GROUT FORMULATIONS AND PROPERTIES FOR TANK FARM CLOSURE (U)

November 12, 2007

Washington Savannah River Company
Savannah River National Laboratory
Aiken, SC 29808

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GROUT FORMULATIONS AND PROPERTIES FOR TANK FARM CLOSURE (U)

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November 12, 2007
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<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACI</td>
<td>American Concrete Institute</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>C-S-H</td>
<td>Calcium silicate hydrate</td>
</tr>
<tr>
<td>FTF</td>
<td>F-Area Tank Farm</td>
</tr>
<tr>
<td>g</td>
<td>gram</td>
</tr>
<tr>
<td>GSA</td>
<td>General Separations Area</td>
</tr>
<tr>
<td>HLW</td>
<td>High Level Waste</td>
</tr>
<tr>
<td>HRWR</td>
<td>High Range Water Reducer</td>
</tr>
<tr>
<td>HTF</td>
<td>H-Area Tank Farm</td>
</tr>
<tr>
<td>LLW</td>
<td>Low Level Waste</td>
</tr>
<tr>
<td>mL</td>
<td>milliliter</td>
</tr>
<tr>
<td>NIST</td>
<td>National Institute of Standards and Testing</td>
</tr>
<tr>
<td>PA</td>
<td>Performance Assessment</td>
</tr>
<tr>
<td>PIT</td>
<td>Planning and Integration Technology</td>
</tr>
<tr>
<td>PS&amp;E</td>
<td>Process Science and Engineering</td>
</tr>
<tr>
<td>psi</td>
<td>pounds per square inch</td>
</tr>
<tr>
<td>RH</td>
<td>Relative Humidity</td>
</tr>
<tr>
<td>SRIP</td>
<td>Site Regulatory Interface and Planning</td>
</tr>
<tr>
<td>SRNL</td>
<td>Savannah River National Laboratory</td>
</tr>
<tr>
<td>SRS</td>
<td>Savannah River Site</td>
</tr>
<tr>
<td>SSD</td>
<td>Surface Saturated Dry</td>
</tr>
<tr>
<td>VMA</td>
<td>Viscosity Modifying Admixture</td>
</tr>
<tr>
<td>W / C</td>
<td>Water to Cement Ratio</td>
</tr>
<tr>
<td>W / CM</td>
<td>Water to Cementitious Materials Ratio</td>
</tr>
<tr>
<td>WSRC</td>
<td>Washington Savannah River Company</td>
</tr>
<tr>
<td>Wt</td>
<td>Weight</td>
</tr>
<tr>
<td>Wt%</td>
<td>Weight percent</td>
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</table>
1.0 EXECUTIVE SUMMARY

The F-Tank Farm (FTF) and H-Tank Farm (HTF) are located within F-Area in the General Separations Area (GSA) of the Savannah River Site (SRS). The GSA contains the F and H-Area Separations Facilities, the S-Area Defense Waste Processing Facility, the Z-Area Saltstone Facility, and the E-Area Low-Level Waste Disposal Facilities. The FTF includes twenty-two waste tanks which were constructed between 1951 and 1976. The HTF includes 30 waste tanks were constructed between 1953 and 1986.

The Site Regulatory Integration and Planning (SRIP) organization\(^1\) is in the process of preparing the regulatory documentation for closing several of the waste tanks in the FTF and the HTF. The current closure concept for all of the FTF and HTF waste tanks is to fill the majority of each tank with a chemically reducing cementitious grout and then to fill the top of certain tanks with a strong grout to protect against inadvertent intrusion after closure.

The Savannah River National Laboratory (SRNL) was tasked to prepare and test the grout formulations in the 2003 Tank Closure Grout Specification, C-SPP-F-0047, Revision 2 \([1]\) and to produce samples for hydraulic property measurements and for radionuclide distribution determinations \([2]\). SRNL was also tasked to design and test two alternative grout reducing formulations to support the FTF and HTF Performance Assessment (PA) \([2]\). The objective of designing and testing alternative grout mixes was to improve the hydraulic properties (reduce the permeability and porosity) to reduce water and contaminant transport and improve durability. The task scope is provided in the Task Technical and QA Plan \([3]\).

Hydraulic property measurements and radionuclide distribution determinations that will be used in the current Tank Closure PA are presented elsewhere \([4, 5, \text{respectively}]\). As a part of this overall effort, there is now mix design information with testing results for both the reducing grout and the strong grout that supports the F Tank Farm Performance Assessment modeling assumptions. The mix design information will be used to prepare a specification for the procurement of tank fill material for the next tank closures scheduled for 2010-2012.

Two strategies were used to reduce the saturated permeability of grout formulations: 1) lower the water to cement ratio by increasing the amount of cementitious material, and 2) lower the amount of paste by decreasing the volume of paste (cementitious material, water, and air voids). Both strategies were successful in producing flowable grouts with lower permeabilities than the Reducing Grout in the 2003 Tank Closure Specification. Results and conclusions of this testing are summarized below.

- New high range water reducing (HRWR) polycarboxylate admixtures were identified to replace the original HRWR which is no longer available. These materials were equally

\(^{1}\) SRIP was formerly referred to as Planning and Integration Technology (PIT).
or more effective in lowering the water requirement of the grouts as long as they were not pre-mixed\(^2\) with the viscosity modifying admixture (VMA), Kelco-crete.\(^3\) However, mixtures of the three new HRWRs and Kelco-crete did not provide the same water reduction as the original Adva flow-Kelco-crete blend. This problem was not overcome in the testing. Consequently the HRWR was added to the grout during mixing. Kelco-crete was added as the last ingredient along with a small amount of cement or fly ash which helped disperse the gum particles in the wet grout. Addition of Kelco-crete or other VMA must be addressed in full-scale production proportioning and mixing.

- Samples of the 2003 Strong Grout were prepared for hydraulic property, radionuclide partitioning determinations, and physical property measurements. The average saturated permeability coefficient for this grout (Mixes 070025.1 and 070025.2) was 2.1E-08 cm/sec. This value is very good for such a high water to cement ratio. However, compared to typical structural concrete mixes containing 600 lbs of cement per cubic yard and lower w/cm ratios between 0.50 and 0.60, the permeability of the strong grout is low. The compressive strengths of the strong grout met the 2000 psi design criteria after 90 days, but did not provide a large margin. The low strengths of the strong grout are attributed to the high water to cement ratio of this mix, 0.985.

Based on the recent testing, the Strong Grout mix in the 2003 SRS Specification should be redesigned to achieve a more robust mix with a lower w/cm ratio that includes fly ash. Shrinkage can be minimized by using less water and a coarse aggregate or aggregate blend. Alternatives to the specification Strong Grout mix that could be used for tank closure were identified as a part of this effort.

- Samples of the 2003 Reducing Grout were prepared for hydraulic property measurements, radionuclide partitioning determinations, and physical property measurements. This grout developed compressive strength over 180 days in a manner consistent with ternary blends of cement, slag, and fly ash. The ternary blend of cementitious materials\(^4\) reacted within 28 days to achieve an average compressive strength of 1720 psi. Over 500 psi additional strength was gained between 28 and 180 days.

The average saturated permeability coefficient for the Specification Reducing Grout (Mix No. 070027.1 and Mix No. 070027.2) was 3.6E-08 cm/s. The relatively high permeability of this mix can be explained by the low cement plus slag content (385 lbs per cubic yard) and the high water to cementitious materials ratio (w/cm) of 0.658 (actual for sample tested).

\(^2\) Pre-mixing the HRWR and the VMA provided a convenient means of adding and dispersing a small amount of Kelco-crete (about one pound) in each cubic yard of grout.

\(^3\) Kelco-crete is included in the mix design to enhance physical stability of the grout (minimizing segregation) and achieve a robust mix.

\(^4\) Cement (75 lbs), slag (210 lbs), and fly ash (375 lbs)
Reducing grout mixes can be designed with lower permeability based on the results of the alternative reducing grout mixes tested in this study. Modifications should include more cement (200 to 240 compared to 75 lbs per cubic yard), the same amount of slag (210 lbs per cubic yard) unless recommended otherwise by Kaplan and Coates [5], and a lower w/cm ratio. Modifications should include evaluating different aggregate gradings to identify a paste volume that balances the desire to minimize shrinkage but achieve the desired very high flow properties.

- Samples of Alternative Reducing Grout mixes were prepared for hydraulic property measurements, radionuclide partitioning determinations, and physical property measurements. Two series of alternative reducing mixes were evaluated. The amount of cementitious material was increased in the Alternate 1 Reducing Grout Series. Mix 070043 and Mix 070044 contain more cementitious material and less water than the 2003 Specification Reducing Grout.

  The total aggregate surface area was decreased in the Alternate 2 Reducing Grout Series by using 3/8 inch aggregate. Mix 070070 is an example of the second series. All of the alternative grout mixes tested met the placement requirements and exceeded the strength requirements for tank closure grout. All of the alternative mixes had lower permeabilities than the 2003 Specification Reducing Grout. The average saturated permeability coefficients for Mixes 070043, 070044, and 070070 were 1.3 E-08, 8.9E-09, and 6.6E-09 cm/s, respectively. Mix 070070 which utilized both strategies for reducing the permeability had the lowest average coefficient value. These alternative grouts are expected to experience less shrinkage because the w/cm ratios are lower than those of the 2003 Specification Grouts.

- Additional testing by SRNL in FY08 (prior to issuing a revised procurement specification for tank closure fill material) is recommended to demonstrate that further improvements can be made with respect to permeability and shrinkage by optimizing the paste and mortar volumes, and aggregate grading (aggregate surface area and volume).

- A hybrid material and performance specification is recommended for acquisition of the tank closure grouts that will be used in Tanks 18 and 19-F in order to take advantage of supplier experience with flowable concrete and admixtures. A hybrid specification also encourages innovative mix designs, materials selections, and batching techniques that can lower the cost of the tank closure materials.

- If materials with significantly lower saturated permeability coefficients are desired, consider more than a single point placement in the tanks to lessen the flow property requirements. Highly flowable grouts or concretes are on the edge of physical stability where slight variations in water content can result in poor flow or bleed water. Use of VMAs addresses this issue to some extent. However, mix design considerations that decrease shrinkage and thereby decrease permeability also decrease flow and self-leveling.
These results of this effort indicate that the following considerations should be used to design the next generation all-in-one tank closure materials with lower saturated permeability coefficients suitable for mass placements:

- Portland cement and slag cement are required to form a matrix with a low permeability. Materials containing cement and slag have a lower permeability than materials with an equivalent amount of cement only.
- The amount of cement plus slag should be limited to about 450 lbs per cubic yard to limit the heat generated during mass placements. Maintaining 210 lbs of slag per cubic yard allows for up to 240 lbs of cement per cubic yard.
- Fly ash or other pozzolanic or non pozzolanic filler (limestone powder) should be included to make up the “powder” fraction (paste volume) required for flowable grout (concrete).
- For trial mixes containing different proportions of the same ingredients, density (unit weight) is a good indication of porosity and shrinkage. The higher the density the less porous and the lower shrinkage is expected. However, flowable grouts and concretes typically need more paste volume to achieve cohesion than conventional concretes. Aggregate grading to reduce aggregate surface area is the primary control for balancing opposing trends.
2.0 INTRODUCTION

2.1 Objective

This work was requested by J. L. Newman, Site Regulatory Integration and Planning (SRIP) in PIT-TTR-2006-00002 [2]. The complete task scope is provided in the Task Technical and QA Plan [3].

The specific objectives of this task were to:
1) Prepare and evaluate the placement and compressive strength properties of the Tank Closure Reducing Grout and Strong Grout identified in the current WSRC Tank Closure Grout Specification, C-SPP-F-0047, Revision 2 [1].

Re-evaluation of the Reducing Grout and Strong Grout mixes identified in Specification C-SPP-F-0047 Revision 2 were based on materials available 5 to 10 years ago. The chemical admixtures used for adjusting the flow properties have been replaced with alternatives or have been modified. Also, the sources of cement and fly ash have changed.

2) Prepare samples for hydraulic property measurements. Results were reported elsewhere [4].

3) Prepare samples for leach testing. Results were reported elsewhere [5].

4) Design, prepare and test two alternative tank fill materials for consideration in future tank closures. The performance objectives of the alternate grouts were to meet the important attributes listed below and to improve permeability (lower permeability). Samples of the alternative grouts were also prepared for hydraulic property and leach testing by Dixon and Kaplan, respectively [4, 5].

The important attributes of the tank fill materials, with respect to properties that control leaching (permeability and chemistry), were provided by the customer, J. L. Newman, PIT [1]. These attributes are listed below in a general order of priority:
   A. Low water infiltration (conductivity) through the in-place grout, over the long term
   B. High reducing capacity, over the long term
   C. High long term strength of in-place grout
   D. Low long term cracking
   E. Low long term degradation of the in-place grout
   F. Adequate flowability of the grout during placement.

2.2 Background

The FTF and HTF are located in the General Separations Area (GSA) of the Savannah River Site (SRS). The GSA contains the F and H-Area Separations Facilities, the S-Area...
Defense Waste Processing Facility, the Z-Area Saltstone Facility, and the E-Area Low-Level Waste Disposal Facilities. The FTF includes twenty-two waste tanks constructed between 1951 and 1976. It comprises approximately 20 acres and is bounded by SRS coordinates N 76,604.5 to N 77,560.0 and E 52,435.0 to E 53,369.0 (Figure 2-1). The HTF includes thirty waste tanks that were constructed between 1952 and 1986 and comprise approximately 75 acres (Figure 2-2).

The Site Regulatory Integration and Planning (SRIP) organization is in the process of preparing the regulatory documentation for closing the FTF waste tanks. The current closure concept for all of the FTF and HTF waste tanks is to fill the majority of each tank with a chemically reducing cementitious grout and then to fill the top of certain tanks with a strong grout to protect against inadvertent intrusion after closure.

Waste removal operations are currently in progress in F Tank Farm to support closure of the non-compliant tanks in accordance with the Federal Facility Agreement (FFA) closure schedule. Additional mechanical cleaning of Tank 5 and chemical cleaning for both Tanks 5 and 6 are currently scheduled for FY 08. Further heel removal is scheduled for both Tanks 18 and 19 in FY 08.
2.3 Previous SRS Tank Grout Mix Designs

In 1997, two single-shell carbon steel tanks (17-F and 20-F) in the FTF were emptied and filled with grout. Both tanks had a capacity of 1.3 million gallons and were originally used to store low-heat waste. The original concept was to use a high strength reducing grout to encapsulate the residual waste, a Controlled Low-Strength Material (CLSM)\(^5\) for filling the bulk of the tank and a 2000 psi grout as an intruder barrier in the top of the tank. The high strength reducing grout was designed by engineers at the Construction Technology Laboratory, Skokie, IL. Ingredients in the grout mixes used to fill these tanks are listed in Table 2-1 [6].

The original SRS CLSM and 2000 psi grout mixes were modified by SRNL to eliminate bleed water. Initial testing of the Site CLSM and 2000 psi grout indicated that a significant amount of bleed water would be generated in the closed tanks. In early 1997, SRNL and BSRI personnel were requested to modify the site CLSM mix and 2000 psi mix to eliminate the need for removing and disposing of radioactively contaminated liquid from

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\(^5\) CLSM is a cementitious flowable fill that is used as backfill or infill and has soil-like properties. It is self compacting and consequently does not required mechanical compaction to achieve design density. CLSM typically contains sand, fly ash and less than 100 pounds of hydraulic material per cubic yard of fill. Hydraulic cementitious material reacts with water to form insoluble hydrated compounds. Portland cement is the best known hydraulic cement. Slag cement is also hydraulic once it has been activated.
the tanks and to improve uniformity of the fill material (reduce settling and stratification). (Bleed water is not a problem when these materials are used in conventional applications where the water can drain off or evaporate). The resulting modified mixes were referred to as SRS zero bleed flowable fill and SRS zero-bleed 2000 psi grout. These mixes were incorporated into subsequent WSRC site concrete specifications.

In 1998, research was conducted to develop an all-in-one HLW tank fill grout that could be used for both encapsulating the residual waste and bulk fill [7]. The driver for this work was the desire to simplify the production requirements for tank fill material. This work resulted in an all-in-one zero bleed reducing fill/grout mix which is also provided in Table 2-1. This mix was adopted for the Reducing CLSM, Mix No OPCEXE-X-P-0-BS, listed in the current SRS Specification C-SPP-F-00047, Revision 2 [1].

2.4 Current Reducing Grout Requirements

The attributes for tank closure grouts provided by J. Newman, SRIP, were combined with and interpreted in terms of engineering properties to derive requirements that could be used for designing and evaluating alternative test mixes. The requirements and bases for the requirements and considerations are summarized in Table 2-2.
Table 2-1. SRS Tank Closure Grout Mix Designs from the 1990’s [6].

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>SRS Reducing Grout</th>
<th>SRS Zero-Bleed Flowable Fill</th>
<th>SRS Zero-Bleed 2000 psi Grout</th>
<th>SRS All-In One Zero Bleed Reducing Fill/Grout***</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement Type I/II</td>
<td>1353</td>
<td>150</td>
<td>550</td>
<td>75</td>
</tr>
<tr>
<td>(lbs / cu yd)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Slag Grade 100</td>
<td>209</td>
<td>---</td>
<td>---</td>
<td>210</td>
</tr>
<tr>
<td>(lbs / cu yd)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fly Ash, Class F</td>
<td>---</td>
<td>500</td>
<td>---</td>
<td>375</td>
</tr>
<tr>
<td>(lbs / cu yd)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Silica Fume</td>
<td>90</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>(lbs / cu yd)</td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Quartz Sand ASTM C-33</td>
<td>1625 (masonry sand)</td>
<td>2300 (concrete sand)</td>
<td>2285 (concrete sand)</td>
<td>2300 (concrete sand)</td>
</tr>
<tr>
<td>(lbs / cu yd)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water (gallons / cu yd)</td>
<td>86.4 (721)</td>
<td>63 (526)</td>
<td>65 (542)</td>
<td>60 (500)</td>
</tr>
<tr>
<td>(lbs / cu yd)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HRWR (fl oz / cu yd)</td>
<td>250</td>
<td>90 (Adva Flow)</td>
<td>140 (Adva Flow)</td>
<td>90 (Adva Flow)</td>
</tr>
<tr>
<td>Viscosifier Kelco-crete®</td>
<td>---</td>
<td>275</td>
<td>275</td>
<td>275</td>
</tr>
<tr>
<td>(grams / cu yd)**</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Set Retarder (fl oz / cu yd)</td>
<td>150</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Sodium Thiosulfate (lbs / cu yd)</td>
<td>2.1</td>
<td>---</td>
<td>---</td>
<td>2.1 (optional)</td>
</tr>
</tbody>
</table>

* HRWR, i.e., Adva flow was used in these mixes. However it was premixed with a viscosifier, Kelco-crete® rather than added as an individual component.

** Advaflow and Kelco-crete® were premixed prior to incorporation in the zero-bleed mixes.

*** This mix was adopted for the Reducing CLSM, Mix No OPCEXE-X-P-0-BS, listed in the current SRS Specification C-SPP-F-00047, Revision 2 [1].
Table 2-2. Tank Closure Reducing Grout (Reducing Material Fill) Requirements.

<table>
<thead>
<tr>
<th>SRIP Attributes [2]</th>
<th>Physical Property</th>
<th>Engineering Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low water infiltration (conductivity) through the in-place grout, over the long term</td>
<td>Saturated Permeability less than current tank fill grout</td>
<td>1. Saturated Permeability ( K_{sat} \leq 2E-08 \text{ cm/sec} )</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2. Durable</td>
</tr>
<tr>
<td>High reducing capacity, over the long term</td>
<td>High long-term negative Eh. Current approach is to use the same amount of slag cement as used in earlier grout mix designs</td>
<td>At least 210 lbs of slag per cubic yard of reducing grout</td>
</tr>
<tr>
<td>High long-term strength of in-place grout</td>
<td>High long-term strength at any time is not required. The PA identifies a 2000 psi intruder barrier. This is also the minimum strength required for low permeability reducing grout.</td>
<td>At least 2500 psi at 90 days to meet 2000 psi req. for strong grout</td>
</tr>
<tr>
<td>Low long-term cracking</td>
<td>Minimize the potential for cracking:</td>
<td>1. Use as much dimensionally stable sand and gravel as possible</td>
</tr>
<tr>
<td></td>
<td>1. Negligible early stage shrinkage</td>
<td>2. Cracking mechanisms due to material incompatibility, phase changes, and corrosives were addressed elsewhere [9]. Continue with same materials unless new testing and research indicate potential for expansion.</td>
</tr>
<tr>
<td></td>
<td>2. Negligible chemical incompatibility of materials</td>
<td>3. Overburden loading is not an issue.</td>
</tr>
<tr>
<td></td>
<td>3. Negligible susceptibility to environmental corrosives</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4. No cracking as the result of overburden loading</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5. Seismic loading not considered</td>
<td></td>
</tr>
<tr>
<td>Low long-term degradation of the in-place grout</td>
<td>Durable: Perform design function over 100s to 1000s of years</td>
<td>1. Chemical degradation is addressed elsewhere [10]. Use equivalent or more portland cement (pH) and slag cement (Eh).</td>
</tr>
<tr>
<td></td>
<td>1. Negligible cracking due to internal expansive reactions and external forces</td>
<td>2. Degradations mechanisms addressed elsewhere [9]. Continue with same materials.</td>
</tr>
<tr>
<td></td>
<td>2. Maintain chemical alkalinity and reducing chemistry</td>
<td></td>
</tr>
<tr>
<td>Adequate flowability of the grout during placement</td>
<td>Flow 35 feet in a tank with a 70 feet diameter from a central discharge point from a 2-10 foot free drop</td>
<td>Grout flow &gt;11 inches per ASTM D 6103 Slump flow &gt; 25 inches ASTM C 1161</td>
</tr>
<tr>
<td>Other Considerations</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Production</td>
<td>1. Suitable for on-site continuous or central mixer batch plant using locally available aggregate and simplify admixture additions if possible</td>
<td>HRWR and VMA compatibility to enable addition as a slurry to support auger mixing.</td>
</tr>
<tr>
<td></td>
<td>2. Production Rate of at least 600 cubic yards/day</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3. Pumpable 1500 feet</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2. Set time &lt;24 hours</td>
<td>2. Set time &lt;24 hours</td>
</tr>
<tr>
<td></td>
<td>3. Minimal bleed water (no bleed water is desirable) and segregation</td>
<td>3. Zero bleed after 24 hours</td>
</tr>
<tr>
<td></td>
<td>4. Air entrainment not required for below grade placement</td>
<td>4. No air entrainment</td>
</tr>
<tr>
<td></td>
<td>5. Cure under moist conditions</td>
<td>5. High unit weight, low air content</td>
</tr>
<tr>
<td></td>
<td>6. Low shrinkage</td>
<td>6. Low paste content, moist cure, zero bleed</td>
</tr>
<tr>
<td>Cured Properties</td>
<td>1. 90 day strength &gt;2000 psi</td>
<td>1. 90 day strength &gt;2000 psi</td>
</tr>
<tr>
<td></td>
<td>2. Permeability &lt; 2E-08 cm/sec (strong grout measurements [4])</td>
<td>2. Permeability &lt; 2E-08 cm/sec (strong grout measurements [4])</td>
</tr>
<tr>
<td></td>
<td>3. Low Shrinkage</td>
<td>3. Low Shrinkage</td>
</tr>
<tr>
<td></td>
<td>4. Heat of hydration suitable for mass pours</td>
<td>4. Heat of hydration suitable for mass pours</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5. Portland cement + slag cement less than about 450 lbs/cubic yard.</td>
</tr>
</tbody>
</table>
3.0 EXPERIMENTAL METHODOLOGY

3.1 Ingredients

Grout mixes tested in this study were prepared with bulk materials obtained from local suppliers and chemical admixtures that are distributed nationwide. The Type I/II cement was manufactured by LaFarge, Inc. at their cement plant in Harleyville SC. The Grade 100 slag cement was manufactured by Holcim, Inc., Birmingham AL, and the fly ash was obtained from Boral Material Technology, Inc. SRS process water was used as the mixing water. The ingredients are listed in Table 3-1. The aggregate properties are listed in Table 3-2.

Table 3-1. Ingredients Used to Prepare Samples of the FTF Closure Grouts.

<table>
<thead>
<tr>
<th>Material</th>
<th>Specification</th>
<th>Supplier / Address</th>
<th>Phone Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland cement (Type I/II)</td>
<td>ASTM C 150</td>
<td>LaFarge, Cement Harleyville, SC obtained from Lafarge Ready Mix Augusta GA</td>
<td>706-823-4471</td>
</tr>
<tr>
<td>Slag cement (Grade 100)</td>
<td>ASTM C 987</td>
<td>Holcim, Inc. 3235 Satellite Blvd. Duluth GA 30096</td>
<td>800-292-4355</td>
</tr>
<tr>
<td>Fly ash (Class F)</td>
<td>ASTM C618</td>
<td>Boral Material Technology Inc. Atlanta GA</td>
<td>800-241-4943</td>
</tr>
<tr>
<td>Concrete sand</td>
<td>ASTM C-33</td>
<td>Foster Dixieanna Clearwater SC obtained from Lafarge Ready Mix Augusta GA</td>
<td>706-823-4471</td>
</tr>
<tr>
<td>No. 8 stone 3/8 inch gravel (granite)</td>
<td>ASTM C-33</td>
<td>Rinker Dogwood Quarry Appling GA</td>
<td>706-541-0187</td>
</tr>
<tr>
<td>HRWR</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Adva 380</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Advaflex</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sika ViscoCrete 2100</td>
<td></td>
<td>Sika Corporation</td>
<td>717 821 3721</td>
</tr>
<tr>
<td>Hydration Stabilizer</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Viscosifier</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kelco-Crete®</td>
<td></td>
<td>CP Kelco 835 Aero Dr. San Diego CA 92123</td>
<td>858-292-4900</td>
</tr>
<tr>
<td>Sodium Thiosulfate</td>
<td></td>
<td>Reagent Chemical</td>
<td>800-343-0660</td>
</tr>
<tr>
<td>SRS process water</td>
<td></td>
<td>SRS</td>
<td></td>
</tr>
</tbody>
</table>
Table 3-2. Size Distribution of the Sand and No. 8 Stone.

<table>
<thead>
<tr>
<th>Property</th>
<th>Concrete Sand</th>
<th>No. 8 Aggregate (3/8 inch)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk Unit Weight (lb/ft³)</td>
<td>85 @ 1.6 wt. % SSD*</td>
<td>93 @ 0.6 wt. % SSD*</td>
</tr>
<tr>
<td>Specific Gravity (particle)</td>
<td>2.65</td>
<td>2.56</td>
</tr>
<tr>
<td>Composition</td>
<td>Quartz</td>
<td>Granite</td>
</tr>
<tr>
<td>Moisture Content (as received)*</td>
<td>0.7-5.5 wt. %</td>
<td>~0</td>
</tr>
</tbody>
</table>

Particle size Distribution

<table>
<thead>
<tr>
<th>Size</th>
<th>Wt. % Passing</th>
<th>Cum. Wt. % Retained</th>
<th>Wt. % Passing</th>
<th>Cum. Wt. % Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>½ inch (12.5 mm)</td>
<td>100</td>
<td>0</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>3/8 inch sieve</td>
<td>100</td>
<td>0</td>
<td>99</td>
<td>1</td>
</tr>
<tr>
<td>#4 sieve (4.75mm)</td>
<td>99</td>
<td>1</td>
<td>29</td>
<td>71</td>
</tr>
<tr>
<td>#8 sieve (2.36 mm)</td>
<td>96</td>
<td>4</td>
<td>4</td>
<td>96</td>
</tr>
<tr>
<td>#16 sieve (1.18 mm)</td>
<td>80</td>
<td>20</td>
<td>1</td>
<td>99</td>
</tr>
<tr>
<td>#30 sieve (600 um)</td>
<td>41</td>
<td>59</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>#50 sieve (300 um)</td>
<td>13</td>
<td>87</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>#100 sieve (150 um)</td>
<td>4</td>
<td>96</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>Fineness Modulus</td>
<td>--</td>
<td>2.67</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>

* Moisture content varied as a function of the storage time and conditions as determined by ASTM C-128 and ASTM C-566 (total moisture). SSD = Surface Saturated Dry.
+ Percentage passing through each sieve as determined by ASTM C136.

3.2 Sample Preparation and Test Methods

Sample preparation and testing were performed in the SRS Civil Engineering Test Laboratory which is operated by Washington Quality and Testing. The laboratory is located in N-Area. Samples were prepared according to ASTM C 192 and cured in a constant temperature (73°F) curing room at 100 % relative humidity. A Hobart planetary mixer equipped with a paddle was used to prepare the grouts which contained sand as the only aggregate (see Figure 3-1). The batch size was approximately 0.5 cubic feet. A three cubic foot concrete mixer was used to prepare the alternative test mixes that contained 3/8 inch No. 8 aggregate (see Figure 3-2).

Laboratory samples of the Specification reducing and strong Grouts and the alternative sand only reducing grouts were prepared by placing all of the sand in the mixing bowl. The mixer was turned on at low speed and a portion of the mixing water, followed by all of the fly ash. If necessary, more water was added. The slag and cement were finally added to the mixing bowl. Water was added as needed up to the predetermined amount. The admixtures were added last. The mixing time was approximately three minutes after all of the ingredients were added. For mixes containing No. 8 aggregate, all of the stone was added prior to turning on the mixer. The sand was added followed by a portion of the water and all of the fly ash. The mixing sequence for the remainder of the ingredients was as described for the sand only mixes. The mixing time for the alternative reducing grout mixes with No. 8 aggregate was as follows: After all of the ingredients were added, the batch was mixed for 5
minutes and then allowed to rest for 5 minutes. A sample for slump-flow was collected. After the test was completed, the material was returned to the mixer and mixed for another 15 minutes. The batch was allowed to rest for another 5 minutes prior to measuring fresh properties. After the flow was measured, the remaining material was used to cast samples for strength, permeability and bleed water.

Three by six inch cylinders were cast for the specification reducing grout and strong grout as well as the first series of alternative reducing grout trial mixes (Alternative Reducing Grout 1 Series). These samples were used for compressive strength and permeability measurements using a flexible wall permeameter (ASTM D 5084). One by twelve inch tubes were cast for permeability measurements determined by the steady state centrifugation unsaturated flow apparatus (SSC-UFA) method (ASTM D6527). Permeability results and other hydraulic and geotechnical test results are presented elsewhere [4].

Four by eight inch cylinders and three by six inch cylinders were cast for the second series of reducing grout trial mixes (Alternative Reducing Grout 2 Series). The larger samples were used for compressive strength measurements and the smaller samples were supplied for the permeability measurements. Twelve inch long tubes were also cast for SSC-UFA permeability measurements.

Test methods are summarized in Table 3-3. More detailed descriptions of the tests methods are provided elsewhere [7, 8]. An additional test was used for measuring flow, ASTM C 1161. The property measured is referred to as slump-flow and the method was recently adapted for evaluating placement of self consolidating and flowable concrete. Examples of the samples and testing are illustrated in Figures 3-1 to 3-5.

<table>
<thead>
<tr>
<th>Properties</th>
<th>ASTM Standard Methods</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Fresh Properties</strong></td>
<td></td>
</tr>
<tr>
<td>Flow</td>
<td>D-6103</td>
</tr>
<tr>
<td>Concrete Spread</td>
<td>C-1161</td>
</tr>
<tr>
<td>J-Ring Passing Ability</td>
<td>C-1621</td>
</tr>
<tr>
<td>Set Time</td>
<td>C-403</td>
</tr>
<tr>
<td>Bleed Water</td>
<td>C-232</td>
</tr>
<tr>
<td>Segregation*</td>
<td>Visual</td>
</tr>
<tr>
<td>Unit Weight</td>
<td>C-138</td>
</tr>
<tr>
<td>Air Content</td>
<td>C-231</td>
</tr>
<tr>
<td><strong>Cured Properties</strong></td>
<td></td>
</tr>
<tr>
<td>Compressive Strength</td>
<td>C-39</td>
</tr>
<tr>
<td>Shrinkage</td>
<td>C-490</td>
</tr>
<tr>
<td>Heat of Hydration</td>
<td>SRNL Adiabatic Calorimeter</td>
</tr>
</tbody>
</table>

* Segregation was measured by visual examination of a washed “green sample. See Figure 3-5. ASTM C-1621 includes a method for quantifying segregation. The necessary test equipment has been acquired and will be used for future testing.
Figure 3-1. Hobart Mixer and Example of Reducing Grout after Mixing.

Figure 3-2. Three Cubic-Foot Concrete Mixer.

Figure 3-3. Examples of (a) ASTM D-6103 Flow (11 inch flow) and (b) ASTM C-1161 Slump-Flow (19 inches) Measurements.
Figure 3-4. Air Content Test Apparatus.

Figure 3-5. Example of Visual Examination for Segregation (a) no segregation (b) significant segregation.

Figure 3-6. Shrinkage Specimens in Molds (a) and in a Length Comparator (b).
3.3 Specification Reducing Grout and Strong Grout Compositions

The purpose of preparing the specification reducing and strong grouts (SRS Specification C-SPP-F-00047, Revision 2, 2003) was to:

1. Produce samples for hydraulic property testing, and
2. Confirm that currently available high range water reducers (HRWR) and viscosity modifying admixtures (VMAs) can be blended to produce a pumpable slurry to simplify metering and addition in a continuous auger batch plant.\(^6\)

Adva flow\(^{TM}\), which was used in the Tank 17 and 20-F closures and which is identified in SRS Specification C-SPP-F-00047, Revision 2, 2003 [1], has been replaced by other chemically similar products.\(^7\) Consequently other chemically similar HRWRs were obtained and tested. Adva 380 and Adva flex were recommended by W. R. Grace technical support. Sika Viscocrete was also tested. Kelco-crete is still available and was tested.\(^8\)

Since the original HRWR and VMA were no longer available, testing had to be performed to obtain dosages for the currently available materials. Several initial mixes were required to determine how to add and dose the current products. Initially additions of Kelco-crete were made by adding this material to a small amount of cement or fly ash to disperse the particles in the grout mixture. The HRWRs were added as liquid.

Ingredients and proportions of the reducing grout, OPDEXE-X-P-0-BS, and strong grout, B 2000-X-0-0-BS samples prepared for hydraulic and physical property testing are provided in Tables 3-4 and 3-5, respectively. Two 0.5 cubic foot batches of each mix were prepared. Lab mix numbers for the strong grout were 070025-1 (batch 1) and 070025-2 (batch 2). Lab mix numbers for the reducing grout were 070027-1 (batch 1) and 070027-2 (batch 2).

---

\(^6\) The Adva flow-Kelco-crete admixture system was developed by SRNL and BSRI personnel for Tank 17 and 20 closures to simplify metering into a central or continuous mixer. Kelco-crete is a gum which hydrates upon contact with water to form a sticky paste. Because it is added in small quantities (less than a kilogram per cubic yard), addition as a slurry which contains a carrier that prevents immediate hydration is the most reliable way of dispersing it in a wet system.

Other options for adding viscosifiers include: 1) pre-hydrating them with water and adding the resulting very viscous material to a central mixer or 2) mixing the powder with other powders, such as fly ash, to assure dispersion in the final product.

\(^7\) Adva flow an early W.R. Grace carboxylated acrylic ester copolymer (polycarboxylate) HRWR.

\(^8\) The currently available material may have been modified over the last 10 years.
Table 3-4. Ingredients in the Specification Strong Grout Mix Used for Property Measurements [1].

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>FTF Strong Grout B 2000-X-0-0-BS (1 cubic yard batch)</th>
<th>FTF Strong Grout B 2000-X-0-0-BS (0.5 cubic foot batch)</th>
<th>Strong Grout Lab No. 070025-1 Lab No. 070025-2 (0.5 cubic foot batch)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement Type I/II (lbs)</td>
<td>550</td>
<td>10.18</td>
<td>10.18</td>
</tr>
<tr>
<td>Quartz Sand ASTM C-33 (lbs)</td>
<td>2285</td>
<td>42.31</td>
<td>44.99*</td>
</tr>
<tr>
<td>Water (gallons)</td>
<td>65</td>
<td>1.2</td>
<td>(7.36)*</td>
</tr>
<tr>
<td>HRWR (fl oz) Carboxylated acrylic ester copolmers (polycarboxylates)</td>
<td>90</td>
<td>1.6</td>
<td>1.7</td>
</tr>
<tr>
<td>Adva flow</td>
<td></td>
<td>Adva 380**</td>
<td>Adva 380**</td>
</tr>
<tr>
<td>Viscosifier Kelco-crete® (grams)</td>
<td>275</td>
<td>5.1</td>
<td>2.0 ***</td>
</tr>
<tr>
<td>Water / Cementitious Materials Ratio (Cementitious materials include cement, slag and fly ash)</td>
<td>0.985 (Assuming maximum water is used)</td>
<td>0.984 (Assuming maximum water is used)</td>
<td>0.723 (Calculated for actual water used. Some water was held back because flow was achieved with less water.)</td>
</tr>
</tbody>
</table>

* Weights adjusted for water sorbed on the sand (sand weigh increased, mixing water weight decreased accordingly). In addition, 0.10 lb of water was held back because it was not needed to achieve flow
** HRWR, i.e., Adva 380 was used in the mixes prepared for this testing because Adva flow is no longer marketed by W. R. Grace.
*** Kelco-crete was mixed with a few grams of cement and added as a powder as the last ingredient. Less Kelco-crete was required to achieve flow than was used in 1997 and identified in the SRS tank closure grout specification.
Table 3-5. Ingredients in the Specification Reducing Grout Used for Property Measurements [1].

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Reducing Grout (OPDEXE-X-P-0-BS) (1 cubic yard batch)</th>
<th>Reducing Grout (OPDEXE-X-P-0-BS) (1 cubic yard batch)</th>
<th>Reducing Grout Lab No. 070027-1 Lab No. 070027-2 (0.5 cubic yard batch)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement Type I/I (lbs)</td>
<td>75</td>
<td>1.39</td>
<td>1.39</td>
</tr>
<tr>
<td>Slag Cement Grade 100 (lbs)</td>
<td>210</td>
<td>3.89</td>
<td>3.89</td>
</tr>
<tr>
<td>Fly Ash Class F (lbs)</td>
<td>375</td>
<td>6.94</td>
<td>6.94</td>
</tr>
<tr>
<td>Quartz Sand ASTM C-33 (lbs)</td>
<td>2300</td>
<td>42.59</td>
<td>44.33*</td>
</tr>
<tr>
<td>Water (gallons)</td>
<td>60</td>
<td>1.11</td>
<td>(8.04)*</td>
</tr>
<tr>
<td>(lb)</td>
<td>(500)</td>
<td>(9.26)</td>
<td></td>
</tr>
<tr>
<td>Sodium Thiosulfate (lbs)</td>
<td>2.1</td>
<td>0.04</td>
<td>17.2 grams</td>
</tr>
<tr>
<td>HRWR (fl oz)</td>
<td>90</td>
<td>1.6</td>
<td>1.6</td>
</tr>
<tr>
<td>Carboxylated acrylic ester copolmers (polycarboxylates)</td>
<td></td>
<td>Adva flow</td>
<td>Adva 380*</td>
</tr>
<tr>
<td>Adva flow</td>
<td></td>
<td>Adva 380*</td>
<td></td>
</tr>
<tr>
<td>Viscosifier (grams) # Kelco-crete*</td>
<td>275</td>
<td>5.1</td>
<td>3</td>
</tr>
<tr>
<td>Water / Cementitious Materials Ratio (Cementitious materials include cement, slag and fly ash)</td>
<td>0.758</td>
<td>0.758</td>
<td>0.658</td>
</tr>
<tr>
<td>(Assuming maximum water is used)</td>
<td></td>
<td>--</td>
<td>(Calculated for actual water used)</td>
</tr>
</tbody>
</table>

* Weights adjusted for water sorbed on the sand (sand weigh increased, mixing water weight decreased accordingly). In addition 1.22 lbs of water were held back because flow was achieved with less than the maximum amount of water.

** HRWR, i.e., Adva 380 was used in the mixes prepared for this testing because Adva flow is no longer marketed by W. R. Grace.

# Kelco-crete was mixed with a few grams of fly ash and added as a powder.
3.4 Approach for Designing Alternate Mixes

The purpose of designing an alternative tank closure reducing grout was to obtain a material with a lower permeability than the currently specified reducing grout. Two strategies for reducing permeability were investigated:

1. Reduce the water to cementitious materials ratio.
2. Increase the volume of the impermeable aggregate while reducing the surface area of the aggregate by modifying the aggregate size and size distribution.  

A low water to cementitious material (w/cm) was used in one set of alternative trial mixes (Alternative 1 Series). Both strategies were combined in another set of alternative trial mixes (Alternative 2 Series). Both strategies were initially tested on mixes without slag. The resulting no-slag mixes can be used as alternatives to the tank closure strong grout.

ACI 237R-07 and ACI 211.1 were used as a guide for proportioning the alternative alternate tank closure mixes [10 and 11]. General features of the mix designs are listed below:

- In general, all of the alternative mixes contained more cementitious materials than is typical of regular concrete (600 to 1200 lbs / cu yd compared to 500 to 800 lbs / cu yd).
- Slag was included in the alternative reducing grout mixes at the same higher proportion as in the Specification Mix.
- Fly ash was substituted for at least 50% of the cementitious materials to control the heat of hydration.
- The fine and coarse aggregates were included as approximately equal proportions for the Alternative 2 series of mixes with the 3/8 inch No. 8 aggregate being a few weight percent greater than the sand.
- Locally available concrete sand and No. 8 aggregate were used. (Aggregate blends for optimizing size grading were considered by not pursued for this study.)
- Carboxylated acrylic ester co-polymers (polycarboxylates) HRWRs were used to achieve very high flow at low water to cementitious materials ratios. Adva 380, Adva flex, and Sika Viscocrete were tested for compatibility and relative effect.
- Kelco-crete, a viscosity modifying admixture, was used to minimize bleeding and segregation in all of the mixes. (Optimal aggregate blends were not used).
- No air entrainment admixture was used.
- A hydration stabilizer, Recover, was used in most of the mixes. (This had the effect of allowing more time for settling under static conditions and accentuated any tendency for segregation. The desire was to produce a robust uniform flowable mix.)

Mixes were screened on the basis of fresh properties. The Alternative concepts were initially tested on cement-sand mixes that incorporated fly ash. Such mixes can be considered as alternatives for the Strong Grout. The ingredients and proportions in the Strong Grout Alternatives are provided in Tables 3-6 and 3-7. The ingredients and proportions of the Reducing Grout alternative mixes are provided in Tables 3-8 and 3-9.

---

9 The total aggregate surface area determines the water demand. The amount of aggregate determines the amount of paste required to fill a unit volume.
Table 3-6. Alternative Strong Grout Mixes (Low-Water to Cementitious Materials).

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Strong grout Alternative Lab No. 070028.1</th>
<th>Strong grout Alternative Lab No. 070035</th>
<th>Strong grout Alternative Lab No. 070036</th>
<th>Strong grout Alternative Lab No. 070037</th>
<th>Strong grout Alternative Lab No. 070049</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement Type I/II (lbs / cu yd)</td>
<td>600</td>
<td>400</td>
<td>500</td>
<td>500</td>
<td>500</td>
</tr>
<tr>
<td>Fly Ash Class F (lbs / cu yd)</td>
<td>600</td>
<td>740</td>
<td>670</td>
<td>670</td>
<td>670</td>
</tr>
<tr>
<td>Quartz Sand ASTM C-33 (Concrete sand) (lbs / cu yd)</td>
<td>1743</td>
<td>1743</td>
<td>1743</td>
<td>1743</td>
<td>1743</td>
</tr>
<tr>
<td>No. 8 Gravel</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Water (gal/cu yd) (lbs / cu yd)</td>
<td>58 (483)</td>
<td>60 (503)</td>
<td>56 (468)</td>
<td>62 (515)</td>
<td>62 (516)</td>
</tr>
<tr>
<td>Hydration Stabilizer Recover (fl oz / cu yd)</td>
<td>36</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>HRWR** (fl oz / cu yd)</td>
<td>120</td>
<td>120</td>
<td>120</td>
<td>35</td>
<td>45</td>
</tr>
<tr>
<td>Viscosifier Kelco-crete® (grams / cu yd)</td>
<td>250</td>
<td>120</td>
<td>120</td>
<td>120</td>
<td>162</td>
</tr>
<tr>
<td>Water / Cement Material Ratio</td>
<td>0.403</td>
<td>0.441</td>
<td>0.400</td>
<td>0.440</td>
<td>0.441</td>
</tr>
</tbody>
</table>
### Table 3-7. Alternative Strong Grout Mixes (Low Water to Cementitious Materials plus No. 8 Aggregate).

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Strong grout Alternative Lab No. 070038</th>
<th>Strong grout Alternative Lab No. 070039</th>
<th>Strong grout Alternative Lab No. 070040</th>
<th>Strong grout Alternative Lab No. 070041</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement Type I/II (lbs / cu yd)</td>
<td>450</td>
<td>450</td>
<td>450</td>
<td>450</td>
</tr>
<tr>
<td>Fly Ash Class F (lbs / cu yd)</td>
<td>450</td>
<td>530</td>
<td>600</td>
<td>600</td>
</tr>
<tr>
<td>Quartz Sand ASTM C 33 (Concrete sand) (lbs / cu yd)</td>
<td>1258</td>
<td>1176</td>
<td>1140</td>
<td>1058</td>
</tr>
<tr>
<td>No. 8 Aggregate ASTM C 33 (lbs / cu yd)</td>
<td>1250</td>
<td>1170</td>
<td>1131</td>
<td>1049</td>
</tr>
<tr>
<td>Water (gal / cu yd)</td>
<td>46.6</td>
<td>52.7</td>
<td>55</td>
<td>50</td>
</tr>
<tr>
<td>Water (lbs / cu yd)</td>
<td>(388)</td>
<td>(439)</td>
<td>(458)</td>
<td>(418)</td>
</tr>
<tr>
<td>Hydration Stabilizer Recover (fl oz / cu yd)</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>HRWR** (fl oz / cu yd)</td>
<td>35</td>
<td>42</td>
<td>42</td>
<td>35</td>
</tr>
<tr>
<td>Viscosifier Kelco-crete® (grams / cu yd)</td>
<td>162</td>
<td>216</td>
<td>216</td>
<td>120</td>
</tr>
<tr>
<td>Water / Cement Material Ratio</td>
<td>0.431</td>
<td>0.448</td>
<td>0.434</td>
<td>0.398</td>
</tr>
<tr>
<td>(Cementitious materials include cement, slag and fly ash)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
### Table 3-8. Alternative 1 Reducing Grout Mixes (Low Water to Cementitious Materials).

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Alternative 1b Reducing Grout Lab No. 070043</th>
<th>Alternative 1a Reducing Grout Lab No. 070044</th>
<th>Alternative Reducing Grout Lab No. 070045</th>
<th>Alternative Reducing Grout Lab No. 070067</th>
<th>Alternative Reducing Grout Lab No. 070068</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement Type I/II (lbs / cu yd)</td>
<td>300</td>
<td>185</td>
<td>242</td>
<td>242</td>
<td>300</td>
</tr>
<tr>
<td>Slag Cement (Grade 100) (lbs / cu yd)</td>
<td>310</td>
<td>260</td>
<td>285</td>
<td>285</td>
<td>310</td>
</tr>
<tr>
<td>Fly Ash Class F (lbs / cu yd)</td>
<td>800</td>
<td>580</td>
<td>690</td>
<td>690</td>
<td>800</td>
</tr>
<tr>
<td>Quartz Sand ASTM C-33 (Concrete sand) (lbs / cu yd)</td>
<td>1420</td>
<td>1885</td>
<td>1741</td>
<td>1741</td>
<td>1470</td>
</tr>
<tr>
<td>Water (gal / cu yd)</td>
<td>63 (525)</td>
<td>59.7 (497)</td>
<td>61 (509)</td>
<td>57.7 (481)</td>
<td>55.6 (463)</td>
</tr>
<tr>
<td>Sodium Thiosulfate (lbs / cu yd)</td>
<td>2.1</td>
<td>2.1</td>
<td>2.1</td>
<td>2.1</td>
<td>2.1</td>
</tr>
<tr>
<td>Hydration Stabilizer Recover (fl oz / cu yd)</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>HRWR (fl oz / cu yd)</td>
<td>54 Sika Viscocrete</td>
<td>54 Sika Viscocrete</td>
<td>54 Sika Viscocrete</td>
<td>61 Adva flex</td>
<td>70 Adva flex</td>
</tr>
<tr>
<td>Viscosifier Kelco-crete® (grams / cu yd)</td>
<td>216</td>
<td>216</td>
<td>216</td>
<td>216</td>
<td>216</td>
</tr>
<tr>
<td>Water / Cement</td>
<td>1.75</td>
<td>2.69</td>
<td>2.1</td>
<td>1.988</td>
<td>1.543</td>
</tr>
<tr>
<td>Water / Cement + Slag</td>
<td>0.86</td>
<td>1.112</td>
<td>0.966</td>
<td>0.913</td>
<td>0.759</td>
</tr>
<tr>
<td>Water / Cement Material Ratio</td>
<td>0.372</td>
<td>0.485</td>
<td>0.418</td>
<td>0.395</td>
<td>0.328</td>
</tr>
</tbody>
</table>

Shading indicates mixes that were tested for permeability.
### Table 3-9. Alternative 2 Reducing Grout Mixes (Low Water to Cementitious Materials Plus No. 8 Aggregate).

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement Type I/II (lbs / cu yd)</td>
<td>185</td>
<td>185</td>
<td>225</td>
<td>225</td>
<td>225</td>
</tr>
<tr>
<td>Slag Cement (Grade 100) (lbs / cu yd)</td>
<td>260</td>
<td>260</td>
<td>213</td>
<td>213</td>
<td>213</td>
</tr>
<tr>
<td>Fly Ash Class F (lbs / cu yd)</td>
<td>580</td>
<td>850</td>
<td>450</td>
<td>530</td>
<td>600</td>
</tr>
<tr>
<td>Quartz Sand ASTM C-33 (Concrete sand) (lbs / cu yd)</td>
<td>942</td>
<td>942</td>
<td>1258</td>
<td>1176</td>
<td>1103</td>
</tr>
<tr>
<td>No. 8 Aggregate ASTM C 33 (3/8 inch granite gravel) (lbs / cu yd)</td>
<td>946</td>
<td>946</td>
<td>1250</td>
<td>1170</td>
<td>990</td>
</tr>
<tr>
<td>Water (gal/cu yd)</td>
<td>51 (424)</td>
<td>61 (506)</td>
<td>51 (423)</td>
<td>54 (450)</td>
<td>57 (475)</td>
</tr>
<tr>
<td>Sodium Thiosulfate (lbs / cu yd)</td>
<td>2.1</td>
<td>2.1</td>
<td>2.1</td>
<td>2.1</td>
<td>2.1</td>
</tr>
<tr>
<td>Hydration Stabilizer Recover (fl oz / cu yd)</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>HRWR (fl oz / cu yd)</td>
<td>51 Adva flex</td>
<td>54 Adva flex</td>
<td>62 Adva flex</td>
<td>97 Adva flex</td>
<td>104 Adva flex</td>
</tr>
<tr>
<td>Viscosifier Kelco-crete® (grams / cu yd)</td>
<td>216</td>
<td>216</td>
<td>216</td>
<td>216</td>
<td>216</td>
</tr>
<tr>
<td>Water / Cement Ratio</td>
<td>2.292</td>
<td>2.735</td>
<td>1.88</td>
<td>2.00</td>
<td>2.11</td>
</tr>
<tr>
<td>Water / Cement + Slag</td>
<td>0.952</td>
<td>1.137</td>
<td>0.966</td>
<td>1.027</td>
<td>1.08</td>
</tr>
<tr>
<td>Water / Cement Material Ratio (Cementitious materials include cement, slag and fly ash)</td>
<td>0.414</td>
<td>0.391</td>
<td>0.476</td>
<td>0.465</td>
<td>0.458</td>
</tr>
</tbody>
</table>

Shading indicates mixes that were tested for permeability.
4.0 RESULTS

4.1 Admixture Changes

The work flow and schedule required that samples of the reducing grout and strong grout identified in SRS Specification C-SPP-F-00047, Revision 2, 2003, be prepared as the first task in this study. This required that alternative admixtures be quickly identified and tested. Adva flow, the HRWR listed in the specification, is no longer available. Adva 380, a material of the same chemical type from the same supplier, was recommended by the manufacturer and used to prepare initial test mixes with compositions in the SRS specification.

A problem with the recommended replacement material was encountered in the initial testing. The Adva 380 mixed with Kelco-crete to form a slurry, but when this slurry was added to the grout as the last ingredient, the grout entrained air and the expected water reducing effect was realized. The mixes did not flow.

An alternative method of adding the Adva 380 and Kelco-crete was tested and found to result in grouts with acceptable fresh properties. The Adva 380 was added to the grout along with a portion of the water. The Kelco-crete was added as the last ingredient as a powder dispersed in 20 grams of cement or fly ash as the last ingredient. This method of addition was used for all of the mixes reported in this study.

The dosages of the HRWRs and VMA were also modified relative to SRS Specification C-SPP-F-00047, Revision 2, 2003. In general, lower dosages of the currently available HRWR and Kelco-crete were needed to achieve acceptable flow properties.

Two other HRWRs of the same chemical type were also tested in some of the alternative mixes, Adva flex and Sika Viscocrete. Both were added as powders along with a small amount of cement or fly ash and were effective water reducers. Viscocrete entrained the least amount of air.

4.2 SRS Strong Grout

Samples of the specification strong grout were made in two different batches using the same ingredients and proportions Lab No. 070025-1 and 070025-2. These samples were used for compressive strength and permeability measurements. The strength results are very low (<2100 psi at 180 days) for a mix containing 600 lbs of cement per cubic yard but can be explained by the high water to cement ratio of this mix, w/c = 0.723. (The allowable w/c for this mix per the specification is 0.985.)

---

10 Combining Adva flow and Kelco-crete to make a single liquid component simplified addition of the Kelco-crete to the grout during full-scale production during closure of Tanks 17 and 20-F.

11 This testing was performed prior to making the specification reducing grout and strong grout and mix numbers were not recorded.
Samples from the first batch barely met the criteria for 2000 psi strength at 90 days. Samples from the second batch, Mix 070025-2, did not meet the strength criteria at any age tested up to 180 days. See Table 4-1. In addition, the second batch segregated as indicated by 0.9 volume % bleed water measured after 24 hours. Although this bleed water was reabsorbed within 48 hours, its presence indicates settling which is expressed as vertical channeling and horizontal stratification. Such a big difference in strength between two batches with the same ingredients and proportions is difficult to explain. Lack of experience with the new admixtures may be in part responsible for this anomaly.

The average permeability value for samples No. 070025-1 and 070025-2 was 2.1E-08 cm/s [4]. This value is higher than expected based on literature data for cement paste or concrete with a w/cm of 0.723 [11].

4.3 SRS Reducing Grout Properties

Samples of the specification reducing grout were made in two different batches using the same ingredients and proportions Lab No. 070027-1 and 070027-2. These samples were used for compressive strength and permeability measurements. Samples from both batches of the reducing grout had similar strengths at 28 and 90 days (990 and compared to 1000 psi at 28 days and 2380 compared to 2220 at 90 days for batches 1 and 2 respectively). See Table 4-1. These strength results are very good considering the mix contained only 75 lbs of cement and 210 lbs of slag cement per cubic yard.

The water to cementitious material ratio for the mix as it was prepared was 0.658. (The maximum allowable w/cm for the reducing grout per the current specification is 0.758). The average permeability value for samples No. 070027-1 and 070027-2 was 3.6E-08 cm/s [4]. This value is higher than expected based on literature data for cement paste or concrete with a w/cm of 0.658 [11] and can be attributed to the low cement and slag content of the mix.
Table 4-1. Properties of the SRS Specification Strong Grout and Reducing Grout.

<table>
<thead>
<tr>
<th>Fresh Properties</th>
<th>Mix ID</th>
<th>Strength (psi)</th>
<th>Water to Cementitious Material Ratio**</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Measured</td>
<td>Average*</td>
</tr>
<tr>
<td>Flow D-6103 (inches)</td>
<td></td>
<td>11</td>
<td>11</td>
</tr>
<tr>
<td>Bleed water (vol. %)</td>
<td></td>
<td>0</td>
<td>0.9</td>
</tr>
<tr>
<td>Set time (hr)</td>
<td></td>
<td>&lt;18</td>
<td>&lt;18</td>
</tr>
<tr>
<td>Air Content (vol. %)</td>
<td></td>
<td>3</td>
<td>Not Measured</td>
</tr>
<tr>
<td>Casting temp. (°F)</td>
<td></td>
<td>72</td>
<td>72</td>
</tr>
<tr>
<td>Cohesive</td>
<td></td>
<td>Yes</td>
<td>Marginal</td>
</tr>
<tr>
<td>Unit Weight (lbs / ft³)</td>
<td></td>
<td>130.7</td>
<td>Not Measured</td>
</tr>
<tr>
<td>Water to Cementitious Material Ratio**</td>
<td></td>
<td>0.723</td>
<td>0.723</td>
</tr>
<tr>
<td>Cured Properties</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Strength (psi)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14 days</td>
<td></td>
<td>1520</td>
<td>1520</td>
</tr>
<tr>
<td>28 days</td>
<td></td>
<td>1520</td>
<td>1520</td>
</tr>
<tr>
<td>56 days</td>
<td></td>
<td>1980</td>
<td>2040</td>
</tr>
<tr>
<td>90 days</td>
<td></td>
<td>2070</td>
<td>2090</td>
</tr>
<tr>
<td>180 days</td>
<td></td>
<td>2110</td>
<td>2080</td>
</tr>
<tr>
<td>Permeability (saturated) [4]</td>
<td></td>
<td>Average = 2.1E-08 cm/s</td>
<td>Average = 3.6E-08 cm/s</td>
</tr>
</tbody>
</table>

* Averaged per ASTM C-150. ** w/cm = water ÷ (cement + slag + fly ash).
4.4 Alternative 1 Strong Grout Properties (Sand only)

A series of alternative strong grout mixes (Alternative 1 Strong Grouts) were prepared to test HRWR admixtures other than Adva 380. Flowable self-consolidating concrete mix design criteria were applied to these mixes. Fly ash was used in these mixes because they require more “powder” (cementitious materials and pozzolans) than is used in conventional mixes. (The strong grout already contained 600 lbs of cement per cubic yard.) The w/cm was maintained in the range of 0.40 to 0.44 with the intent of producing flowable grouts with lower permeabilities than the specification strong grout. Fresh and cured properties of the Alternative 1 Strong Grout mixes are presented in Table 4-2.

All of the mixes were adjusted with HRWR and kelco-crete to achieve excellent flow properties and no bleed. The mixes were cohesive and demonstrated flow creep in the ASTM C 1611 and C1621 tests. The dosages of Sika viscocrete (Mix 070037) and Adva flex (070049) required to achieve flow were less than half the dose of Adva 380 Mixes 070028.1, 070035, and 070036). The mixes containing Adva 380 were unacceptable because they entrained too much air.

The 28 day compressive strengths of all of the alternative 1 strong grout mixes were lower than expected at 28 days. However, after 90 days and 180 days the strengths were appropriate for the cement content and w/cm. No explanation for this variability is clear at this time. Permeability was not measured on any of these mixes. However all of these mixes are expected to have lower permeabilities than the Specification strong grout based on w/cm ratios.

4.5 Alternative 2 Strong Grout Properties (3/8 inch Aggregate)

A second series of alternative strong grout mixes (Alternative 2 Strong Grouts) were prepared with the objective of improving the aggregate size grading and thereby lowering the amount of cementitious material required to achieve a flowable grout with a low w/cm ratio. No. 8 crushed granite aggregate (3/8 inch) was included in the mix.

Flowable self-consolidating concrete mix design criteria were applied to these mixes. The amount of cement was reduced in these mixes relative to the Alternate 1 strong grout mixes so it would be more in line with the cement content of mass pour concrete mix designs. Fly ash was added to meet the need for “powder.” The w/cm ratios were maintained in the range of 0.40 to 0.45 with the intent of producing flowable grouts with lower permeabilities than the specification strong grout. Fresh and cured properties of the Alternative 2 Strong Grout mixes are presented in Table 4-3.

All of these mixes were adjusted with Sika Viscocrete and Kelco-crete. Three of the mixes, 070038, 070039 and 070041 had excellent flow properties and did not bleed. Mix 070040 had bleed water. All of the mixes had 28 day compressive strength in excess of 2000 psi and 90 day compressive strength in excess of 4000 psi. Permeability was not measured on any of these mixes. However all of these mixes are expected to have lower permeabilities than the Specification strong grout based on w/cm ratios.
Table 4-2. Properties of Alternative 1 Strong Grout Mixes (Low Water to Cementitious Materials).

<table>
<thead>
<tr>
<th>Fresh Properties</th>
<th>Mix ID</th>
<th>Mix ID</th>
<th>Mix ID</th>
<th>Mix ID</th>
<th>Mix ID</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Alternative 1 Strong Grout Lab No. 070028.1</td>
<td>Alternative 1 Strong Grout Lab No. 070035</td>
<td>Alternative 1 Strong Grout Lab No. 070036</td>
<td>Alternative 1 Strong Grout Lab No. 070037</td>
<td>Alternative 1 Strong Grout Lab No. 070049</td>
</tr>
<tr>
<td>Flow D-6103 (inches)</td>
<td>14</td>
<td>15</td>
<td>15.5</td>
<td>14</td>
<td>13</td>
</tr>
<tr>
<td>(after 5 Min. mixing, 5 min rest)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Flow D-6103 (inches)</td>
<td>14.5</td>
<td>17</td>
<td>15.5</td>
<td>14</td>
<td>12.5</td>
</tr>
<tr>
<td>(after 15 Min. mixing, 5 min rest)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bleed water (vol. %)</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Set time (hr)</td>
<td>&lt;18</td>
<td>~20</td>
<td>~20</td>
<td>~19</td>
<td>&lt;18</td>
</tr>
<tr>
<td>Air Content (vol. %)</td>
<td>4</td>
<td>8</td>
<td>12</td>
<td>4.5</td>
<td>3</td>
</tr>
<tr>
<td>Cohesive</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>3es</td>
</tr>
<tr>
<td>Unit Weight (lbs / ft³)</td>
<td>123.9</td>
<td>119.8</td>
<td>116.8</td>
<td>126.3</td>
<td>126.2</td>
</tr>
<tr>
<td>Yield</td>
<td>0.96</td>
<td>1.02</td>
<td>1.07</td>
<td>1.00</td>
<td>Not Measured</td>
</tr>
<tr>
<td>Water to Cementitious Material Ratio**</td>
<td>0.403</td>
<td>0.441</td>
<td>0.400</td>
<td>0.440</td>
<td>0.441</td>
</tr>
</tbody>
</table>

Cured Properties

<table>
<thead>
<tr>
<th>Strength (psi)</th>
<th>Measured</th>
<th>Average*</th>
<th>Measured</th>
<th>Average*</th>
<th>Measured</th>
<th>Average*</th>
<th>Measured</th>
<th>Average*</th>
<th>Measured</th>
<th>Average*</th>
</tr>
</thead>
<tbody>
<tr>
<td>14 days</td>
<td>1540</td>
<td>1540</td>
<td>1370</td>
<td>1370</td>
<td>1010</td>
<td>1010</td>
<td>2110</td>
<td>2110</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>28 days</td>
<td>2930</td>
<td>2930</td>
<td>1420</td>
<td>1500</td>
<td>1460</td>
<td>1770</td>
<td>1650</td>
<td>1710</td>
<td>2650</td>
<td>2540</td>
</tr>
<tr>
<td>56 days</td>
<td>3640</td>
<td>3610</td>
<td>2830</td>
<td>2840</td>
<td>2840</td>
<td>3470</td>
<td>3440</td>
<td>3460</td>
<td>4270</td>
<td>4450</td>
</tr>
<tr>
<td>90 days</td>
<td>4280</td>
<td>4370</td>
<td>4320</td>
<td>4260</td>
<td>4320</td>
<td>4990</td>
<td>4890</td>
<td>4940</td>
<td>5900</td>
<td>6320</td>
</tr>
<tr>
<td>180 days</td>
<td>5340</td>
<td>5500</td>
<td>5420</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>4650</td>
<td>4730</td>
</tr>
</tbody>
</table>

* Averaged per ASTM C-150. ** w/cm = water ÷ (cement + slag + fly ash).
Table 4-3. Properties of Alternative 2 Strong Grout Mixes (Low Water to Cementitious Materials Plus No. 8 Aggregate).

<table>
<thead>
<tr>
<th>Fresh Properties</th>
<th>Mix ID</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Alternative 2</td>
</tr>
<tr>
<td></td>
<td>Strong Grout</td>
</tr>
<tr>
<td></td>
<td>Lab No. 070038</td>
</tr>
<tr>
<td></td>
<td>Alternative 2</td>
</tr>
<tr>
<td></td>
<td>Strong Grout</td>
</tr>
<tr>
<td></td>
<td>Lab No. 070039</td>
</tr>
<tr>
<td></td>
<td>Alternative 2</td>
</tr>
<tr>
<td></td>
<td>Strong Grout</td>
</tr>
<tr>
<td></td>
<td>Lab No. 070040</td>
</tr>
<tr>
<td></td>
<td>Alternative 2</td>
</tr>
<tr>
<td></td>
<td>Strong Grout</td>
</tr>
<tr>
<td></td>
<td>Lab No. 070041</td>
</tr>
<tr>
<td>Flow D-6103 (inches)</td>
<td>13</td>
</tr>
<tr>
<td>(after 5 Min. mixing, 5 min rest)</td>
<td>13</td>
</tr>
<tr>
<td></td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>12</td>
</tr>
<tr>
<td>Flow D-6103 (inches)</td>
<td>13</td>
</tr>
<tr>
<td>(after 15 Min. mixing, 5 min rest)</td>
<td>14</td>
</tr>
<tr>
<td></td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>12</td>
</tr>
<tr>
<td>Bleed water (vol. %)</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>2.5</td>
</tr>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td>Set time (hr)</td>
<td>&lt;18</td>
</tr>
<tr>
<td></td>
<td>&lt;18</td>
</tr>
<tr>
<td></td>
<td>&lt;18</td>
</tr>
<tr>
<td></td>
<td>&lt;18</td>
</tr>
<tr>
<td>Air Content (vol. %)</td>
<td>0.8</td>
</tr>
<tr>
<td></td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>3</td>
</tr>
<tr>
<td>Cohesive</td>
<td>Marginal</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
</tr>
<tr>
<td></td>
<td>No</td>
</tr>
<tr>
<td></td>
<td>Yes</td>
</tr>
<tr>
<td>Unit Weight (lbs / ft³)</td>
<td>139.9</td>
</tr>
<tr>
<td></td>
<td>138.0</td>
</tr>
<tr>
<td></td>
<td>134.9</td>
</tr>
<tr>
<td></td>
<td>126.2</td>
</tr>
<tr>
<td>Yield</td>
<td>1.01</td>
</tr>
<tr>
<td></td>
<td>1.00</td>
</tr>
<tr>
<td></td>
<td>1.03</td>
</tr>
<tr>
<td></td>
<td>Not measured</td>
</tr>
<tr>
<td>Water to Cementitious Material Ratio**</td>
<td>0.431</td>
</tr>
<tr>
<td></td>
<td>0.448</td>
</tr>
<tr>
<td></td>
<td>0.434</td>
</tr>
<tr>
<td></td>
<td>0.398</td>
</tr>
<tr>
<td>Cured Properties</td>
<td></td>
</tr>
<tr>
<td>Strength (psi)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Measured</td>
</tr>
<tr>
<td>14 days</td>
<td>1880</td>
</tr>
<tr>
<td>28 days</td>
<td>3260</td>
</tr>
<tr>
<td>56 days</td>
<td>--</td>
</tr>
<tr>
<td>90 days</td>
<td>5480</td>
</tr>
<tr>
<td>180 days</td>
<td>5280</td>
</tr>
</tbody>
</table>

* Averaged per ASTM C-150. ** w/cm = water ÷ (cement + slag + fly ash).
4.6 Alternative 1 Reducing Grout Properties (Sand Only)

Alternative reducing grout mixes (Alternative 1 Reducing Grouts) with lower w/cm ratios than SRS specification reducing grout were prepared to generate samples for permeability measurements. The objective was to design fill materials with lower permeabilities than the current materials. These mixes contained at least ~210 to 260 lbs of slag (same as in the SRS reducing grout) and more cement (185 to 300 lb / cu yd) and fly ash (580 to 800 lbs / cu yd) than the reducing grout in the SRS specification (75 lbs cement and 375 lbs fly ash / cu yd). The w/cm ratios for these alternative grouts ranged between 0.328 and 0.485. Fresh and cured properties of the Alternative 1 Reducing Grout mixes are presented in Table 4-4.

All of the mixes were adjusted with HRWR and kelco-crete to achieve excellent flow properties and no bleed. Sika viscocrete was used in Mixes 070043, 070044, and 070045. Adva flex was used in Mixes 070067 and 070068. The mixes were cohesive and demonstrated flow creep in the ASTM D 6103 test.

The 28 day compressive strengths of all of the alternative 1 reducing grout mixes were very high and ranged from 3560 to 5160 psi at 28 days to 5810 to 8180 psi at 180 days. The compressive strengths of these mixes were surprisingly high given the low cement plus slag content. Mix 070044 contained only 185 lbs of cement and 260 lbs of slag per cubic yard but reached 5300 after 90 days (lowest compressive strength mix of the series). All of these mixes have strengths well above the strength requirement for tank closure reducing grout.

Permeability measurements were made on two of the mixes from this series of alternate reducing grouts [4]. The average permeability value for Mix 070043, referred to as Alternative 1B, was 1.3E-08 cm/s. The average permeability value for Mix 070044, referred to as mix 1A, was 8.9E-9 cm/s.

Mix 070043 was expected to have a lower permeability than Mix 070044 based on the amount of cement and slag in each mix and the w/cm ratios. No explanation can be made for difference between measured and expected values. All of the mixes in this series are expected to have lower permeabilities than the specification strong grout based on w/cm ratios.
Table 4-4. Properties of Alternative 1 Reducing Grout Mixes (Low Water to Cementitious Materials).

<table>
<thead>
<tr>
<th>Fresh Properties</th>
<th>Mix ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow D-6103 (inches) (after 5 Min. mixing, 5 min rest)</td>
<td>17</td>
</tr>
<tr>
<td>Flow D-6103 (inches) (after 15 Min. mixing, 5 min rest)</td>
<td>17.5</td>
</tr>
<tr>
<td>Bleed water (vol. %)</td>
<td>0</td>
</tr>
<tr>
<td>Set time (hr)</td>
<td>&lt;18</td>
</tr>
<tr>
<td>Air Content (vol. %)</td>
<td>1.2</td>
</tr>
<tr>
<td>Cohesive</td>
<td>Yes</td>
</tr>
<tr>
<td>Unit Weight (lbs / ft³)</td>
<td>128.0</td>
</tr>
<tr>
<td>Yield</td>
<td>0.98</td>
</tr>
<tr>
<td>Water to Cementitious Material Ratio**</td>
<td>0.372</td>
</tr>
</tbody>
</table>

Cured Properties

| Permeability [4] (cm/s) | 1.3E-08 | 8.9E-09 |

<table>
<thead>
<tr>
<th>Strength (psi)</th>
<th>Measured</th>
<th>Average*</th>
</tr>
</thead>
<tbody>
<tr>
<td>14 days</td>
<td>2420</td>
<td>2420</td>
</tr>
<tr>
<td>28 days</td>
<td>5080</td>
<td>5160</td>
</tr>
<tr>
<td>90 days</td>
<td>7360</td>
<td>7460</td>
</tr>
<tr>
<td>180 days</td>
<td>8320</td>
<td>8180</td>
</tr>
</tbody>
</table>

* Averaged per ASTM C-150. ** w/cm = water ÷ (cement + slag + fly ash).
4.7 Alternative 2 Reducing Grout Properties (3/8 inch Aggregate)

Alternative reducing grout mixes (Alternative 2 Reducing Grouts) containing No.8 aggregate (3/8 inch) and with lower w/cm ratios than SRS specification reducing grout were prepared to generate samples for permeability measurements. The objective was to design fill materials with lower permeabilities than the current materials. These mixes contained at least ~210 to 260 lbs of slag (same as in the SRS reducing grout) and more cement (185 to 225 lb / cu yd) and fly ash (450 to 850 lbs / cu yd) than the reducing grout in the SRS specification (75 lbs cement and 375 lbs fly ash / cu yd). The w/cm ratios for these alternative grouts ranged between 0.391 and 0.476. Fresh and cured properties of the Alternative 2 Reducing Grout mixes are presented in Table 4-5.

All of the mixes were adjusted with Advaflex (HRWR) and kelco-crete to achieve excellent flow properties and no bleed. The mixes were cohesive and demonstrated flow creep in the ASTM C1611 and C1621 tests.

The 28 day compressive strengths of all of the alternative 2 reducing grout mixes were very high and ranged from 3380 to 4640 psi at 28 days to 6260 to 4840 psi at 90 days. The compressive strengths of these mixes were surprisingly high given the low cement plus slag content. Mix 070070 contained only 185 lbs of cement and 260 lbs of slag per cubic yard but reached 4840 after 90 days (lowest compressive strength mix of the series). All of these mixes have strengths well above the strength requirement for tank closure reducing grout.

Permeability measurements were made on one of the mixes from this series of alternate reducing grouts [4]. The average permeability value for Mix 070070, referred to as Alternative 2, was 6.6E-09 cm/s. This grout had the lowest permeability of any of the samples measured to date. All of the mixes in this series are expected to have lower permeabilities than the specification strong grout based on w/cm ratios.
<table>
<thead>
<tr>
<th>Fresh Properties</th>
<th>Mix ID</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flow D-6103 (inches) (after 5 Min. mixing, 5 min rest)</td>
<td>12</td>
</tr>
<tr>
<td>Flow D-6103 (inches) (after 15 Min. mixing, 5 min rest)</td>
<td>13</td>
</tr>
<tr>
<td>Spread C1611 (inches) (after 25 minutes)</td>
<td>31</td>
</tr>
<tr>
<td>Passing Ability J-Ring C 1621 (inches) (after 25 minutes)</td>
<td>31</td>
</tr>
<tr>
<td>Bleed water (vol. %)</td>
<td>0</td>
</tr>
<tr>
<td>Set time (hr)</td>
<td>15</td>
</tr>
<tr>
<td>Air Content (vol. %)</td>
<td>0.5</td>
</tr>
<tr>
<td>Cohesive</td>
<td>Yes</td>
</tr>
<tr>
<td>Unit Weight (lbs / ft³)</td>
<td>137.5</td>
</tr>
<tr>
<td>Yield</td>
<td>--</td>
</tr>
<tr>
<td>Water to Cementitious Material Ratio**</td>
<td>0.414</td>
</tr>
<tr>
<td>Cured Properties</td>
<td></td>
</tr>
<tr>
<td>Permeability [4] (cm/s)</td>
<td>Measured</td>
</tr>
<tr>
<td>14 days</td>
<td>--</td>
</tr>
<tr>
<td>28 days</td>
<td>3330</td>
</tr>
<tr>
<td>90 days</td>
<td>5480</td>
</tr>
<tr>
<td>180 days</td>
<td>57700</td>
</tr>
</tbody>
</table>

* Averaged per ASTM C-150. ** w/cm = water ÷ (cement + slag + fly ash).
5.0 DISCUSSION

5.1 Current Strong Grout and Reducing Grout Mix Designs

Samples of the specification strong grout and reducing grout were prepared and evaluated for flow and other fresh properties, compressive strength and permeability. The HRWR and VAM admixtures available today perform differently than the materials used in the late 1990's when the mix designs for the strong grout and reducing grout were developed.

Adva 380 was initially substituted for Adva flow. The Adva 380 resulted in a significant amount of air entrainment in the mixes. Two other polycarboxylate-type HRWR were tested, Adva flex and Sika Viscocrete. Blends of the HRWRs and Kelco-crete were effective in reducing the water as was the blend used in the Tank 17 and 20-F grouts. Consequently, in the test mixes prepared for this study, blends were not used. Each admixture was added separately. The Kelco-crete was added as the last ingredient with a small amount of cement or fly ash to achieve dispersion in the grout.

The compressive strengths of the Strong Grout samples, prepared according to the 2003 Specification, barely met the design criteria even after 90 days. The low strengths of the strong grout are attributed to the high water to cement ratio of this mix, 0.985. The average saturated permeability coefficient for this grout (Mixes 070025.1 and 070025.2) was 2.1E-08 cm/sec. The Strong Grout permeability is high (more permeable) compared to typical structural concretes containing 600 lbs of cement per cubic yard and lower w/cm ratios, typically 0.50 to 0.60. See Figure 5-1.

![Figure 5-1. Influence of w/c Ratio on Permeability of Cement Paste and Concrete [12].](image)

Lack of experience in using these new admixtures and admixture addition sequencing may have been responsible for inconsistent compressive strength results between the two batches of each mix prepared for cured property testing.

The 2003 Specification Reducing Grout samples developed compressive strength over 180 days in a manner consistent with ternary blends containing cement, slag, and fly ash. The combined 385 lbs of cement (75 lbs) and slag (210 lbs) reacted within 28 days to achieve an average compressive strength of 1720 psi. Over 500 psi additional strength was gained between 28 and 180 days. (Both batches had similar compressive strengths for samples tested as a function of curing time.)

The saturated permeability coefficient of the Specification Reducing Grout (Mix No. 070027.1 and 070027.2) was 3.6E-08 cm/s which is slightly higher than the strong grout. The relatively high permeability of this mix can be explained by the low cement plus slag content of this mix, 385 lbs per cubic yard and the high w/cm of 0.658 (actual for sample tested).

5.2 Alternative Grout Mix Designs

The original reducing grout placed in Tanks 17 and 20-F was a very high strength cement-slag-silica fume mix with a w/cm ratio of 0.436. This material was designed by CTL, Skolkie IL (See Table 2-1). At the time Tanks 17 and 20-F were filled, the only cured property requirement for the fill material was a compressive strength of >500 psi at 28 days. During the interim 10 years since closing Tanks 17 and 20-F, the concept of using an All-In-One material to provide the functions of both the reducing grout and the fill grout was adopted. Consequently, a procurement specification for the All-In-One grout developed in the late 1990’s was written.

Recent PA modeling calculations indicated that the saturated permeability coefficient is an important design criterion in the overall release predictions. Consequently, scoping studies to identify options for alternative tank closure mix designs were investigated.

Several competing factors must be balanced in the design of a low permeability, flowable grout suitable for mass pour placements. Flowable grout and concrete mixes generally require a higher paste (grout) or mortar volume (concrete) than non-flowable, pumpable grouts and concrete to promote flow. However, grouts and concretes with a high fraction of paste are more susceptible to shrinkage. Shrinkage results in cracking in the matrix material itself as well as in the structural element.

12 The design w/cm ratio for the reducing grout is even higher, 0.758. Some water was held back at the time the test specimens were prepared.

13 The Reducing Grout used to close Tanks 17 and 20-F was not highly flowable. Multiple entry points in the top of the tank were required to distribute the grout over the bottom of the tanks.
The paste volume is the fraction of the volume of the cementitious materials plus the volume of water plus the entrained air. The paste volume can be increased by increasing the amount of water and air as was done with the 2003 Specification Strong and Reducing Grouts which have maximum allowable \( w/cm \) ratios of 0.985 and 0.758, respectively. The approach used for the Specification Strong and Reducing grouts is not used for structural mix designs.\(^{14}\)

Two alternative approaches for increasing the paste (mortar) fraction in flowble mixes were tested in this study. The first approach involved increasing the amount of cementitious materials. The second approach also included decreasing the aggregate surface area and thereby the water demand by including 3/8 inch aggregate plus sand.\(^{15}\) Both approaches were initially tested on mixes without slag and the resulting mixes are referred to as Alternate 1 and Alternate 2 Series of Strong Grouts. Permeability data was not obtained on any of the alternate strong grout samples but are expected to lower than the value for the strong grout mix in the 2003 SRS Specification.

Mix 070043 and Mix 070044 (Alternate 1 Reducing Grout Series) contained more cementitious material and less water than the 2003 Specification Reducing Grout. Mix 070070 (Alternate 2 Reducing Grout Series) contained more cementitious material plus 3/8 inch aggregate. In addition, the fraction of the paste volume made up of “powder” was higher for the alternative reducing grouts compared to the specification reducing grout.

All of the alternative reducing grout mixes tested met the placement requirements and exceeded the strength requirements for tank closure grout. All of the alternative mixes had lower permeabilities than the 2003 Specification Reducing Grout. The average saturated permeability coefficients for Mixes 070043, 070044, and 070070 were 1.3 E-08, 8.9E-09, and 6.6E-09 cm/s. Mix 070070 which utilized both strategies for reducing the permeability had the lowest average coefficient value.

The HRW reducer was added to reduce the water required to achieve the high flow properties. The VMA was used to prevent segregation by providing robustness to these mixes which are required to have very high flows (self-leveling). Alternative mixes included a polycarboxylate HRWR, and Kelco-crete, a VMA. The Adva 380 was initially substituted for Adva flow. The Adva 380 resulted in a significant amount of air entrainment in the mixes. Two other polycarboxylate-type HRWR were tested, Adva flex and Sika Viscocrete. Each required unique proportioning. The lowest dosages were obtained with Sika Viscocrete. The resulting grouts had the least amount of entrained air.

\(^{14}\) At the time these tank fill mixes were designed, the requirements did not address permeability or other hydraulic properties. These mixes were designed as zero bleed Controlled Low Strength (fill) Materials (CLSM). The functional requirements were related to the ability to fill the tanks rather than on hydraulic properties.

\(^{15}\) The slurry properties (fresh properties) of flowable grouts and concretes are also more sensitive to aggregate grading than conventional materials.
A small amount of a hydration stabilizer (set retarder) was also included in the mix to accentuate the tendency for settlement under static conditions and thereby identify slurries with marginal physical stability.

Coarse and fine aggregate gradations were not optimized in the alternative mixes even though the 3/8 inch aggregate was known to be gap graded. The objective was to demonstrate that lower permeability flowable grouts (concrete) could be designed using readily available material. (Use of VMA is one method of minimizing the negative consequences of aggregates that were not optimized.)
6.0 CONCLUSIONS and RECOMMENDATIONS

1. Alternative admixture identification for the Strong Grout and Reducing Grout in SRS Specification C-SPP-F-00047, Revision 2, 2003:

Samples of the tank fill grouts identified in SRS Specification C-SPP-F-00047, Revision 2, 2003 were prepared for permeability testing. In the process, new HRWR admixtures of the same chemical type were identified because the product used 10 years ago is no longer available.

Three new admixtures tested were more effective water reducers for a given dose than the original Adva flow product. However, the new admixtures lost their water reducing effect when pre-mixed with Kelco-crete, a VMA. (Addition of Kelco-crete to the grout produced for closing Tanks 17 and 20-F was simplified by pre-mixing the Kelco-crete and HRWR and metering the slurry into the mix. Consequently, for the 2003 Strong Grout and Reducing Grout mixes, accurate Kelco-crete metering (about 1 lb per ~3200 lbs of grout) is an issue that will need to be addressed by the grout supplier for full-scale production.

In this testing, the HRWR was added as a liquid and the Kelco crete was added as a powder mixed with a small amount of cement or fly ash to achieve dispersion in the grout. Kelco-crete was added as the final ingredient.

2. SRS Specification Strong Grout samples prepared for hydraulic property, radionuclide partitioning determinations and physical property measurements:

Samples of the Strong Grout identified in the 2003 SRS Specification C-SPP-F-00047, Revision 2 were prepared and provided for hydraulic property measurements and radionuclide partitioning determinations. A detailed discussion of the test methods and results are presented elsewhere [4, 5, respectively]. Samples for fresh property measurements and compressive strength were also prepared.

The average saturated permeability coefficient for this grout (Mixes 070025.1 and 070025.2) was 2.1E-08 cm/sec. This value is good for such a high water to cement ratio. However, compared to typical structural concrete mixes containing 600 lbs of cement per cubic yard and lower w/cm ratios between 0.50 and 0.60, the permeability of the tank strong grout is low. The average compressive strength of the strong grout samples were barely met the 2000 psi design criteria even after 90 days. The low strengths of the strong grout are attributed to the high water to cement ratio of this mix, 0.985.16

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16 Cementitious materials with such high w/cm ratios are very susceptible to drying shrinkage (especially small samples). Drying shrinkage in such materials is expressed as nano cracks throughout the material in addition to micro and macro cracks through the structure. Samples are expected to be very sensitive handling conditions.
Based on the recent testing, the 2003 Strong Grout mix should be redesigned to achieve a more robust mix with a lower w/cm ratio (less water) that includes fly ash. Alternative mixes that meet the current design criteria were identified.

3. SRS Specification Reducing Grout samples prepared for hydraulic property, radionuclide partitioning determinations and physical property measurements:

Samples of the Reducing Grout identified in the 2003 SRS Specification C-SPP-F-00047, Revision 2 were prepared and provided for hydraulic property measurements and radionuclide partitioning determinations. A detailed discussion of the test methods and results are presented elsewhere [4, 5, respectively]. Samples for fresh property measurements and compressive strength were also prepared.

The average saturated permeability coefficient of the Reducing Grout (Mix No. 070027.1 and Mix No. 070027.2) was 3.6E-08 cm/s which is slightly higher than the Strong Grout. The relatively high permeability of this mix can be explained by the low cement plus slag content, 385 lbs per cubic yard and the high w / cm of 0.658 (actual for sample tested).17

The reducing grout samples developed compressive strength over 180 days in a manner consistent with ternary blends containing cement, slag, and fly ash. The average strength after 28 days was 1720 psi. Over 500 psi additional strength was gained between 28 and 180 days.

Based on the recent testing and if it is determined to be needed to reduce the permeability of the tank closure fill materials, the Reducing Grout mix could be modified or replaced as follows.18

- A modified mix should contain more cement (200 to 240 compared to 75 lbs per cubic yard), the same amount of slag as in the current Reducing Grout (210 lbs per cubic yard) unless recommended otherwise by Kaplan and Coates [5], a lower w/cm ratio.

- The mix design strategies tested in this study should be applied to an alternative mix.

4. Alternative Reducing Grout samples for hydraulic property, radionuclide partitioning determinations and physical property measurements:

Two series of alternative reducing mixes were formulated and tested.

- The w/cm ratios were decreased in the Alternate 1 Reducing Grout Series by increasing amount of cementitious material. The amount of cement was also increased relative the reducing grout mix in the 2003 specification. Mix 070043 and Mix 070044 are examples of the first series.

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17 This mix contained only 75 lbs of cement per cubic yard and only 210 lbs of slag per cubic yard. Low permeability mixes require more hydraulic material (cement or cement plus slag) per unit volume.

18 Simple changes in the mix design for the Reducing Grout in the 2003 specification are expected to result in lower permeability materials. The alternative grout mix strategies have been demonstrated to result in materials with lower permeabilities.
The surface area of the aggregate was reduced in the Alternate 2 Reducing Grout Series by replacing about half of the sand with 3/8 inch aggregate. Mix 070070 is an example of the second series. This mix also had a lower w/cm ratio.

All of the alternative grout mixes tested met the placement requirements and exceeded the strength requirements for tank closure grout. All of the alternative mixes tested had lower permeabilities than the 2003 Specification Reducing Grout. The average saturated permeability coefficients for Mixes 070043, 070044, and 070070 were 1.3 E-08, 8.9E-09, and 6.6E-09 cm/s, respectively. Mix 070070 which utilized both strategies for reducing the permeability had the lowest average coefficient value.

5. Low permeability flowable grout / concrete mix designs:

Several competing factors must be balanced in the design of a low permeability, flowable grout or concrete suitable for SRS tank closure mass pour placements (assuming an on-site continuous or central mixer batch plant. These requirements include:

- Highly flowable material
- No bleed water
- Low permeability
- Low heat of hydration for mass pour application
- Low w/cm ratio
- Set time that can be adjusted to minimize cold joints assuming daily pours.
- Production rate of at least 600 cubic yards per day with

Additional testing is recommended to demonstrate that further improvements can be made with respect to permeability by optimizing the paste and mortar volumes and aggregate grading by preparing and testing mixes containing:

- Approximately 200 to 240 lbs of cement and 210 lbs of slag per cubic yard.
- No. 7 aggregate in addition to No. 8 aggregate
- ¾ inch aggregate or special other aggregate blends
- Mortar sand and concrete sand mixtures.

6. Revised Specification for Tank Closure Fill Materials:

A hybrid material and performance specification should be considered for acquisition of the tank closure grout in order to take advantage of supplier experience with flowable concrete and admixtures. A hybrid specification also encourages innovative mix designs and batching techniques that can lower the cost of the tank closure materials.
7.0 REFERENCES


DISTRIBUTION:

H. H. Burns, 999-W
T. W. Coffield, 766-H
R. D. Deshpande, 766-H
R. E. Edwards, 773-A
G. P. Flach, 773-42A
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