength and lower hydraulic conductivity specimens due to the smaller particle size and enhanced reactivity of Grade 120. Differences in slag chemistry may also impact fresh and hardened properties of cementitious grouts, which could affect long-term performance. Salt waste simulant was combined with dry premixes at a water-to-premix ratio of 0.6 typical of saltstone, rather than at a ratio of 0.5 typical of clean cap grout (such as that placed into Tank 16H). Based upon heat release, slag reactivity of the five alternatives ranked as follows: Holcim (lowest) < Lehigh Hanson < Argos < Lafarge < Essroc (highest). Argos and Essroc slags were not recommended for use due to (i) Argos offering a substantially lower reductive capacity than the other materials and (ii) Essroc exhibiting the greatest heat release. Lehigh Hanson was preferred due to its (i) high reductive capacity, which was 12 percent greater than Holcim, and (ii) low heat release. Lafarge was considered the next-best alternative.

WSRC-STI-2008-00172. Harbour, J.R., et. al. "Closure of HLW Tanks—Formulation for a Cooling Coil Grout." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. April 2008.

The document summarized the development of the cooling coil grout formulation [90 wt% Masterflow 816 (BASF Corporation, Shakopee, Minnesota) and 10 wt% ground granulated blast furnace slag, Grade 100] and recommended a laboratory-scale investigation be performed to determine the impact of operational variations such as temperature and mixing time on cooling coil grout properties. NRC staff reviewed Revision 0 of this document in the technical review report "U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures" (ADAMS Accession No. ML14342A784). The reader is referred to NRC staff's prior technical review for a summary of the reference.

WSRC-STI-2008-00298. Hansen, E.K. et al. "Closure of HLW Tanks—Phase 2, Full Scale Cooling Coils Grout Fill Demonstrations." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. June 2008.

The document described full-scale testing of the recommended cooling coil grout formulation. NRC staff reviewed this document in the technical review report "U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures" (ADAMS Accession No. ML14342A784). The reader is referred to NRC staff's prior technical review for a summary of the reference.

#### **Review Status of Tank 18F and 19F Documents:**

SRNL-STI-2011-00551. Stefanko, D.B. and C.A. Langton. "Tanks 18 and 19-F Structural Flowable Grout Fill Material Evaluation and Recommendations." Revision 1. Aiken, South Carolina: Savannah River National Laboratory. April 2013.

This report summarized the development of a zero bleed tank grout formulation and testing of specimens. The NRC staff reviewed Revision 0 of this document in the technical review report "U.S. DOE Documentation Related to Tanks 18 and 19 Final Configurations with an Emphasis on Grouting from Recommendations and Testing, to Final Specifications and Procedures" (ADAMS Accession No. ML13269A365). Later, staff also reviewed Revision 1 of this document in the technical review report "U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures" (ADAMS Accession No. ML14342A784). In Revision 1, DOE corrected a value in Table 3-6 {"Moisture retention as a function of applied pressure for [grout formulas] LP#8-016 and LP#8-020"} that followed a correction in a contractor test report. At a March 26–27, 2014 onsite observation visit, DOE staff indicated that moisture retention data, which had been forecast by this report to be obtained in the near future over the high-pressure range from 15 to 45 bars, were never obtained due to inadequate funding levels (ADAMS Accession No. ML14342A784). Revisions 0 and 1 are identical in all other respects and the reader should refer to NRC staff's Revision 0 technical review for a summary of the reference.

SRNL-STI-2011-00564. Stefanko, D.B. and C.A. Langton. "Tank 18 and 19-F Tier 1A Equipment Fill Mock Up Test Summary." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. September 2011.

This report summarized the results of equipment fill mock up testing, which was focused on internally grouting the Advanced Design Mixer Pump and pipes of 1-in (2.5-cm)-diameter and greater. NRC staff reviewed this document in the technical review report "U.S. DOE Documentation Related to Tanks 18 and 19 Final Configurations with an Emphasis on Grouting from Recommendations and Testing, to Final Specifications and Procedures" (ADAMS Accession No. ML13269A365). The reader is referred to NRC staff's prior technical review for a summary of the reference.

SRNL-STI-2011-00592. Stefanko, D.B. and C.A. Langton. "Tanks 18 and 19-F Equipment Grout Fill Material Evaluation and Recommendations." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. November 2011.

This report documents laboratory testing performed to identify an equipment grout formulation. NRC staff reviewed this document in the technical review report "U.S. DOE Documentation Related to Tanks 18 and 19 Final Configurations with an Emphasis on Grouting from Recommendations and Testing, to Final Specifications and Procedures" (ADAMS Accession No. ML13269A365). The reader is referred to NRC staff's prior technical review for a summary of the reference.

SRNL-STI-2011-00749. "Tank 18F and 19F Tank Fill Grout Scale Up Test Summary." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. December 2011.

This report documents a bulk fill scale-up test of the tank grout formulation, which was intended to demonstrate proportioning, mixing, and transportation of material produced in a full-scale ready-mix concrete batch plant. In addition, the tank grout was characterized with respect to fresh properties, thermal properties, and compressive strength as a function of set time. NRC staff reviewed this document in the technical review report "U.S. DOE Documentation Related to Tanks 18 and 19 Final Configurations with an Emphasis on Grouting from Recommendations and Testing, to Final Specifications and Procedures" (ADAMS Accession No. ML13269A365). The reader is referred to NRC staff's prior technical review for a summary of the reference.

SRR-CES-2012-00031. "Summary Report of the Equipment Grout Mock-up Test." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. April 13, 2012.

This report summarized results obtained from a scaled-up equipment grout mock-up test (SRNL-STI-2011-00564). NRC staff reviewed this document in the technical review report "U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures" (ADAMS Accession No. ML14342A784). The reader is referred to NRC staff's prior technical review for a summary of the reference.

### **Review Status of Tank 5F and 6F Documents:**

2013-NCR-15-WFC-0006. "F Tank Farm Grout - Tank 6 (Non-Conformance Report)." Aiken, South Carolina: Savannah River Remediation. October 2, 2014;  
USQ-FTF-2013-00317. "New Data – Use-As-Is Disposition of the Non-Conformance Report (NCR) 2013 NCR-15-WFC-0006 'F Tank Farm Grout – Tank 6.' Non-Conformance Tank 6 Grout Water Content Higher than Allowed per C-SPP-F-00055, Rev. 4 'Furnishing and Delivery of Tank Closure Grout.'" Revision 0. Aiken, South Carolina: Savannah River Remediation. October 3, 2014; and tank grout batch tickets for September 19, 2013.

Altogether, these records documented that 25 batches of tank grout placed into Tank 6 on September 19, 2013, had water content approximately 3 percent greater than allowed by the procurement specification "Furnishing and Delivery of Tank Closure Grout" (C-SPP-F-00055). NRC staff reviewed the non-conformance report (2013-NCR-15-WFC-0006), waste management question evaluation (USQ-FTF-2013-00317), and associated tank grout batch tickets in the technical review report "U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures" (ADAMS Accession No. ML14342A784). The reader is referred to NRC staff's prior technical review for a summary and evaluation of the references.

SRR-CWDA-2014-00015. Cantrell, J.R. "Tank 5 and 6 Grouting Project Lessons Learned." Revision 0. Aiken, South Carolina: Savannah River Remediation. February 6, 2014.

NRC staff reviewed DOE's lessons learned from Tank 5F and 6F grouting operations in the technical review report "U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures" (ADAMS Accession No. ML14342A784). Lessons learned during grouting operations at Tanks 5F and 6F resulted in the following recommendations: (i) to minimize tremie adjustment and radiation exposure, evaluate effects of increasing the grout drop height; (ii) work with camera crew to ensure satisfactory camera placement; (iii) use less Slick Willie during the final lifts to minimize potential for unincorporated liquid; (iv) prevent cooling coil grout addition line pluggage through increasing flush frequency, increasing flush water velocity, installing screens to prevent solids from plugging the line, increasing the pig diameter, and pre-charging the line with water. To minimize unintended use of out-of-specification grout, the report recommended refresher training for personnel reviewing batch tickets to aid identification of nonconforming grout and to modify procedures to include a specific review of formulation values at delivery.

SRR-LWE-2013-00214. Chandler, T.L. "Engineering Path Forward – Tanks 5 & 6: Record of Additional Grouting Actions. Revision 0. Aiken, South Carolina: Savannah River Remediation. February 10, 2014.

To decrease the likelihood of discovering unexpected conditions during future grouting efforts through identification of potential causes, this report documented several situations that occurred during the process of completing operational closure of Tanks 5F and 6F. The report outlined (i) events that occurred, (ii) actions taken or planned to be taken to properly manage such events, and (iii) the reasoning behind decision(s) made. The situations enumerated were:

1. Tank 6, Riser 4 transfer jet not disassembled as planned; upon discovery, decision was made to grout the equipment to eliminate a potential vertical fast flow path.
2. Near completion of tank grouting, displaced liquid was found in several of the tank risers of both tanks; upon discovery, a new work order (Work Order No. 01199254-65) was issued to allow, if required, the liquid to be removed. The liquid levels present were estimated at less than a few inches to more than 12 inches (cm) within the risers. The report stated, however, that “as a result of liquid reconstitution during grout addition and evaporation, the liquid level was deemed negligible and its removal unnecessary.”
3. Between the two tanks, five of 72 cooling coils could not be fully grouted, and two other coils did not receive the intended 132 L (35 gal) of excess grout required to transition them from being filled with flushwater to filled with grout. Reasons for incomplete filling included blockage of grout addition lines, high radiation levels during operations, blockage of perforated (slightly damaged) cooling coils with intruded tank grout, and inadequate coil grout batch size. In response, actions were taken to minimize the likelihood of line blockages with hardened grout and two batches of coil grout were prepared in advance of each subsequent coil grouting operation. It was decided that these partially filled coils did not represent a change to the assumed waste tank final equipment configuration. The FTF PA and supporting documentation (e.g., SRR-CWDA-2012-00051) assumed that coils would be filled only to the extent practical and recognized the potential for coils to be partially filled.

Work Order No. 01199254-65. “Tanks 5–6 Pump Standing Water in Risers.” Revision 0. Aiken, South Carolina: Savannah River National Laboratory. December 10, 2013.

This work order instructed workers on use of a pump to remove excess liquid from Tank 5F and 6F risers near the end of grouting activities, if necessary. NRC staff reviewed this work order in technical review report: “U.S. DOE Documentation Related to Tanks 5F and 6F Final Configurations with an Emphasis on Grouting from Recommendations and Testing to Final Specifications and Procedures” (ADAMS Accession No. ML14342A784). The reader is referred to NRC staff’s prior technical review for a summary and evaluation of the reference.

#### **Review Summaries of Tank 16H Documents:**

C-SPP-Z-00012. Patel, R. “Vault 4 Clean Cap Grout (Procurement Specification).” Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. March 20, 2014.

This document provided procurement specifications for furnishing and delivery of three alternative mix designs for clean cap grout (see Attachment 5.5 for formulations, all of which have dry feed 45:45:10 flyash:slag:cement ratios), only one of which was selected for use in Saltstone Disposal Units. The specification called for a measured slump flow (ASTM C1611) within the working range of 26–38 in (66–97 cm) as a field test acceptance criterion for clean cap grout. Other procurement specifications included trial batching to demonstrate the production grout would meet specification requirements, plant and delivery schedule capacity, mix time, drum revolution, and grout temperature limits at point of delivery, provision of batch tickets, inspection reports, and test results containing specified data, as well as quality assurance, record-keeping, and testing laboratory requirements. Collection of samples for ASTM C39 compressive strength testing was not addressed in the procurement specification for clean cap grout. This document is relevant to Tank 16H grouting operations because clean cap grout was placed above its mounded tank grout to fill remaining void space.

Clean Cap Grout Design Quantity per Cubic Yard based on Batch Ticket #042239; $w:p = 0.50$			
Type I/II Portland Cement (lbs)	Class F Flyash (lbs)	Grade 100 GGBF Slag Cement (lbs)	Water (gal)
174	868	868	114.5

SRR-CWDA-2013-00091. "Industrial Wastewater Closure Module for Liquid Waste Tank 16H, H-Area Tank Farm, Savannah River Site." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. April 2015.

This report is one of the key Tier 2 closure documents for Tank 16H that supports Tier 2 closure authorization, which is DOE-SR's final authorization to proceed with permanent stabilization of the waste tank system. Section 7.3 of this closure module described anticipated Tank 16H grouting operations, and suggested that grouting of the annulus ventilation duct may require an unspecified grout more flowable than tank grout. Notably, Section 7.3.2 of the document indicated awareness of a potential need to provide access points into the tank to address bleed water build-up. Grouting plans described by the closure module were summaries of more detailed information contained in the "Tank 16H Grout Strategy" (SRR-LWE-2014-00013) document; therefore, the reader is referred to the in-depth summary of SRR-LWE-2014-00013 contained herein for information on planned Tank 16H grouting operations.

SRR-CWDA-2014-00011. Smith, F.M. "Evaluation of Vendor Supplied Clean Cap Material for Saltstone Disposal Units." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. March 2014.

The report was prepared to support issuance of an Unreviewed Waste Management Question Evaluation (UWMQE) that assessed clean cap grout proposed for use in Saltstone Disposal Units at the Saltstone Disposal Facility. This report is relevant to Tank 16H grouting operations because clean cap grout was placed in the tank above mounded tank grout to fill remaining void space. The report states that clean cap grout is known to form bleed water, but that the amount of bleed had not been defined for normal clean cap operations. Vitreous State Laboratory (VSL-14R3330-1) tested grouts mixed with the standard dry feeds while varying the water-to-premix ratio and adding NaOH solution to understand their resulting properties (e.g., total bleed, re-absorption time, flowability, and uniformity). The water-to-premix ratio was reduced from 0.59 for saltstone (SRNL-STI-2012-00558) to either 0.45 or 0.50 (C-SPP-Z-00012) for the proposed clean cap grout formulations to reduce bleed; however, this also reduced its flowability (see also SRNL-STI-2012-00558, sample WP023). Use of a caustic additive [6 wt% (1.6M) NaOH], intended to simulate the average concentration of free hydroxide in decontaminated salt solution, decreased total bleed while maintaining grout flowability. Finally, saturated hydraulic conductivity decreased with use of lower water-to-premix ratios (SRNL-STI-2012-00558).

SRR-CWDA-2015-00096. Layton, M. "Unreviewed Waste Management Question Evaluation—Use Alternative Tank 16 Fill Grout (Per Specification C-SPP-Z-00012)." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. September 2015.

This form described a proposed action and evaluation of the action to use flowable clean cap grout (C-SPP-Z-00012) to complete Lifts 5 and 6 near and at the top of Tank 16H. Clean cap grout was proposed to provide an approximately 2-ft (0.6-m)-thick cap on mounded tank grout in the primary and annulus of Tank 16H to minimize potential for voids to remain after grouting

operations had been completed. DOE estimated that the amount of clean cap grout placed in the primary and annulus would not exceed 84,000 and 10,280 gal, respectively. Clean cap grout was not expected to meet all assumed mechanical and chemical properties for tank grout that had been specified in the HTF PA (SRR-CWDA-2010-00128), and therefore placement of clean cap grout could result in a condition or impairment to critical design features for the waste tank outside the bounds of critical PA assumptions. To examine the impact of this action, DOE undertook an evaluation of the potential to meet tank closure performance objectives using the alternative grout fill (SRR-CWDA-2015-00100). Although clean cap grout may not perform as assumed by the PA, DOE suggested that clean cap grout may perform satisfactorily such that the Tank 16H closure performance objectives could be met despite any violation(s) of PA assumptions. The UWMQE Peer Reviewer concurred with UWMQEO's conclusion and also found that placement of clean cap grout would comply with the WD Basis Document (DOE/SRS-WD-2014-001). Final reviewer signatures were obtained on September 8, 2015, more than one week after placement of clean cap grout into Lift 5 of Tank 16H began on August 31, 2015.

SRR-CWDA-2015-00100. "Evaluation of the Use of an Alternative Tank 16 Fill Grout (Per Specification C-SPP-Z-00012) (Interoffice Memorandum to G.C. Arthur from M.H. Layton)." Revision 2. Aiken, South Carolina: Savannah River Remediation, LLC. September 2015.

This interoffice memorandum was included as a supporting reference to SRR-CWDA-2015-00096. The memo documents DOE's evaluation of the impact of switching from placement of tank grout to a more flowable clean cap grout (used in the saltstone disposal facility) in the final Tank 16H primary and annulus grout lifts, Lifts 5 and 6, respectively. Less than 10 percent (a maximum of 84,000 gal) of the total volume of the primary (1,030,000 gal) would be filled with clean cap grout and up to 10,280 gal would be placed in the annulus.

The HTF PA (SRR-CWDA-2010-00128) contained assumptions about the chemical and physical properties and performance of tank grout that would affect waste tank stability, protection from inadvertent intrusion and waste release. In regard to chemical properties, DOE concluded that placing clean cap grout above tank grout during completion of Lifts 5 and 6 in Tank 16H would not negatively impact the reductive capacity of grout placed in the tank because clean cap grout has a greater weight percent ground granulated blast furnace slag cement than tank grout (i.e., 45 weight percent in clean cap grout versus 30 weight percent in tank grout).

In regard to physical properties, this report indicated that clean cap grout may not attain the minimum adequate compressive strength of 2000 psi (138 bars) assumed by the HTF PA (SRR-CWDA-2010-00128; cf. SRR-CWDA-2015-00160). But because its use would both minimize void space and be volumetrically limited, DOE concluded that tank stability and the inadvertent intruder barrier provided by the two-component Tank 16H grout monolith would be maintained. Additionally, the hydraulic properties of clean cap grout are consistent with those assumed by the PA. The mean saturated hydraulic conductivity of three Vault 4 clean cap grout specimens,  $2.2 \times 10^{-9}$  cm/s (Amec Foster Wheeler, 2015; SRR-CWDA-2015-00160) is only slightly greater

than that of tank grout ( $2.1 \times 10^{-9}$  cm/s; SRR-CWDA-2010-00128, Table 4.2-28), and this difference likely would result in only slightly faster water infiltration to the waste layer; however, the clean cap grout layer would be thin and located at the top of the tank, such that the overall effect of this higher conductivity layer on flow out of the contamination zone may be limited.

DOE staff explored these effects on waste release using what DOE indicated was a conservative HTF Goldsim model that was run deterministically with an increased infiltration rate of 16.45 in/yr (compared to 11.67 in/yr) and with early hydraulic degradation of the grout (flow run 17 in Table 5.6-7 of the HTF PA). The resulting impact on peak contaminant release was minor (SRR-CWDA-2015-00100; Figs. 1 and 2). The 1-m and 100-m peak doses remained well below the peak HTF doses documented in the Tank 12 Special Analysis (SRR-CWDA-2015-00073), albeit occurring earlier. DOE concluded that the change in grout formula to one with enhanced reductive capacity but decreased compressive strength and increased hydraulic conductivity at the top of the Tank 16H primary and annulus would not impact compliance with HTF performance objectives.

SRR-CWDA-2015-00159. "Tank 16 Final Configuration Report for H-Tank Farm at the Savannah River Site." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. May 2016.

This report documented data obtained from the grouting of Tank 16H and clarified exceptions that occurred relative to the intended configuration described in the closure module (SRR-CWDA-2013-00091). Data presented included grouting operation dates, average 28-day compressive strength test results obtained from tank grout (but not from clean cap grout) test cylinders, bleedwater test results, and bulk plus clean cap grout fill, cooling coil grout fill, and equipment grout fill volume actuals versus estimates. The report also described three cooling coils that were not completely filled with coil grout, and documented a change in configuration related to use of clean cap grout to complete filling the void space at the top of the primary and annulus after bulk fill tank grout exhibited significant mound development beneath active risers. Due to discrepancies in the closure module description, the report also clarified the nature of equipment remaining in Tank 16H that was filled with grout.

DOE reported average compressive strength test results from a total of 272 ASTM C39 test cylinders. The 28-day post-casting compressive strength average was 2,788 psi (192 bars). DOE also reported that the volume of reducing grout to be placed inside the primary was estimated at 5,552 cubic yards (4,245 cubic meters), while the actual volume of reducing grout placed in the primary was 5,425 cubic yards (4,148 cubic meters), which is 97.7 percent of the estimate. Likewise, the estimated volume of reducing grout required to fill the annulus was 687 cubic yards (525 cubic meters) compared to an actual volume placed of 697 cubic yards (533 cubic meters). Finally, 21 cubic yards (16.1 cubic meters) of reducing grout were estimated as needed to fill Tank 16H risers, whereas 23 cubic yards (17.6 cubic meters) were placed.

SRR-CWDA-2015-00160. "Evaluation of the Performance Assessment Impact of using an Alternative Fill Grout in the H-Area Tank Farm (Interoffice Memorandum to G.C. Arthur from M.H. Layton)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. January 4, 2016.

This interoffice memorandum evaluated the impact to meeting HTF performance objectives (SRR-CWDA-2010-00128) of using more flowable clean cap grout (C-SPP-Z-00012) to complete filling certain tanks in the HTF (e.g., Tank 16H) rather than tank grout. The HTF PA contains assumptions about the performance of tank-filling grout with respect to (i) its chemical properties, (ii) waste tank stability, (iii) inadvertent intruder prevention, and (iv) flow modeling. Use of clean cap grout should not decrease the overall reductive capacity of the grout fill because clean cap grout contains substantially greater weight percent slag than does tank grout [i.e., 45 wt% slag per C-SPP-Z-00012 (Revision 1) compared to approximately 30 wt% slag per C-SPP-F-00055 (Revision 4)]. Use of clean cap grout is not expected to decrease waste tank stability or provide any less a physical barrier to intruders because tested cylinders of Vault 4 (now known as Saltstone Disposal Unit 4) clean cap grout prepared with Grade 100 slag cement yielded compressive strengths >5000 psi (>345 bar) when tested after more than 500 days of hydration. The average compressive strength of four Vault 4 test cylinders (2014-07V1JE4002-0002; 2014-07V1JE4002-0003) was 6202 psi (428 bar), but the memo notes that any clean cap grout that might be prepared for future use in HTF tank closure would use Grade 120 slag cement rather than Grade 100 slag that had been used in Tank 16H, which potentially would further improve its compressive strength. Use of clean cap grout is not expected to increase the overall hydraulic conductivity of the grout fill or invalidate related flow modeling assumptions because three tested cylinders of Vault 4 clean cap grout prepared with Grade 100 slag cement yielded an average saturated hydraulic conductivity of  $2.2 \times 10^{-9}$  cm/s (Amec Foster Wheeler, 2015), compared to the slightly lower value of  $2.1 \times 10^{-9}$  cm/s assumed in the HTF PA. Given that any clean cap grout that might be prepared for future use in tank closure would be batched using Grade 120 slag cement, the hydraulic conductivity of that grout fill would likely be lower. The memo concludes that use of clean cap grout (C-SPP-Z-00012) during HTF tank closures can be executed while yet in compliance with the HTF performance objectives (SRR-CWDA-2010-00128).

SRR-LWE-2014-00013. Walters, C.D. "Tank 16H Grout Strategy." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. January 6, 2015.

This planning document described DOE's intended strategy for grouting Tank 16H, including grout functions, requirements and formulation (C-SPP-F-00055, Revision 4, Attachment 5.5), pour methodology, in-tank equipment, preferred grout-discharge risers, intended ventilation risers, video-camera-based inspections, cooling coil flushing, and tank, annulus, equipment, cooling coil, and riser grouting sequences. DOE's Tank 16H grouting strategy was revised as grouting operations occurred, as reported in the final configuration report (SRR-CWDA-2015-00159), including required adjustments and configuration changes based upon field conditions and tank inspections.

For pour methodology, the grout strategy document stated that "Lessons learned from previous tank closures identified the benefit of a minimal time gap between grout trucks during select lifts in the annulus and the primary tank." It is unclear what "select lifts" DOE is referring to (e.g., lifts associated with ventilation duct grouting). This report indicates that the grout delivery contract did not specify a required grout delivery frequency or a minimum number of cement trucks in

rotation. DOE stated that while that the optimal number of grout trucks in rotation varies from one tank to another due to route length and personnel interaction, ideally there should be 8–10 cement trucks in rotation.

DOE planned to place tank grout in the Tank 16H annulus and inside its ventilation duct. DOE stated that because the duct was already suitably supported, placing an initial bed of grout to provide support to the duct was unnecessary. The ductwork was said to contain numerous holes with the implication being that the grout could enter the duct through the holes. Additionally, the duct work contained waste material that DOE indicated could lead to plugging of the duct if grout were placed through the pants legs directly into the duct. The horizontal portion of the ventilation duct was to be filled with grout that entered it from above through open registers in its top surface and from below through various openings caused by degradation of the ductwork (ADAMS Accession No. ML16167A237). The vertical portions of the ventilation duct (“pant legs” or ventilation riser inlet/outlet) were filled to grade level with tank grout (not clean cap grout), but only after the rest of the annulus, including Lift 6, had been filled with tank and clean cap grout (ADAMS Accession No. ML16167A237). The Tank 16H procedure contrasts with all other duct grouting operations to date, including those later conducted for Tank 12H, which involved preliminary filling of the horizontal duct by directing grout into and through the vertical pant legs. The Tank 16H duct-grouting procedure assumed that there was no danger of the vertical duct being crushed from external pressure applied by grout fill in the annulus; therefore, the vertical duct was filled last, after all annulus lifts had been placed (ADAMS Accession No. ML16167A237).

Open equipment was to be filled with equipment grout only after any low-level openings were visually determined to be encased in tank grout. Vertically oriented cooling coils in the tank primary require that structural support be provided by an external bed of tank grout to help prevent their failure and or roof collapse during internal grouting. This report called for exercise of engineering judgement to limit the external level of supporting tank grout to the minimum necessary that would provide support to the cooling coils while maximizing the potential for guillotined or severed coils to vent. Guillotined coils were to be grouted simultaneously from each end until full, with any residual flushwater remaining pocketed inside the coil. Any guillotined coils not connected to coil inlets or outlets would not directly receive cooling coil grout, and could only be passively filled with tank grout to some extent, thereby potentially leaving empty pore space within. The Tank 16H grout strategy specified that intact cooling coils containing chromate water should be flushed with *one* pore volume of water immediately prior to initiating internal grouting to ensure a uniformly wetted path exists for grout to follow. In contrast, the Tanks 5F and 6F lessons learned document (SRR-CWDA-2014-00015) had recommended triple rinsing of cooling coils in the future, including those of Tanks 16H and 12H. The Tank 16H grout strategy called for flushwater (and later grout) to be collected at each coil outlet in a tote or disposed of in another waste tank. After cooling coil grout was visually detected at each outlet, additional grout was to be introduced to ensure complete filling. Finally, the strategy document indicated that 12 primary tank and 18 annulus risers were slated to be filled with tank grout (cf. SRR-CWDA-2015-00159) up to the level of the riser opening, generally above grade level.

SRR-LWE-2014-000150. Ostler, M. "Tank 16-H Closure Assurance Plan." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. March 2015.

This planning document identified process requirements from the Tier 1 and Tier 2 closure documentation and provided a strategy (and checklist) to ensure that requirements were met and that required grouting operation documentation (including various surveillances, batch tickets, grout testing, and grouting operation video) was generated and archived during stabilization of residual waste (i.e., during grouting operations). The plan was a tool for SRR to ensure that Tank 16H was closed successfully, having met all regulatory process and documentation requirements. The plan stated that the grout testing laboratory qualification must be documented and that DOE should verify that the SRR Testing Laboratory performed grout testing per ASTM standards listed in Attachment 5.3 of the grout procurement specification (C-SPP-F-00055, Revision 4). The planning document also stated that any changes to the initially qualified tank grout formulation (C-SPP-F-00055, Revision 4, Attachment 5.5) must be evaluated for impact to qualification and to previous SRNL testing. If re-qualification was not required, then an evaluation and justification should be provided by SRR Closure Engineering in either a technical report or a waste management question evaluation (e.g., SRR-CWDA-2015-00100). The plan stated that any non-conformances should be documented.

SRR-LWP-2014-00049. Sareen, H. "SRR Subcontractor Surveillance Plan Furnishing and Delivery of Tank Closure Grout (TK 16) (P.O. No. SRR-064184-1)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. June 1, 2015.

This document described the method that SRR Construction Quality Services would use for performing oversight of the subcontractor during furnishing and delivery of tank grout for Tank 16H. The plan outlined the tank grout quality control surveillance and quality assurance assessments to be performed, and inspection records and checklists to be generated. This document did not anticipate use of clean cap grout in the Tank 16H primary and annulus.

SRR-TCR-2015-00024. "Tank 16 Grouting Lessons Learned (Interoffice Memorandum from B. Davis to L. Blackford and J. Williams)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. January 27, 2016.

This report documented lessons learned from Tank 16H grout and tank preparation and grouting operations, as well as recommendations for future operations including: (i) to reduce plugging of slick lines with grout, remove diversion valves and use removable spool pieces to route grout to tank fill points; (ii) to significantly reduce hazards to workers and work stoppages, eliminate decant totes and grout intact coils directly to a waste tote, especially when only a few intact coils are to be grouted; (iii) to allow use of flowable clean cap grout to complete tank grouting in potential future cases where significant mounding of tank grout occurs, complete all necessary reviews, evaluations and testing in advance; (iv) to enable use of less restrictive grout placement or lift thicknesses, provide actual grout properties (specific gravity, set time, etc.) rather than bounding values to the structural department for use in their calculations; (v) to significantly reduce hazards to workers, evaluate the potential to eliminate (or modify) cooling coil grouting; (vi) to significantly reduce related work stoppages, conduct both failed and intact cooling coil grouting practice dry runs when all equipment and hoses are in place; (vii) to maintain lubrication of the slick line, evaluate use of additional pigs or other methods; (viii) determine a reasonable or acceptable range for bleed water exuded by tank grout by analyzing data from Tank 16H grout testing; (ix) anticipate seasonal weather conditions that will

effect planned grouting operations and make appropriate preparations in advance (weather related contingency for slicklines, i.e., hot weather vs. cold weather); (x) to reduce set up costs, resource use, process time and risks, develop a method to flush cooling coils immediately followed by grouting of same (flushing and grouting has been conducted as two separate processes to date).

Finally, DOE and SCDHEC entered into an agreement that clean cap grout would not be used to cap Tank 12H without their express and prior approval. Therefore, DOE is to be notified as soon as mounding or pluggage issues occur that may later warrant use of clean cap grout so that they can immediately notify SCDHEC. It was recommended in the lessons learned document that associated requirements be included in the Tank 12H grout strategy document, communications plan, grouting work packages, and job briefings.

Work Order No. 01324150-64. Fail, J.A. "TK Clos & Reg Cn to Perform Grout Prep/Grout Placement TK 16." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. August 22, 2014.

This work order provided detailed lists of activities to be performed during grouting of Tank 16H. The order called for six grout placements (HTF-SKM-2014-00031, Grout Placement Plan) to be poured through tremies in multiple risers in the primary and annulus with a maximum drop height of 5 ft (1.5 m), as well as riser placements. The work orders included safety precautions and limitations that were to be followed (including radiation control procedures) during grouting.

### **Review Summaries of Tank 12H Documents:**

LWO-LWQ-2016-00001. Thompson, J.W. "SRR Subcontractor Surveillance Plan Furnishing and Delivery of Tank Closure Grout (TK 12) (P.O. No. SRRA-064184-1)." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. January 13, 2016.

This document described the method that SRR Construction Quality Services would use for performing oversight of the subcontractor during furnishing and delivery of tank grout (C-SPP-F-00055) and clean cap grout (C-SPP-Z-00012) for Tank 12H. The plan outlined the grout quality control surveillance and quality assurance assessments to be performed, and inspection records and checklists to be generated.

SRR-CWDA-2014-00086. "Industrial Wastewater Closure Module for Liquid Waste Tank 12H, H-Area Tank Farm, Savannah River Site." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. May 2015.

This report is one of the key Tier 2 closure documents for Tank 12H that supports Tier 2 Closure Authorization, which is DOE-SR's final authorization to proceed with permanent stabilization of the waste tank system. Section 7.3 of this closure module described anticipated Tank 12H grouting operations, and suggested that grouting of the annulus ventilation duct may require an unspecified grout more flowable than tank grout. Notably, Section 7.3.2 of the document indicated awareness of a potential need to provide access points into the waste tank to address bleed water build-up. Grouting plans described by the closure module were summaries of more detailed information contained in the "Tank 12H Grout Strategy" (SRR-LWE-2014-00147) document; therefore, the reader is referred to the in-depth summary of the Tank 12H grout strategy for information on planned Tank 12H grouting operations.

SRR-CWDA-2015-00057. "Evaluation of the Use of Grade 120 Slag Cement in Tank Closure Grout versus Performance Assessment Assumptions (Interoffice Memorandum to G.C. Arthur from M.H. Layton)." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. August 27, 2015.

This interoffice memorandum was included as a supporting reference to SRR-CWDA-2015-00088. The memo documents DOE's evaluation of the impact of switching from use of ground granulated blast furnace slag cement Grade 100 to Grade 120 (cf. ASTM C989) in the tank grout formulation. Section 3.2.3.3 of the tank grout procurement specification (C-SPP-F-00055, Revision 4; see also Attachment 5.5) specified that only Grade 100 slag cement should be used in the mix. However, DOE staff defended the position that allowing use of Grade 120 slag in place of Grade 100 would not result in a tank grout mix incapable of meeting PA assumptions, would be consistent with the inputs and assumptions of the PAs, and could be carried out in compliance with performance objectives assuming other grout performance and testing requirements were met.

Blast furnace slag is a byproduct of the iron and steel industry that is finely ground and marketed as a partial substitute for Portland cement. The particle size distribution of slag is generally finer than or similar to that of Portland cement (WSRC-TR-2001-00359). Major components are oxides of silicon, aluminum, calcium, and magnesium; minor components include compounds containing manganese, iron, and sulfur (SRNL-PSE-2007-00282, Table 1). Elemental composition varies slightly depending on source material and

additives used in iron or steel production. The reductive capacity of slag is relatively insensitive to its chemical composition (PNNL-22977/EMSP-RPT-015, Table 3.1). The FTF and HTF PAs (SRS-REG-2007-00002 and SRR-CWDA-2010-00128) each contained assumptions about the reductive capacity of tank grout. The quantity of slag used in tank grout was specified to produce desirable chemistry and extend the reductive capacity timeline. Slag grade and associated physical properties (i.e., particle size distribution and activity index) do not impact reductive capacity (VSL-15R3740-1, Table 5.5); therefore, use of a carefully selected Grade 120 slag in tank grout, such as Lehigh Hanson or Lafarge, would not violate PA assumptions about its chemical properties. For example, compared to the lower reductive capacity (i.e., 722  $\mu\text{eq/g}$ ) of the Holcim, Inc. Grade 100 slag that was originally used in tank grout, reductive capacities of Lehigh Hanson (preferred) and Lafarge (optional) Grade 120 slags are 812  $\mu\text{eq/g}$  and 740  $\mu\text{eq/g}$ , respectively (VSL-15R3740-1, Table 5.5). Switching to use of either one of these slags would therefore preserve or enhance the desirable chemical properties (i.e., reductive capacity) of any cementitious grouts that these slags are used in.

The Tank Farm PAs also assume that tank grout has adequate compressive strength [i.e., minimum of 2000 psi (138 bars) at 28 days post-placement, per FTF Table 3.2-4 and HTF Table 3.2-9] to withstand the overburden load on the tank at closure. Slag grade does not negatively impact waste tank stability because tank grout will still be required to achieve the minimum 28-day compressive strength of 2000 psi (138 bars). Testing performed on tank grout using Grade 120 slag has an average 28-day compressive strength of 2180 psi (150 bars), exceeding the minimum PA requirement (SDDR No. 13182).

Finally, the Tank Farm PA flow modeling assumed tank grout material properties (FTF PA Section 4.2.3.2.3 and HTF PA Section 4.2.2.2.4) that would limit infiltration of water to the contamination zone at the bottom of the tank. The tank grout formulation was developed to meet assumed material properties, with conformance to the grout formulation validated through adherence to tank grout specification requirements (C-SPP-F-00055). DOE staff maintain that use of Grade 120 slag in tank grout, while not relaxing other tank grout specification testing and performance requirements (e.g., flowability, weight of grout ingredients such as cement, sand, water, fly ash, slag, etc.), will not negatively impact the grout material functionality with regards to water flow and diffusion, because the hydraulic conductivity of tank grout will not be impacted by the change in slag grade.

SRR-CWDA-2015-00088. Layton, M.H. "Unreviewed Waste Management Question Evaluation – Use of Grade 120 Slag in Tank Closure Grout." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. September 2015.

This form described a proposed action and evaluation of the action to use ground granulated blast furnace slag cement Grade 120 in place of Grade 100 in the waste tank grout formulation. Specifically, a deviation (SDDR No. 13182) from Section 3.2.3.3 of the tank grout procurement specification (C-SPP-F-00055), which currently requires use of Grade 100, was proposed to allow either grade to be used. To examine the impact of this action, DOE undertook an evaluation of the potential to meet tank closure performance objectives using Grade 120 instead of Grade 100 slag (SRR-CWDA-2015-00057). DOE indicated that use of Grade 120 as an alternative to Grade 100 would be consistent with the inputs and assumptions contained in the Tank Farm PAs (SRS-REG-2007-00002 and SRR-CWDA-2010-00128), and also stated that because the FTF and HTF WD Basis Documents (DOE/SRS-WD-2012-001 and DOE/SRS-WD-2014-001) do not prescribe a grout formulation but only describe general grout properties, the

proposed action was within the bounds of the WD Basis Documents. The justification indicated that because the proposed action would not relax other grout specification requirements and tank grout would continue to perform as assumed in the Tank Farm PAs, the action would not impact compliance with performance objectives.

SRR-LWE-2012-00030. "Tank 12 Cooling Coil Flushing Strategy (Interoffice Memorandum to M.D. Buxton from S.J. Worthy and J.R. Tihey)." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. November 28, 2012.

This interoffice memorandum documented DOE's guillotined and broken cooling coil decontamination and flushing strategy that would be employed to remove residual contamination from Tank 12H. Exterior contamination was to be removed from all cooling coils during mechanical mixing, oxalic acid cleaning, and water wash campaigns. Twenty-seven failed vertical coils and one failed horizontal coil were to be flushed from both sides (supply and return) simultaneously with H-Area well water; flushwater was to drain through coil leak sites into the tank. DOE estimated vertical and horizontal cooling coil volumes as approximately 454 and 625 L (120 and 165 gal), respectively, and that the flushwater volume added to the primary tank would exceed 11,000 L (3000 gal). The memo did not describe the flushing strategy for eight intact cooling coils in Tank 12H, indicating instead that flushing of these coils would occur only during the grouting phase.

SRR-LWE-2014-00147. Chandler, T. "Tank 12H Grout Strategy." Revision 1. Aiken, South Carolina: Savannah River Remediation, LLC. August 31, 2015.

This planning document described DOE's intended strategy for grouting Tank 12H, including grout functions, requirements and formulation, pour methodology, in-tank equipment, preferred grout-discharge risers, intended ventilation risers, video-camera-based inspections, cooling coil flushing, and tank, annulus, equipment, cooling coil, and riser grouting sequences. DOE's Tank 12H grouting strategy may have been revised as grouting operations occurred and the final grouted tank configuration will be reported in the final configuration report, including any required adjustments and configuration changes based upon field conditions and detailed inspections. As with Tank 16H, the grout delivery contract for Tank 12H did not specify a required tank grout delivery frequency or minimum number of trucks in rotation. In this document, DOE clarified that the ideal grout delivery frequency is thought to be 8 to 10 trucks *per hour*. This differed from the Tank 16H Grout Strategy document, which had suggested that there should be 8 to 10 cement trucks *in rotation*.

DOE planned to place tank grout in the Tank 12H annulus and its ventilation duct. Grout was to be placed in the annulus between the outside radius of the ventilation duct and the annulus steel pan. Unlike the strategy used to grout Tank 16H, an approximately 6-to-12-in (15-to-30-cm)-deep grout layer was to be placed on the annulus pan floor to support the horizontal ductwork during grouting. The horizontal part of the duct was then to be filled by placing grout directly into the vertical inlet piping system ("pant legs" or ventilation riser inlet/outlet) until grout was observed to exit the horizontal ductwork through its register openings. As the annulus grout level was raised, grout was to flow through any remaining register openings into any unfilled portions of the horizontal ductwork. SRR-LWE-2014-00147 indicates both of the following for Tank 12H: (1) vertical portions of the duct were to be filled after completion of the annulus bulk fill, and (2) to lessen the potential for duct collapse, the vertical section of the ventilation inlet duct will be filled with grout to grade level in parallel with bulk filling of the

annulus<sup>17</sup>. It is unclear which statement was intended. The annulus exhaust riser was to be grouted to grade level after the grout level in the annulus reached the bottom of the riser. Because DOE anticipated that groundwater intrusion into the annulus could be a problem, a temporary water removal system was installed (SRR-LWE-2015-00048) to transfer any water in the annulus into Tank 10H prior to grouting. DOE expected water levels in the annulus could be reduced to no more than 2 in (5 cm) by this water removal system. If necessary, DOE was also prepared to remove accumulated water from the annulus into decant totes after annulus grouting had begun.

To alleviate mounding beneath the tremie, the Tank 12H grout strategy report identified distribution pipe mobility within risers as a means to allow some limited directional control of discharging grout. The report also allowed for potential elimination of the tremie based on grout freefall experimental results obtained during grout drop testing (RPT-5539-EG-0016), which indicated that grout properties would not be adversely affected by freefall. Also based on grout drop test results (i.e., Test 2), DOE indicated that they planned to avoid placing grout directly into wet areas of the tank because doing so could enhance bleed water segregation.

Open equipment was to be filled with equipment grout only after any low-level openings were visually determined to be encased in tank grout. Vertically oriented cooling coils in the tank primary require structural support be provided by an external bed of tank grout to help prevent their failure and or roof collapse during internal grouting. This report called for the exercise of engineering judgement to limit the external level of supporting tank grout to the minimum necessary that would provide support to the cooling coils while maximizing the potential for guillotined or severed coils to vent. Twenty-eight (28) of thirty-six (36) cooling coils in Tank 12H had failed prior to commencing grouting operations, and were flushed previously (SRR-LWE-2012-00030). Guillotined coils were to be grouted simultaneously from each end until full, with any residual flushwater remaining pocketed inside the coil. Any guillotined coils not connected to coil inlets or outlets would not directly receive cooling coil grout, and could only be passively filled to some extent with tank grout. The report indicated that intact cooling coils containing chromate water would be flushed with water. The Tank 12H grout strategy did not discuss disposal of chromate-laden flushwater in any detail, instead referring to a related work order that would be developed. The strategy document also did not discuss how DOE would ensure complete filling of any remaining intact cooling coils. Rather, the report described how DOE would test the eight remaining cooling coils to determine if they remained intact or had also failed. Nine primary tank and six annulus risers were slated to be filled with tank grout up to the level of the riser opening, generally above grade level.

SRR-LWE-2014-00161. Walters, C.D. "Tank 12 Internal Equipment Evaluation." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. February 12, 2015.

This report describes equipment (and inventory) left in the Tank 12 primary and annulus that will be grouted in place after cleaning operations. Two sampling crawlers were abandoned inside the primary, one near Riser 6, and the other northeast of the center column. Riser 6 contains an abandoned transfer jet and pump. Riser 7 contains a submersible transfer pump and a

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<sup>17</sup> SRR-LWE-2015-00048 indicates the vertical section of the annulus inlet duct was to be filled with cooling coil grout (not tank grout) during Placements 5, 7, and 9, whereas the annulus exhaust piping was to be filled during riser placements near the end of grouting operations.

caisson with thermowell. The North riser contains an annulus transfer jet. The equipment remaining in Tank 12 contains insignificant inventory estimated at a volume of 46.6 L (12.3 gal).

SRR-LWE-2015-00032. Ostler, M. "Tank 12-H Closure Assurance Plan." Revision 0. Aiken, South Carolina: Savannah River Remediation, LLC. April 2015.

This planning document identified process requirements from the Tier 1 and Tier 2 closure documentation and provided a strategy to ensure that requirements were met and that required documentation (including various surveillances, batch tickets, grout testing, and grouting operation video) was generated and archived during stabilization of residual waste (i.e., during grouting operations). The plan was a tool for SRR to ensure that Tank 12H was closed successfully, having met all regulatory process and documentation requirements. The plan stated that the grout testing laboratory qualification must be documented and that DOE should verify that the SRR Testing Laboratory performed grout testing per ASTM standards listed in Attachment 5.3 of the grout procurement specification (C-SPP-F-00055, Revision 4). The planning document also stated that any changes to the initially qualified tank grout formulation must be evaluated for impact to qualification and to previous SRNL testing. This document indicated that the tank grout formulation to be placed into Tank 12H could exercise the option to use Grade 120 slag cement, whereas Grade 100 had been used during prior tank grouting operations. If re-qualification was not required, then an evaluation and justification should be provided by SRR Closure Engineering in either a technical report or a waste management question evaluation. The plan stated that any non-conformances should be documented.

SRR-LWE-2015-00048. Griffin, A.L. "Path Forward for Tank 12 Annulus Liquid Removal (Interoffice Memorandum to E. Patten et al. from A.L. Griffin and G.C. Arthur)." Aiken, South Carolina: Savannah River Remediation, LLC. Revision 0. July 7, 2015.

This interoffice memorandum provided DOE's path forward on removal of water from the Tank 12H annulus (as a non-waste transfer), prior to initiation of grouting. Tank 12H has a history of groundwater intrusion. Removal of groundwater was necessitated when an unanticipated water level of approximately 28 cm (11 in) was identified during video inspection of the annulus on May 16, 2015. Subsequently, video inspection frequency of the Tank 12H annulus was increased to daily on May 20, 2015. The annulus liquid sampled on May 21, 2015 (Attachment 1) had low-level contamination, and therefore was classified as non-waste. The recommended path forward was to transfer intruded groundwater out of the Tank 12H annulus and into adjacent Tank 10H. To promote annulus drying, supplementary ventilation was installed and operated. It was also recommended that the equipment required to pump groundwater out of the annulus be installed as a temporary modification.

Work Order No. 01337683-33. Patton, G.W. "Placement of Bulk Fill Grout: Tank 12." Revision 0. Aiken, South Carolina: Savannah River National Laboratory. April 29, 2015.

This work order provided detailed lists of activities to be performed during grouting of Tank 12H. The order called for nine grout placements (HTF-SKM-2015-00021, Grout Placement Plan) to be poured through tremies in multiple risers in the primary and annulus with a maximum drop height of 10 ft (3 m), as well as multiple riser placements. The work orders included safety precautions and limitations that were to be followed (including radiation control procedures) during grouting. Note that this is the first tank closure work order for which the maximum drop

height was increased to greater than 5 ft (1.5 m). Placement 3 involved grouting of the horizontal section of the ventilation duct. A tremie was to be installed in the annulus inlet to fill the horizontal ventilation duct to the point at which grout was observed exiting the “horizontal ducting distribution holes”. The vertical section of the annulus inlet duct was to be filled with cooling coil grout (not tank grout) during Placements 5, 7, and 9, whereas the annulus exhaust piping was to be filled during riser placements near the end of grouting operations. If workers observed water in risers, they were to add dry grout per guidance of SRR-CWDA-2012-00051, Revision 2.

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## Attachment 1

SRR-CWDA-2015-00170 transmitted more than 40 DVDs of Tank 16H grouting operation video. This attachment provides a description of *horizontal ductwork grouting* in the annulus of Tank 16H, which occurred on June 11, 2015. During the early minutes of annulus grouting that day (11:33 am+), the West Riser video camera pointed straight down rather than to where it could have observed grout directly entering nearby ventilation duct register(s). By the time the camera panned downstream (11:36 am), any nearby duct(s) were covered with grout and no longer visible. It is unknown the extent to which the ventilation duct is fully filled with grout.

At 12:18 pm, the East Riser video camera first glimpsed stagnant, non-flowing grout already at rest inside the horizontal duct via a register in its field of view. A few minutes later (12:21 pm), fresh grout was observed flowing inside the duct as viewed through this register (Figure A-1A). Upstream, the duct was not completely full of grout; flow in the duct was under open channel conditions, not conduit flow/plug flow conditions. By 12:23 pm, grout had ceased flowing through the duct. Subsequent truckloads of grout did not continue to fill the interior of the duct (e.g., still stagnant at 12:42 pm) in this location until, as head in the annulus increased, a fresh batch sent grout flowing *from above* down into the partially filled duct via the register (Figure A-1B) observed by the East Riser camera (1:00 pm). Grout had been immobile and setting up inside the partially filled duct for almost 40 minutes before fresh grout entered this part of the duct again, this time from above. This new batch of fresh grout pouring into the open register may or may not have expired (i.e., truck emptied or discharge ceased) before the partially filled duct became completely filled up to that register location. Because grout placed earlier had been flowing in the duct under open channel conditions, some of the fresh grout filling the register from above had to reverse course and flow backward in the duct to backfill the remaining empty porosity. If the truckload of grout expired while filling was ongoing, empty space may be preserved inside the duct away from the register opening because hardening grout had now been placed around this register opening on all sides; additional truckloads of grout would not necessarily have been able to overcome the inertia of the setting grout.

Meanwhile, on the other side of the East Riser camera's field of view, it took until 12:52 pm for grout to be observed flowing *inside* the duct (as viewed from a register). The initial pulse of grout flowing inside the duct from this other direction stopped flowing by approximately 12:54 pm. Then, 14 minutes later at 1:08 pm, grout flowing in the annulus from the original direction overtopped this register opening and flowed into that part of the duct from above.

Visual landmarks such as register openings disappeared beneath pulses of grout even while inflow down into the register opening was likely still occurring. Cameras would pan away and then possibly return to view a register opening beneath flowing grout, but the most obvious visual cue (the register opening) was essentially buried such that subtle grout flow behaviors that may have indicated continued duct infilling were not easily interpreted. If visual guides for duct openings (perhaps a mark on the tank or vault walls above) had been present in the annulus of Tank 16H, DOE may have been better able to associate observed surficial grout flow behavior with the duct filling process.



**Figure A-1. Still Shots of Grout Placement in the Annulus Duct of Tank 16H.**

## Attachment 2

### Development of an Acoustic Emission Crack Signal Detection Technique for Waste Stabilizing Tank Grout

The U.S. Nuclear Regulatory Commission (NRC) previously sponsored studies at the Center for Nuclear Waste Regulatory Analyses (CNWRA®) to better understand the potential for cracks and other preferential pathways to form in tank grout monoliths during the early years after grout is placed. Results from this study indicated that grout monoliths may be at risk for multiple cracking regimes, particularly in the first 24 hours post-placement. The CNWRA is now providing technical assistance to the NRC to test the feasibility of using acoustic emission (AE) technology for passively monitoring crack formation within cementitious tank grout, including monitoring during the early stages of hydration when the cementitious materials are in a gel form and difficult to monitor acoustically. Using AE monitoring, it may be possible to record the timing and location of cracking and to map crack propagation throughout cementitious materials. Such data might be used to improve understanding of the mechanisms of crack development and distribution of cracks within the cementitious materials.

During fiscal year 2015, CNWRA staff performed a sequence of experiments to develop an understanding of the ultrasonic properties of tank grout and saltstone. The resulting property data were used to develop an AE monitoring technique capable of detecting and locating cracking events in the grout, which was demonstrated on a mesoscale specimen of tank grout during hydration. Ultrasonic experiments were performed using standard ultrasonic probes and driver instrumentation. Acoustic emission experiments were performed using a 16 channel Physical Acoustics Corporation DiSP Acoustic Emission Workstation, Model PCI-2 (i.e., an AE instrument). Two types of reducing tank grout and two types of non-radioactive saltstone were used to develop specimens for the experiments, where early experiments were conducted on proxy formulations, such as a specimen cored from the intermediate-scale grout monolith, until materials necessary to develop the LP#8 tank grout and saltstone formulas became available. All specimens contained Grade 100 ground granulated blast furnace slag cement.

Initial testing was performed on tall cylindrical specimens [70 mm (2.8 in) diameter and more than 200 mm (7.9 in) height] of both grout types to determine their ultrasonic properties in their hardened state. Ultrasonic through-transmission measurements were conducted using a variation of the method defined in Appendix X2 of ASTM E494. From these tests, tank grout was determined to have a nominal longitudinal wave speed of 4.14 mm/μs, a nominal shear wave speed of 2.9 mm/μs, and an approximate attenuation of 65–75 dB/m (20–23 dB/ft). Saltstone (identical to clean cap grout except for the presence of additional salts) was determined to have a nominal longitudinal wave speed of 2.39 mm/μs, a nominal shear wave speed of 1.13 mm/μs, and an approximate attenuation of 80–100 dB/m (24–30 dB/ft). Both grout materials exhibited peak propagation amplitudes in the 100–150 kHz range and both materials permitted longitudinal wave signals to propagate with greater signal response than their shear wave counterparts.

The initial findings on the fully hardened specimens were used to define preliminary AE acquisition settings that could be tested on 1-to-2 L (1-to-2 qt) bench-top specimens. Investigations were conducted by installing various configurations of AE sensors on the specimens and then performing ASTM E976 Hsu Nielsen source tests (pencil lead breaks) at known locations around their surfaces. The recorded pencil lead break signals were used to

evaluate and refine AE sensor arrangement, acquisition settings, and data processing logic used to locate signal sources. Through initial refinement of the AE monitoring technique on the bench-top specimens, a sensor arrangement capable of detecting and locating pencil break events with an average error of less than 26 mm (1 in; comparable to 1 wavelength) was selected.

An experiment was then performed to passively monitor the hydration process of a mesoscale, 108-L (114-qt) specimen of tank grout from initial placement through the first full month of hydration and hardening. The intent of the experiment was to evaluate the detection sensitivity and location capability of the AE monitoring technique against artificial crack sources throughout the grout hardening process and to collect ultrasonic property data throughout the grout hydration process. Given past experience with specimens of similar size, significant natural cracks were not expected to form, so artificial signals were periodically introduced via pencil break tests performed across the top surface of the specimen. Over the 32-day monitoring period, the AE system detected artificial signals with increasing consistency and accuracy. In the first few days of testing, signal detection and source location were prevented by high attenuation in the gel-like grout, which severely limited the measurement of key ultrasonic property data. By the end of the test, the system was demonstrating 100 percent detection of pencil lead breaks and location accuracy of less than 12 mm (0.5 in) across the top surface of the specimen.

In light of the severe attenuation observed in the initial mesoscale AE monitoring experiment, an additional experiment was devised to measure wave speed and attenuation throughout the grout hydration process. The method relies on performing periodic automated through-transmission tests on two specimens of grout with two known thicknesses and on a reference of water, using their relative signal responses and times-of-flight to calculate attenuation and phase velocity at each stage in hydration. The group velocity data could then be derived from the phase velocity data. Initial testing was performed on LP#8 tank grout. Initial data indicate that there are two frequency bands conducive to AE monitoring—one in the 100–150 kHz range and a second near 50 kHz. Although detailed analysis of the velocities in these frequency bands was prevented by experimental complications, the phase velocities at higher frequencies were successfully captured, and attenuation response was consistent with previous test observations.

In future work, additional testing is required to complete characterization of the ultrasonic properties of tank grout and saltstone. Once the remaining property data are measured, CNWRA staff can optimize the AE monitoring technique. If AE-based crack detection and location is proven successful at the mesoscale, then the monitoring approach could be further refined for implementation on waste tanks for detecting cracks that may affect the capability of tank grout and clean cap grout to provide a low-permeability reducing environment that limits release of key radionuclides.

### Attachment 3

#### List of Outstanding References

##### Follow-up Action Items from the May 17, 2016, teleconference:

1. DOE indicated that they did not measure the accumulated volume of water at the top of Tanks 5F/6F that led to creation of a work order to remove water from the tank top prior to grouting the risers. The NRC indicated that they recall DOE reporting that about 12 inches of standing water was observed in one of the tank risers. DOE indicated that they may have pumped standing water out of one riser but they would need to go back and confirm the quantity of water pumped out of Tanks 5F/6F, if any.
2. DOE to provide follow-up information on how much water was pumped out of Tank 12H annulus and primary (DOE estimated about 1000 gallons was pumped out of the Tank 12H annulus).
3. DOE to provide information on the quantity of Slick Willie/water used in Tanks 18/19 as a pump priming agent and slick line lubricant (similar information was previously provided for Tanks 5F/6F but not for Tanks 18F/19F).
4. DOE will provide the grout formulation used for clean cap grout in Tank 16H (3 formulations were provided in procurement specification CSP-SPP-Z-00012 and it is not clear which was used). The design water-to-cement ratio listed on 2 batch tickets for clean cap grout was 0.5 (but two formulations had 0.5 in CSP-SPP-Z-00012). DOE will also look at the batch tickets for clean cap grout that were provided to NRC (042239 and 042345) and clarify differences between the two batch tickets (one included the ingredient TEMPER in the mix and one did not). The NRC specifically asked DOE to clarify if caustic was used in the formulation (DOE did not think caustic was added because that would speed up the hydration reactions and cause the cement to be less flowable) and to clarify what the ingredient "TEMPER" is. DOE offered an on-the-spot description of "TEMPER" but may have some additional follow-up information that they can provide to the NRC.

### **Follow-up list of questions from the May 17, 2016, teleconference**

The NRC provided DOE with a list of requested references via email on February 26, 2016, and updated the requested reference list via e-mail on March 30, 2016. Most of these references were provided to the NRC by DOE prior to the May 17, 2016, teleconference. Due to time constraints during the May 17, 2016, teleconference, the NRC was unable to ask a few questions related to the new reference reviews and other lingering questions. The NRC requests DOE to respond to the following questions via email or letter. Alternatively, interested parties could participate in a follow-up teleconference to discuss these questions, if preferable to DOE:

1. NRC requested the final specification for the clean cap grout as a follow-up action to the May 17, 2016, teleconference. Could DOE clarify how it achieves the minimum flowability given that SRNL-STI-2012-00558 indicates that flowability would be compromised at a water-to-cement ratio of 0.51, and that the one most-relevant sample tested in SRNL-STI-2012-00558 (sample WP023 with a water-to-cement ratio of 0.51) had slump flow of only 18.6 cm (7.5 in) and no sample had greater slump flow than 29 cm? Could DOE clarify if any Daratard or any admixtures were used in the Tank 16H clean cap specification, or if there is an option to use admixtures in the future?
2. Could DOE clarify why compressive strength measurements are not required for the clean cap grout?
3. Could DOE provide reference VSL-14R3330-1, which provides test results for clean cap grout mixed at varying water-to-premix ratios and adding NaOH solution (measures total bleed, reabsorption time, flowability, and uniformity).
4. NRC requested and DOE provided Work Order Nos. 01324150-64 and 01337683-33. However, key attachments were not provided. Could DOE provide key attachments to these work orders? For example, key attachments HTF-SKM-2014-00031 and HTF-SKM-2015-00021 are requested. NRC also requested references related to the change to and testing of Grade 120 slag.
5. Could DOE clarify if all testing of Grade 120 slag is provided in VSL-15R3740-1? DOE indicated that information is provided in SRR-CWDA-2015-00088, but testing results do not appear to be included. What testing, if any, has been completed for tank fill, equipment, cooling coil, and clean cap grout prepared with Grade 120 slag?
6. With regard to presence of standing water and water removal in Tank 12H, DOE indicated an expectation that water levels in the annulus could be reduced to no more than 2 inches by the water removal system and that the remainder had to evaporate. DOE was also prepared to remove accumulated water from the annulus into decant totes after annulus grouting had begun. Could DOE clarify how much water remained in the Tank 12H annulus prior to grouting and if 2 inches represents the limit to how much water can be removed by the water removal system (e.g., is the ventilation system used to remove additional water).
7. With regard to use of RECOVER, could DOE clarify why RECOVER dose was changed from 50 oz in Tanks 5F and 6F to either 30 oz or 60 oz in Tank 16H? Although a range is allowed to be used, it is unclear what led to the change.

### **Other Follow-up List of References, Questions from Tank 12/16 Grout TRR**

1. Grout shrinkage testing report(s) (when completed).
2. Modeling files associated with SRR-CWDA-2015-00100 (evaluation of impact of clean cap grout in Tank 16H), if not already provided.
3. Most recent version of SRR-CWDA-2012-00051. NRC staff think the most recent version may be "Critical Assumptions in the F-Tank Farm Operational Closure Documentation Regarding Waste Tank Internal Configurations." Revision 2. Aiken, South Carolina: Savannah River Remediation, LLC. March 28, 2012.
4. Reference VSL-14R3330-1, which apparently provides documentation of tests with clean cap grouts mixed with the standard dry feeds while varying the water-to-premix ratio and adding NaOH solution to understand their resulting properties (e.g., total bleed, re-absorbtion time, flowability, and uniformity). This report was cited in SRR-CWDA-2014-00011.
5. SRR-CWDA-2014-00011 appears to contradict information provided by DOE during the May 17, 2016, teleconference regarding the impact of caustic on clean cap grout flowability. The report indicates, "As expected, lower water-to-premix [ratio] formulations produced less bleed water but also reduced the flowability of the mix. In addition, use of 6 wt% (1.6M) NaOH decreased the total bleed while still maintaining sufficient slump to facilitate flow once discharged into the SDU. The use of a caustic solution was intended to simulate the average concentration of free hydroxide in the DSS; the presence of hydroxide is known to enhance the dissolution of the slag component and the rate of slag hydration, which subsequently increases the degree of water chemically incorporated into the saltstone matrix." In contrast, DOE contractors indicated that caustic would increase reactivity and decrease flowability. Could DOE please clarify?
6. Could DOE clarify if and why there are significant differences in saltstone versus clean cap grout compressive strength (see SRNL-STI-2010-00515 versus SRR-CWDA-2015-00160). Although the cure time and other factors may differ between different test results, it is unclear to NRC if significantly different compressive strengths should be expected for the two grouts under typical field conditions.
7. NRC staff requested a copy of the UWMQE that includes information on Grade 120 tank grout wet chemistry test, flow test, compressive strength test, bleed test, and heat of hydration charted over time. DOE indicates this information is contained in SRR-CWDA-2015-00088. However, it is unclear that the requested information is recorded in this document. Also, has DOE performed similar evaluation for other reducing tank-closure grouts such as equipment, cooling coil, and clean cap grout?

8. The Tank 12H grout strategy did not discuss disposal of chromate-laden flushwater or a strategy for completely filling of the intact coils in any detail, instead referring to a related work order that was to have been developed. Could DOE clarify if the same strategy was used in Tank 12H which was used in Tank 16H and provide any related work orders for disposal of chromate-laden flushwater?
9. A supplier deviation disposition request (SDDR No. 13307) addressed the two highest bleed water test results, which were 3.3 and 8.9 percent for Tank 16H (SRR-CWDA-2015-00159). Could NRC obtain a copy of SDDR No. 13307 and any associated deviation disposition documents?
10. SRR-LWE-2014-00147 indicates contradictory approaches for Tank 12H: (1) vertical portions of the [ventilation] duct were to be filled after completion of the annulus bulk fill, and (2) to lessen the potential for duct collapse, the vertical section of the ventilation inlet duct will be filled with grout to grade level in parallel with bulk filling of the annulus. Could DOE clarify which approach was used?
11. Tank 12H was the first tank closure work order for which the maximum drop height was increased to greater than 5 ft (1.5 m). Could DOE confirm that the drop height was not, in fact, increased, and that there are now no plans to increase the drop height for tanks yet to be grouted as part of the closure process?