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Tank 18-F and 19-F
Tank Fill Grout Scale Up Test Summary

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Bob Fogle, SRNL R&D Engineering, provided the thermocouples and assistance with calibration before the sensors were installed in the semi-adiabatic test form.

EXECUTIVE SUMMARY

High-level waste (HLW) tanks 18-F and 19-F have been isolated from FTF facilities [1]. To complete operational closure the tanks will be filled with grout for the purpose of: 1) physically stabilizing the tanks, 2) limiting / eliminating vertical pathways to residual waste, 3) entombing waste removal equipment, 4) discouraging future intrusion, and 5) providing an alkaline, chemical reducing environment within the closure boundary to control speciation and solubility of select radionuclides.

This report documents the results of a four cubic yard bulk fill scale up test on the grout formulation recommended for filling Tanks 18-F and 19-F. Details of the scale up test are provided in a Test Plan [2]. The work was authorized under a Technical Task Request (TTR), HLE-TTR-2011-008 [3], and was performed according to Task Technical and Quality Assurance Plan (TTQAP), SRNL-RP-2011-00587 [4].

The bulk fill scale up test described in this report was intended to demonstrate proportioning, mixing, and transportation of material produced in a full scale ready mix concrete batch plant. In addition, the material produced for the scale up test was characterized with respect to fresh properties, thermal properties, and compressive strength as a function of curing time.

A grout formulation for filling Tanks 18-F and 19-F was developed by SRNL during 2011 [5, 6]. The recommended material is a flowable zero bleed structural fill containing 3/8 inch gravel. The ingredients and proportions in the mix are listed in the table. Properties of this grout are provided elsewhere [6].

Mix Number	Cement Type I/II	Slag Grade 100	Fly Ash Class F	Type G Shrinkage Compensating Component	Sand Quartz	Gravel No. 8 3/8 in.	Water	HRWR SIKA Visco Crete 2100	VMA Diutan Gum Kelco-Crete DG
	Lbs / cyd						Gal / cyd	Fl oz / cyd	g / cyd
LP#8-16	125	210	363	0	1790	800	48.5	41	200

Four cubic yards of grout were batched at the LaFarge North America[a] batch plant in Jackson SC. LaFarge substituted two W. R. Grace products for the admixtures used in the recommended tank fill. The alternative admixtures were approved by SRNL and were used in some of the SRNS reactor in-situ decommissioning grouts. The order of addition of these admixtures was to 1) add W. R. Grace ADVA 575, high range water reducer (HRWR), at the central mixing station and 2) add a stabilized mixture of ADVA 575 and Diutan Gum to the truck at the test station. The amount of the stabilized mixture was determined based on the ASTM C1611 slump flow results at the test station.

Cement contacted the water in the transit mixer at 0724 hr. The material was approved at the batch plant at 0745 hr based on slump flow. The delivery truck arrived at the Site at 0800 hr. At 0815 hr, the first sample was collected from the truck at the F-Tank Farm test site.

Several property measurements were identified in the bulk fill grout scale up test plan. Some of the properties were measured at both the batch plant and at the point of delivery in F-Area. The slump flow per ASTM C1611 was 25.5 inches for material measured in F-Area which is 2.5 inch less than the slump flow measured at the batch plant. The value measured in F-Area was within the acceptable range

[a] LaFarge was recently acquired by ARGOS Ready Mix, LLC.

in the tank fill procurement specification (24 to 28 inches) and corresponded to values measured in the laboratory [6].

The static gel time was significantly shorter than the time measured for a sample prepared in the laboratory, 9.5 inches at 30 minutes (laboratory sample) compared to 0 inches at 30 minutes (production sample). Different mixing conditions, a longer time between batching and testing, and ambient conditions may have contributed to part of this difference. However, it is more likely that ADVA 575 was not completely equivalent to the SIKA ViscoCrete 2100 and had slightly less gel retardation effect. Concrete admixtures are complex blends of several active chemicals and need to be adjusted to obtain desirable results. In this case a small amount of admixture to extend the static working time or adjustment of the ADVA 575 and EXP 958 (mixture of ADVA 575 and Diutan Gum) is warranted. Such adjustments may be required often during full-scale production.

There was no significant change in the air content, unit weight and temperature of the grout for values measured at the concrete batch plant versus values measured at F-Tank Farm. Air content in the grout increased 0.3 volume percent after leaving the LaFarge batch plant. This reduced the measured unit weight from 136.6 to 135.1 lb/cft. The increase in the ambient temperature and grout temperature was < 3°F.

The set time of the scale up mix was 7.5 hours. Set time was determined using the Ultrasonic Pulse Velocity (UPV) method. A small decrease in signal velocity was noticed just before the grout set. The cause for the slight velocity decrease is unknown and attributed to someone checking the sample during the test. The measured set time was less than the 24 hour requirement to sustain next day operations and meets the production requirement for filling the waste tanks.

The scale up testing confirmed that offsite batching at a commercial plant and delivering the bulk fill material for filling Tanks 18-F and 19-F is feasible. Material batching and delivery to the F area Tank Farm was achieved in less than one hour.

The average compressive strength measured from samples cured 28 days was 2800 psi. This meets the Performance Assessment (PA) and Engineering requirement (> 2000 psi at 28 day).

A one cubic yard insulated plywood form with an insulated lid was poured with the tank fill grout for measuring the semi-adiabatic temperature rise. Thermocouples were installed at the center of the box at several elevations. Additional thermocouples were placed along the center of one side and in a corner of the box. Temperature readings were collected for approximately one month. The peak temperature occurred after 82 hours. The semi-adiabatic temperature rise was 23°C. This meets the objective for a grout that can be mass placed.

Saturated hydraulic conductivity, density and porosity were identified as optional parameters in the scale up test plan and were not measured.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

1.0 INTRODUCTION

High-level waste (HLW) tanks 18-F and 19-F have been isolated from FTF facilities [1]. To complete operational closure the tanks will be filled with grout for the purpose of: 1) physically stabilizing the tanks, 2) limiting / eliminating vertical pathways to residual waste, 3) entombing waste removal equipment, 4) discouraging future intrusion, and 5) providing an alkaline, chemical reducing environment within the closure boundary to control speciation and solubility of select radionuclides.

This report documents the results of a four cubic yard bulk fill scale up test on the grout formulation recommended for filling Tanks 18-F and 19-F. Details of the scale up test are provided in a Test Plan [2]. The work was authorized under a Technical Task Request (TTR), HLE-TTR-2011-008 [3], and was performed according to Task Technical and Quality Assurance Plan (TTQAP), SRNL-RP-2011-00587 [4].

1.1 Objective

The bulk fill scale up test described in this report was intended to demonstrate proportioning, mixing, and transportation, of material produced in a full scale ready mix concrete batch plant. In addition, the material produced for the scale up test was characterized with respect to fresh properties, thermal properties, and compressive strength as a function of curing time.

2.0 BACKGROUND

2.1 Bulk Fill Grout Formulation

A grout formulation for filling Tanks 18-F and 19-F was developed by SRNL during 2011 [5, 6]. The recommended material is a flowable zero bleed structural fill containing 3/8 inch gravel. The ingredients and proportions in the mix are listed in Table 2-1. Properties of this grout are provided elsewhere [6].

Table 2-1. Tanks 18 and 19-F Bulk Fill Material Recommendation [6].

Mix Number	Cement Type I/II	Slag Grade 100	Fly Ash Class F	Type G Shrinkage Compensating Component	Sand Quartz	Gravel No. 8 3/8 in.	Water	HRWR SIKA Visco Crete 2100	VMA Diutan Gum Kelco-Crete DG
	Lbs / cyd						Gal / cyd	Fl oz / cyd	g / cyd
LP#8-16	125	210	363	0	1790	800	48.5	41	200

2.2 Bulk Fill Grout Production

Four cubic yards of grout were batched at the LaFarge North America[2] batch plant in Jackson SC. The batch ticket for the material ordered for the scale up test is provided in Figure 2-1. Material suppliers for the grout ingredients are listed in Table 2-2. LaFarge substituted two W. R. Grace products for the admixtures used in the tank fill mix development testing. The alternative admixtures were approved by

[2] LaFarge was recently acquired by ARGOS Ready Mix, LLC.

SRNL and were used in some of the SRNS reactor in-situ decommissioning grouts. The order of addition of these admixtures was to 1) add W. R. Grace ADVA 575, high range water reducer (HRWR), at the central mixing station and 2) add a stabilized mixture of ADVA 575 and Diutan Gum to the truck at the test station. The amount of the stabilized mixture was determined based on the ASTM C1611 slump flow results at the test station. See Figure 2-2.

LAFARGE
NORTH AMERICA
509 Main St. • Jackson, S.C. 29831 • (803) 471-9207

JK NO. 029411

Plant: JACKSON 377

Truck Number	Batch User	Disp Ticket Num	Ticket ID	Time	Date
0825	dsellers	37715216	0	7:24	8/31/11

Load Size	Mix Code	Returned Mix	Qty	Seq	Load ID
4.00	CYDSRMXEUS9003W			W	50

Mix: SRS F-TANK FARM CLOSURE MIX

3 Mins 24 Secs Customer: RDM COD NON CONTRACTOR US PO:

Material	Description	Design Qty	Required	Batched	% Var	% Moisture	Actual Wat
600	TYPE I/II CEMENT	125.0 lb	500.0 lb	495.0 lb	-1.00%		
670	CLASS F ASH	363 lb	1452 lb	1449 lb	-0.49%		
200	NATURAL SAND	1730 lb	7581 lb	7520 lb	-0.81%	5.69% A	60 gl
342	8 STONE	800 lb	3200 lb	3220 lb	0.63%		
101	CITY WATER	46.50 gl	.00 gl	.00 gl			
610	SLAG	210.0 lb	840.0 lb	845.0 lb	0.60%		
591	ADVA 575	41.00 oz	164.00 oz	160.00 oz	-2.44%		
102	WATER	100.00 % *	139.52 gl	139.00 gl	-0.37%		139.00 gl

Actual Load 14695 lb Num Batches: 1 Design W/C: 0.580 Water/Cement: 0.591 T Design 194.0 gl Manual 7:28:14 Actual 189.1 gl To Add 4.9 gl

Slump 4.00 in Water in Truck: 0.0 gl Adjust Water 0.0 gl / Load Trim Water: -1.0 gl / CYD

MIXER B: 1 Mixed 0 of 60 Sec.

FLOW AT PLANT - 28"
[illegible], 8/31/11

165 fl.oz. ADDED (*EXP [illegible]) AT PLANT [illegible] 8/31/11
[illegible] 8/31/11

Figure 2-1. Batch ticket for grout ordered for the scale up test.

Cement contacted the water in the transit mixer at 0724 hr. The material was approved at the batch plant at 0745 hr based on slump flow of 30 x 26 inches, (surface supporting test board was slightly irregular). The delivery truck arrived at the Site at 0800 hr. At 0815 hr, the first sample was collected from the truck at the F-Tank Farm test site.

Table 2-2. Ingredients Used to Prepare Grout.

Material	Specification	Supplier / Address
Portland cement (Type I/II)	ASTM C150	LaFarge,Cement Harleyville, SC obtained from Lafarge Ready Mix Augusta, GA
Slag cement (Grade 100)	ASTM C989	Holcim, Inc., 3235 Satellite Blvd. Duluth, GA 30096
Fly ash (Class F)	ASTM C618	Wateree Power Plant, SC SEFA, Inc.
Concrete sand	ASTM C33	SCMI, Clearwater SC obtained from LaFarge Ready Mix, Jackson, SC
No. 8 stone 3/8 inch gravel (granite)	ASTM C33	Martin Marietta Quarry Augusta, GA obtained from LaFarge Ready Mix, Jackson, SC
HRWR		
ADVA 575*	ASTM C494 Type F	W. R. Grace Corporation
Viscosifier		
EXP 958** (Diutan Gum)		W. R. Grace Corporation
Potable water		Jackson, SC Municipal Water Supply

* Sika ViscoCrete 2100 was used in the laboratory testing.
** EXP 958 is a stabilized mixture of ADVA 575 and Kelco-Crete Diutan® provided by CP Kelco, Inc., 8355 Aero Dr., San Diego, CA 92123.

Table 2-3. Size Distribution of the Sand and No. 8 Stone [Waymer, 2011].

Property	Concrete Sand		No. 8 Aggregate (3/8 inch)	
Bulk Unit Weight (lb/ft^3)	85 @ 1.6 wt. % SSD*		93 @ 0.6 wt. % SSD*	
Specific Gravity (particle)	2.65		2.65	
Composition	Quartz		Granite	
Particle Size Distribution $^+$	Wt. % Passing	Cum. Wt. % Retained	Wt. % Passing	Cum. Wt. % Retained
½ inch (12.5 mm)	100	0	99.4	0.6
3/8 inch sieve	100	0	91.8	8.2
¼ inch sieve	--	--	40.0	60.0
#4 sieve (4.75mm)	99	1	14.2	85.8
#5 sieve (4.00 mm)	--	--	6.3	93.7
#8 sieve (2.36 mm)	96	4	0.6	99.4
#16 sieve (1.18 mm)	81	19	--	--
#30 sieve (600 µm)	50	50	--	--
#50 sieve (300 µm)	17	83	--	--
#100 sieve (150 µm)	2	98	--	--
Fineness Modulus	--	2.6	--	--

$^+$ Percentage passing through each sieve as determined by ASTM C136.

Figure 2-2. Admixture dose adjustment at the LaFarge batch plant based on ASTM C1611 test results.

2.3 Test Methods

Test methods are provided in Table 2-4. Descriptions of the test methods for evaluating fresh properties and cured grout properties are covered elsewhere [6].

Table 2-4. Test Methods Used to Determine Grout Properties.

Properties	ASTM Methods
Fresh Properties	
Flow (Initial and Static Flow)	D6103
Slump Flow	C1611
Set Time	UPV and visual
Bleed Water (24 hr.)	C232
Segregation	Visual
Unit Weight	C138
Air Content	C231
Grout Temperature	C1064
Thermal Property	
Semi adiabatic temperature rise	Insulated 1 cubic yard monolith with embedded thermocouples
Cured Properties	
Compressive Strength	C39
Saturated Hydraulic Conductivity	D5084 Methods C or F

2.4 Description of Semi Adiabatic Form

SRR Construction fabricated a one cubic yard insulated plywood form with an insulated lid for the semi- adiabatic temperature rise measurement. The box was lined with a plastic sheet. Thermocouples were installed at the center of the box at the following elevations: 6, 12, 18, 24, and 30 inches from the bottom of the box and were supported by a PVC pipe. Additional thermocouples were placed along the center of one side and in a corner of the box 18 inches from the bottom and a few inches off the walls. The leads for the thermocouples were fed through the top of the box and were connected to a data logger. In addition, ambient temperature next to the form and 5 ft from the form were also monitored for the duration of the test.

(a) (b)

Figure 2-3. (a) Semi adiabatic test form and (b) Data logger set up.

2.5 Semi Adiabatic Form Filling

The semi-adiabatic form was filled by discharging directly from the truck into the form. The grout was more or less self-leveling and did not require finishing. See Figures 2-4(a) and (b). After the form was filled the insulated lid was placed on the box and was left in place for approximately one month as temperature readings were taken.

(a) (b)

Figure 2-4. (a) Bulk tank fill grout placed into the semi adiabatic form and (b) Near full form.

3.0 RESULTS

3.1 Fresh Properties

Several property measurements were identified in the bulk fill grout scale up test plan. Some of the fresh properties were measured at both the batch plant and at the point of delivery in F-Area. The slump flow per ASTM C1611 was 25.5 inches for material measured in F-Area which is 2.5 inch less than the slump flow measured at the batch plant. The values measured in F-Area were within the acceptable range and corresponded to values measured in the laboratory [6]. The initial spread, Figure 3-1 (a), and spread after static conditions for 15 and 30 minutes, Figure 3-1 (b) top left and top right, respectively illustrate the static working time

(a) (b)

Figure 3-1. Spread under static conditions, (a) initial, (b) Top left 15 minutes, Top right 30 minutes.

This static gel time was significantly shorter than the time measured for a sample prepared in the laboratory, 9.5 inches at 30 minutes (laboratory sample) compared to 0 inches at 30 minutes (production sample). Different mixing conditions, a longer time between batching and testing, and ambient conditions may have contributed to part of this difference. However, it is more likely that ADVA 575 was not completely equivalent to the SIKA ViscoCrete 2100 and had slightly less gel retardation effect. Concrete admixtures are complex blends of several active chemicals and need to be adjusted to obtain desirable results. In this case a small amount of admixture to extend the static working time or adjustment of the ADVA 575 and EXP 958 (mixture of ADVA 575 and Diutan Gum) is warranted. Such adjustments may be required often during full-scale production.

Air content in the grout increased 0.3 volume percent after leaving the LaFarge batch plant. This reduced the measured unit weight from 136.6 to 135.1 lb/cft. There was also a small increase in the ambient temperature and grout temperature (< 3°F).

The set time of the scale up mix, LP#8-016SU, was 7.5 hours. Set time was determined using the Ultrasonic Pulse Velocity (UPV) method and the data is graphed in Figure 3-2. A small decrease in signal velocity was noticed just before the grout set. The cause for the slight velocity decrease is unknown and attributed to someone checking the sample during the test.

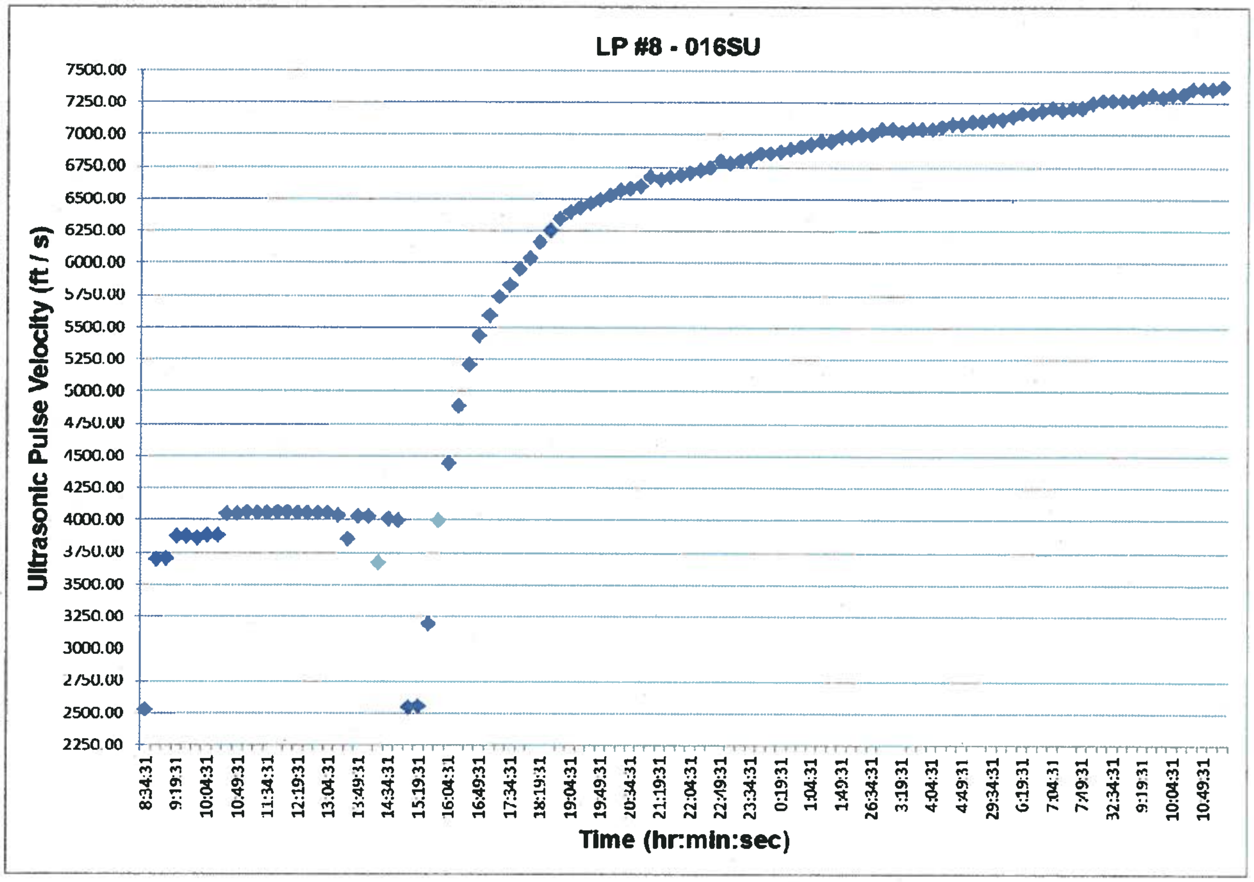


Figure 3-2. Velocity variation with time through a sample of the bulk fill grout collected from the scale up test batch.

The fresh properties are summarized in Table 3-1.

Table 3-1. Fresh Properties of the Bulk Fill Scale Up Mix.

Properties	ASTM Methods	Batch Plant	FTF
Slump Flow (inches)	C1611	30 x 26 Ave. 28	25.5 x 25.5 Ave. 25.5
Spread Initial (inches)	D6103	Not measured	10 x 10
Spread (inches) after static condition 15, 30, 45 min.	SRNL Modified D6103	Not measured	T_{15} = 7 x 6.5 (Ave. 6.75) T_{30} = 0 x 0 (Ave. 0) T_{45} = 0 x 0 (Ave. 0)
Set Time (hr)	UPV and visual	Not measured	7.5
Bleed Water (24 hr.)	C232	Not measured	0
Segregation	Visual	Not measured	0
Unit Weight (lbs/cft)	C138	136.6	135.1
Air Content (vol. %)	C231	0.8	1.1
Grout Temperature	C1064	75°F	77°F
Ambient Temperature	C1064	73.0°F	76.6°F

3.2 Thermal Properties

The curing temperatures for the one cubic yard monolith are provided in Figure 3-3. Nine thermocouple locations are included in the graph. See Figure 3-4. Thermocouple data was collected over a period of 29 days. The peak temperature, 47°C, occurred 82 hours after pouring the test form. The location was at the center of the box and 24 inches from the bottom. The temperature rise for the one cubic yard monolith was 23°C. After 82 hours, the block temperature declined over the next 180 hours before leveling off for the next 120 hours. After 380 hours into the test, the outside temperatures fell during the day and the block temperature started declining again.

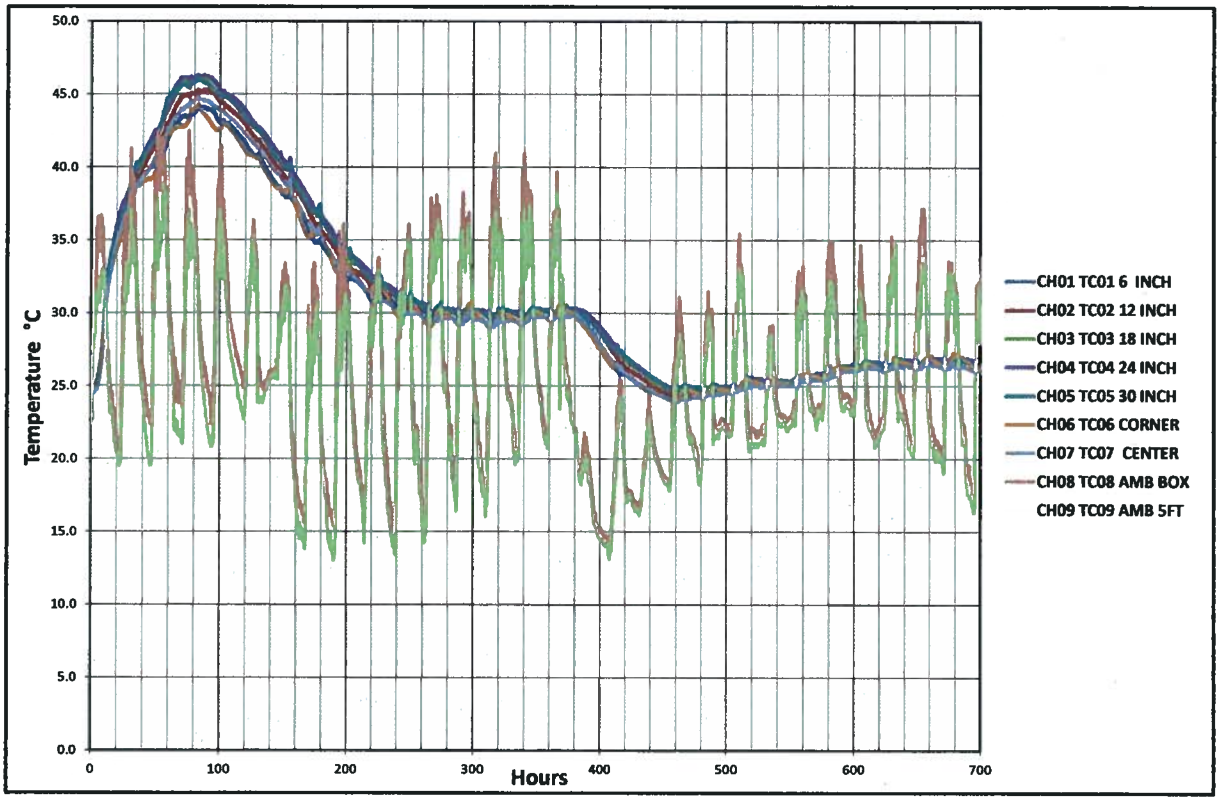
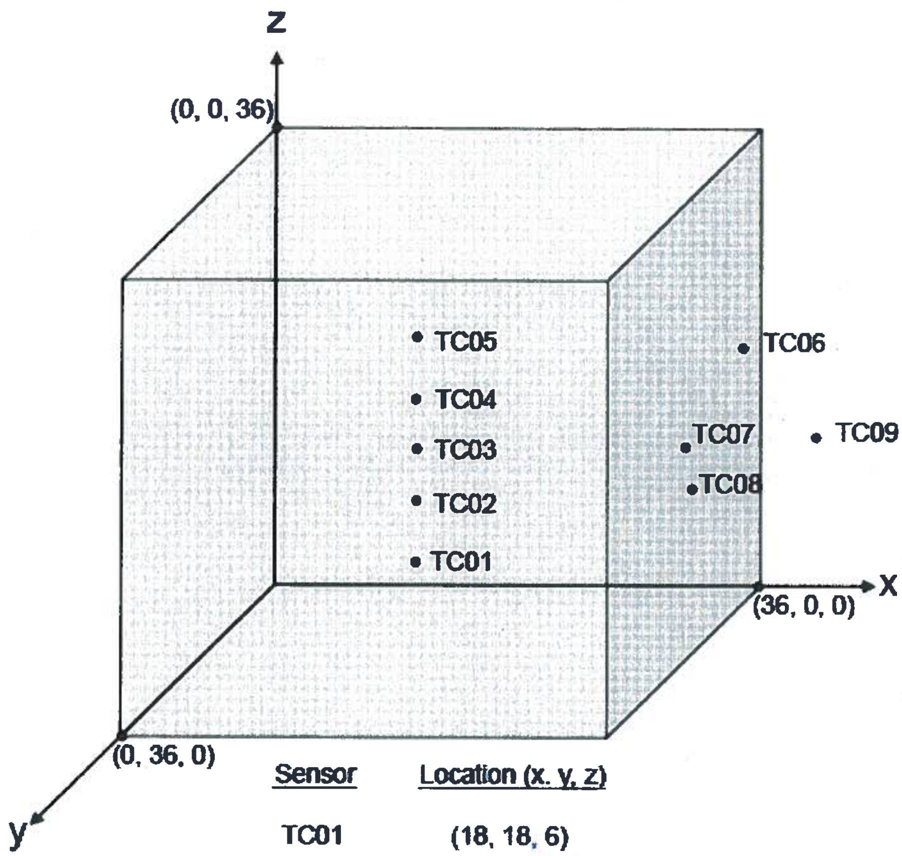


Figure 3-3. Tank fill grout - Semi-adiabatic temperature results for the one cubic yard monolith prepared on 8-31-2011.



Sensor	Location (x, y, z)
TC01	(18, 18, 6)
TC02	(18, 18, 12)
TC03	(18, 18, 18)
TC04	(18, 18, 24)
TC05	(18, 18, 30)
TC06	(34, 2, 18)
TC07	(34, 18, 18)
TC08	~ (37, 18, 18)
TC09	~ (97, 18, 18)

Figure 3-4. Map of thermocouple sensor locations for one cubic yard monolith.

3.3 Cured Properties

The cured properties results are provided in Table 3-2. Four inch by eight inch cylinders were cast for compressive strength measurements as a function of curing times (7, 28 and 90 days). Two by four inch cylinders and three by six inch cylinders were cast for hydraulic conductivity samples. Samples were prepared according to ASTM C192 and cured in a constant temperature (73°F ± 2°F) curing room at 100% relative humidity until ready for testing. Two cylinders were broken during each compressive strength time interval. These strengths and averages are included in Table 3-2.

Saturated hydraulic conductivity, density, and porosity were identified as optional parameters in the scale up test plan. These properties were not measured. Segregation was evaluated by visual examination. The grout did not segregate.

Table 3-2. Cured Properties of the Bulk Fill Scale Up Mix.

Properties	ASTM Methods	Result
Compressive Strength (psi)	C39	
7 days (2)		350, 380 (365 ave.)
28 days (2)		2870, 2770 (2820 ave.)
90 days (2)		5020, 4790 (4905 ave.)

4.0 CONCLUSIONS AND RECOMMENDATIONS

The scale up testing confirmed that offsite batching at a commercial plant and delivering the bulk fill material recommended by SRNL for filling Tanks 18-F and 19-F is feasible. Material batching and delivery to the F area Tank Farm was achieved in less than one hour.

The slump flow measured per ASTM C1611 in F-Area was within the acceptable range (24 to 28 inch) in the procurement specification and corresponded to values measured in the laboratory [6]. The static gel time was significantly shorter than the time measured for samples prepared in the laboratory, 9.5 inches at 30 minutes in the laboratory compared to 0 inches at the 30 minutes at the point of delivery. This difference is attributed to a longer time between batching and testing and the concrete admixture differences (Sika ViscoCrete 2100 during laboratory samples versus ADVA 575 and EXP 958 during scale up testing).

There was no significant change in the air content, unit weight and temperature of the grout for values measured at the concrete batch plant versus values measured at F-Tank Farm.

The set time of the scale up mix was 7.5 hours. This is less than the 24 hours requirement to sustain next day operations and meets the production requirement for filling the waste tanks.

The average compressive strength measured from samples cured for 28 days was 2800 psi. This meets the Performance Assessment (PA) and Engineering requirement (> 2000 psi at 28 day).

The temperature rise under semi-adiabatic conditions was 23°C for the insulated 1 cubic yard monolith poured, and occurred after 82 hours. Beyond 82 hours, the block temperature declined. This meets the objective for developing a grout that can be mass placed.

Saturated hydraulic conductivity, density and porosity were identified as optional parameters in the scale up test plan and not measured.

5.0 REFERENCES

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