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PILOT-SCALE PRODUCTION OF GROUT WITH
SIMULATED DOUBLE-SHELL SLURRY FEED

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MASTER

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FOREWORD

This report describes the pilot-scale production of grout with simulated double-shell slurry feed (DSSF) waste performed in November 1988, and the subsequent thermal behavior of the grout as it cured in a large, insulated vessel. The report was issued in draft form in April 1989 and comments were subsequently received; however, the report was not finalized until 1994. The characterization of the grout produced during this pilot-scale test is described in Lokken et al. (1992).

A subsequent pilot-scale production run of grout has since been performed using a simulated Tank 106-AN waste and a different dry blend, as described by Bagaasen (1993). The characterization of the grout produced in this subsequent pilot-scale test is described in Lokken et al. (1993).

In finalizing this report, references or information gained after the report was drafted in April 1989 have not been incorporated to preserve the report's historical perspective. This report makes use of criteria from Riddelle (1987)^(a) to establish formulation criteria. This document has since been superseded by a document prepared by Reibling and Fadeff (1991). However, the reference to Riddelle (1987) and any analysis based on its content have been maintained within this report. In addition, grout is no longer being considered as the waste form for disposal of Hanford's low-level waste. However, grout disposal is being maintained as an option in case there is an emergency need to provide additional tank space. Current plans are to vitrify low-level wastes into a glass matrix.

(a) Riddelle, J. G. 1987. *Formulation Criterion for the Hanford Grout Disposal Program*. Internal Report SD-WM-SS-004, Westinghouse Hanford Company, Richland, Washington.

SUMMARY

Grout consists of liquid waste mixed with cementitious dry solids to form a solid material that immobilizes the waste. At the Hanford Site in Richland, Washington, grout is produced and disposed at the Grout Treatment Facility (GTF), which is operated by Westinghouse Hanford Company (Westinghouse) for the U.S. Department of Energy. Plans currently exist to dispose of the low-level fraction of double-shell tank waste in a grout waste form that will be placed inside large vaults designed to minimize migration of contaminants. Pilot-scale testing is performed by Pacific Northwest Laboratory in an approximately 1/4-scale process to ensure that the mix of the waste and cementitious dry solids can be processed in equipment similar to the equipment in the GTF.

This report discusses the pilot-scale production test of grout produced from simulated double-shell slurry feed (DSSF), a low-level liquid waste. Some observations from the test are listed below:

- Measurements of grout rheology indicated that the grout thinned while flowing in the pipe. The DSSF grout at the mixer outlet was much thicker than the grout samples mixed in the laboratory. The grout at the pipe exit was similar to the laboratory-mixed samples. If the grout entering the pipe in the GTF has the rheological properties measured at the mixer outlet in the pilot-scale test, the recommended grout production rate for the GTF will be 65-70 gpm.
- The heat evolved during curing was substantially greater than previously estimated. An actual temperature rise of 61°C was observed. From heat loss estimations it is believed that the adiabatic temperature rise would have been 83°C. This heat rise is a concern for grout that will cure inside large vaults.
- The first mixer paddle in the grout mixer showed substantial wear upon completion of the test. The amount of wear is significant since the run lasted only 4.5 hours. However, the mixer in the GTF has stellite tips, which should reduce wear.
- The grout set within 20 hours and no separated liquid was observed. The grout experienced extremely small dimensional changes after hardening. Short-term dimensional changes are not a concern.

ACKNOWLEDGMENTS

I would like to thank Technical Specialist Chuck R. Hymas for his valuable contributions in preparing the pilot-scale equipment for production, and his assistance in operating the equipment during grout production.

The work described in this report was performed by Pacific Northwest Laboratory (PNL) as part of the Hanford Grout Technology Program (HGTP). At the time this test was run, PNL's work on the HGTP was managed by Don Mitchell at PNL. Rick Farnsworth (PNL) took samples during production. Ryan Lokken and Paul Martin (PNL) assisted in grout production and performed rheological and thermal conductivity measurements. Acoustic wave speed and attenuation measurements during and after grout curing were performed by George Schuster and Morris Goode (PNL). Jeff Allison and Elizabeth Carter of Westinghouse Hanford Company operated an inline grout sampler. This report was edited by Kathy Blanchard, and word processing was completed by members of 300 Area Publications Support team. The work described was funded by the Grout Disposal Program at Westinghouse Hanford Company.

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1.0 INTRODUCTION

At the Hanford Site near Richland, Washington, plans currently exist for disposing of the low-level fraction of liquid double-shell tank waste in a grout form. Grout is a mixture of liquid waste and grout formers, including fly ash, blast furnace slag, and portland cement. Processing of these liquid wastes into grout will be performed in the Grout Treatment Facility (GTF), which is operated by Westinghouse Hanford Company (Westinghouse) for the U.S. Department of Energy (DOE). The grout slurry that is produced by mixing liquid waste and grout formers will be pumped to engineered vaults and will harden into large grout masses, thereby immobilizing the waste. The vault in which the solid grout will be contained is designed to further retard migration of waste species to the environment. Pacific Northwest Laboratory^(a) (PNL) is verifying that grouts produced using waste from the Hanford site can be safely and efficiently processed.

A similar concept for disposal of low-level radioactive wastes is being planned at the Savannah River Laboratory site (Langton 1984). In addition, waste has been disposed of in grout at the Oak Ridge National Laboratory using a process called "hydrofracture grouting" (Dole 1985). In this process the grout slurry is injected into shale formations 200 to 300 m below the surface.

The GTF has previously been used to dispose of phosphate/sulfate waste (PSW), which is a very low activity waste originating from the N Reactor operations at Hanford. Production of grout made from PSW began in August 1988.

The GTF includes the Dry Materials Facility (DMF), the Transportable Grout Equipment (TGE), and grout disposal vaults (Guymon, Vanselow and Campbell 1988). The dry materials that compose the grout formers are received, stored, and blended at the DMF. The dry blend is then transported by truck to the TGE, where it is mixed with waste in a continuous process to form grout. The liquid grout is then pumped to disposal vaults where it

(a) Pacific Northwest Laboratory is operated by Battelle Memorial Institute for the U.S. Department of Energy under contract DE-AC06-76RLO 1830.

solidifies and immobilizes the waste. Pilot-scale work to support the operation of the GTF is performed by PNL. The equipment described in this report produces grout at up to 25% of the TGE processing rate. The equipment was used previously in a 24-hr processing study of PSW grout production (Fow et al. 1987).

For the work reported here, the pilot-scale equipment was used to produce grout from simulated double-shell slurry feed (DSSF), a type of waste stored in double-shell tanks at Hanford. The simulated waste was similar to actual wastes that will be grouted, although it did not contain radionuclides. The dry blended solids consisted of 47 wt% class F fly ash, 47 wt% blast furnace slag, and 6 wt% type I,II portland cement. This blend is also planned for use in the GTF for grouting double-shell slurry waste.

This report presents results from a 4.5-hr pilot-scale production test of DSSF grout.

1.1 OBJECTIVES OF THE PILOT-SCALE TEST

The primary objectives of the test described in this report were 1) to demonstrate the processability of the DSSF grout formulation that was developed using laboratory equipment and 2) to provide information for scale-up. The temperature of the grout feed ($>40^{\circ}\text{C}$) was higher than that in previous tests with PSW grout. One desirable outcome was to show that the properties of grouts mixed in the laboratory are similar to those for grouts mixed in equipment similar to the TGE.

A third objective was to provide information on the behavior of grout after production, including: 1) an estimate of the heat of hydration during curing, 2) measurements of thermal conductivity, 3) measurements of volume changes during curing, 4) determination of possible effects from the 34-ft fall the grout will initially experience when entering the vault, 5) observations of the amount of separated liquid occurring after placement, 6) determination of the restart pressures that can be expected if the pump operation is interrupted, and 7) provision of samples for characterizing the grout waste form. The results of this characterization will be discussed in a separate report.

1.2 SCOPE OF THE PILOT-SCALE TEST

The simulated DSSF waste was processed for 4.5 hours at a rate of 7.3 gpm (10.4 gpm of grout), for a total volume of 2800 gal of grout. The grout discharged into an 8-ft-dia., 7.5-ft-high insulated tank. Access hatches in the tank lid permitted sampling of the grout and observation of curing.

During production, a data logger was used to monitor feed and production rates, pressures, and temperatures. Density and rheological properties were measured on grout samples taken at the exit of the mixer and the discharge of the grout pipe. The production equipment used in the pilot-scale test was also evaluated to identify the effects of grout processing.

The data logger also collected long-term temperature data from the grout monolith to determine the extent of heat generation due to hydration of the grout. Strain information was also collected to determine if the grout changed in volume during curing. Thermal conductivity of the grout was measured using in situ probes.

Chapter 2.0 describes the equipment used in the pilot-scale test, the instrumentation used to collect data, the sampling strategy, and the test materials. Chapter 3.0 reports results of the test, and Chapter 4.0 lists the conclusions and recommendations. References are listed in Chapter 5.0.

2.0 EXPERIMENTAL DESCRIPTION

This chapter describes the equipment, instrumentation, materials, and experimental configuration used in the pilot-scale production test of simulated DSSF grout.

2.1 PROCESS EQUIPMENT

This section describes the pilot-scale equipment used to process the grout. A schematic of the grout process is shown in Figure 2.1.

2.1.1 Makeup and Storage Tank for the Simulated DSSF

The waste was made in a closed 4000-gal tank (tank HB-13 in the 324 Building high bay). Appendix A describes the procedure used for waste makeup. The tank is equipped with a 10-hP, variable-speed agitator, and an 18-kW circulation heater with temperature control to heat the waste, if required. The tank is insulated except on the top and an area on the bottom, where a water spray is used for external cooling.

2.1.2 Delivery System for the Simulated DSSF

Two centrifugal pumps are located underneath the waste storage tank. Either pump can function as the circulation pump for the tank heater or as the delivery pump for providing waste to the grout process. The waste was pumped through a 1-in. line from the storage tank to a pneumatically actuated pinch valve controlled by a proportional/integral (PI) controller, which uses a magnetic flow meter for an input signal. The flow of simulated DSSF is controlled independently of the dry blend flow rate. The flow control system allows the waste flow to the grout mixer to be maintained within tight tolerances, thus reducing the variability in the final product.

2.1.3 Dry Blend Feed System

The dry blend feed system consists of ground-level hoppers in which the dry blend was initially stored, a pneumatic transfer system, a storage bin/baghouse, an active bin/feeder, and a vibrating screen.

FIGURE 2.1. Schematic of the Pilot-Scale Grout Process

Dry blend, a mixture of 47% blast furnace slag, 47% class F fly ash, and 6% type I,II portland cement, was stored in hoppers that were placed at ground level along the outer east wall of the 324 Building high bay. Two layers of tarp covered the hoppers to protect the blend from moisture. The dry blend was pneumatically transferred in batches from the hoppers to a storage bin with a 27-ft³ capacity. The dry blend was drawn through a wand that was directed by an operator during transfer (Figure 2.2).

The storage bin is above the active bin/feeder, and is equipped with a baghouse that separates the dry blend from the transfer air. The transfer

FIGURE 2.2. Loading of Dry Blend Solids Using Suction Wand

system and storage bin were manufactured by Vac-U-Max.[®] The contents of the storage bin were automatically dumped to the active bin on a signal from the feeder controller. Figure 2.3 shows the baghouse/storage bin, active bin/feeder, and vacuum/transfer blower. The active bin has a capacity of 36.6 ft³ and an active volume of 30 ft³. The feeder is an Acrison[®] gravimetric, auger-type feeder with a weight rate accuracy of 0.5% of the set point. The dry blend feed rate is controlled independently of the DSSF flow. When the feed

[®] Vac-U-Max, Belleville, New Jersey.

[®] Acrison, Inc., Moonachie, New Jersey.

FIGURE 2.3. Dry Blend Feeder, Storage Bin, and Pneumatic Conveying Equipment

bin reaches a predetermined low level, the feeder is automatically switched to a volumetric mode (constant auger speed) and a valve between the feeder and the storage bin is opened for reloading. At this time, a vibrator and air pads on the storage bin are activated to promote the discharge of dry blend from the storage bin. The reload valve is closed when the weight reaches 80% of the feeder capacity. The feeder discharges through an 18-in.-dia. Sweco®

® Sweco Inc., Los Angeles, California

vibrating screen designed to remove material greater than 0.20 in. from the dry feed. The oversize material is collected in a separate container. In the previous pilot-scale grout production run in which PSW grout was produced, a flooding problem was encountered with the feeder. Pre-run tests indicated that this problem was caused by the auto-compensation feature, so the feature was disabled. The dry blend feed system operated smoothly during DSSF grout production. In the GTF, a weigh belt rather than an auger is used to meter feed.

2.1.4 Grout Mixer

Dry blend and simulated waste are combined in the mixer to produce a grout slurry. The grout mixer is a 5.25-in. continuous processor manufactured by Teledyne Readco® (Figure 2.4). This mixer is the same brand and type used in the GTF. Dry blend and simulated waste enter at the top of one end of the mixer, and the grout slurry discharges at the opposite end into a surge tank. Figure 2.5 shows the interior of the mixer. Dry blend is introduced at the left end into the screw section. Liquid is introduced where the mixer paddles begin. The paddles are 1-in. wide and provide low-shear mixing of grout during mixer operation.

Mixing speed is adjustable from 50 to 270 rpm. For this pilot-scale test the mixer was operated at 250 rpm, the speed selected based on previous tests with a different grout. The mixer in the TGE has 7-in.-dia. mixing paddles, compared to 5.25 in. for the pilot-scale mixer. The speed of the mixer is scaled by the tip speed of the mixer paddles.

Unlike the mixer in the GTF, the pilot-scale mixer has an adjustable discharge gate that can be used to adjust residence time in the mixer. The mixer gate was left wide open and was not adjusted during the run.

® Teledyne Readco, York, Pennsylvania.

FIGURE 2.4. Pilot-Scale Grout Mixer

The mixer discharges into a 19-in.-dia., 20-in.-deep surge tank with a cone-shaped bottom that leads to the grout pump suction through a 4-in.-dia., 34-in.-long hose. The surge tank is equipped with a screen along the bottom that prevents particles larger than ≈ 0.5 in. from entering the grout pump. This screen prevents tools and other objects from falling into the surge tank and entering the grout pump. The screen was removed during the run because of concerns over buildup of grout in the bottom of the surge tank.

FIGURE 2.5. View of Internal Components of the Grout Mixer

2.1.5 Grout Pump

The grout pump is a Roper® two-stage progressive cavity pump (Figure 2.6) with an ethylene-propylene-diene-monomer (EPDM) stator. The pump seal is a water-lubricated packing gland. The pump speed is selected by

® Roper Pump Co., Commerce, Georgia.

FIGURE 2.6. Progressive Cavity Grout Pump

manually entering a set point into a speed controller. Speed and amperage are displayed at a control station and recorded by a data logger. A high-pressure, automatic shut-off feature stops the pump if the pressure indicated by the exit pressure transducer is greater than 114 psi. The grout pump inlet is equipped with an automatic flush. This flush was set to flush every 15 min for 3 sec at a flow rate of 6 gpm. The inlet flush nozzle is shown in Figure 3.13.

The grout pump in the GTF is of a similar design to the pilot-scale pump, although it is larger and is capable of operating at higher pressures and flow rates.

2.1.6 Grout Pipe

The pump discharged grout into a 3/4-in. schedule 40, carbon steel pipe that ran to the receiver vessel. The piping is sized to maintain turbulent flow at the planned production rate of 10 gpm, based on typical rheological

properties of DSSF grout mixed in the laboratory. For this pilot-scale test, the piping was 170 ft long and contained 14 4-in.-radius bends and a 3-way valve. The 3-way valve was installed so that the grout would flow over the back of the plug since this is the intended installation at the GTF. The valve was used to divert material not intended for the receiver vessel into an alternate receiver bin.

A high-pressure flushing pump was available for clearing the grout pipe in case a plug developed that could not be cleared by the pressure of the process water. This pump was not required during the run.

2.1.7 Receiver Vessel

The grout receiver system consisted of a receiver vessel and a drop chute (Figure 2.7). The receiver vessel was an 8-ft-dia., 7.5-ft-high steel culvert, with a bottom seal welded on one end. The vessel lid rested on top of the culvert and had several hinged hatches that provided access to the vessel. These hatches permitted sampling of the grout and observation of curing. A nonsealed, metal-to-metal contact provided the contact between the top of the vessel and the vessel wall and access hatches. The lid and sides of the vessel were insulated with 6 in. of fiberglass insulation. The vessel rested on a steel plate that was placed over a frame of 6-in. square steel tubes. The air space between the steel plate and the ground provided insulation for the bottom of the vessel. A 2-ft-dia., 27.5-ft-long polypropylene pipe was mounted vertically over the center of the vessel lid. This pipe, referred to as the drop chute, allowed the grout to fall 34 ft before hitting the bottom of the vessel, which matched the distance that the grout will fall when entering a full-scale vault.

After grout production was complete, the drop chute was removed and the hole in the top of the vessel was insulated. The entire vessel was covered with a plastic tarp to form a tent that protected the vessel from wind and rain. A small heater was placed inside of this tent to help reduce heat loss. Silica gel was spread on drum lids that were placed under the tent to reduce condensation and wetting of the insulation.

FIGURE 2.7. Receiver Vessel and Drop Chute. (The drop chute is a 2-ft-dia. polypropylene pipe through which the grout fell. The receiver bin to the right received pretest grout and flush water.)

A seal-welded, carbon steel receiver bin was placed alongside of the vessel to receive material that was not desired in the receiver vessel. Material sent to the dumpster included grout produced during tests of equipment before the actual pilot-scale test, and water flushes of the grout pipe during process shutdowns. Flushing of the grout pump inlet during production is a normal occurrence, and this water went with the grout to the receiver vessel.

2.2 INSTRUMENTATION

This section describes the instrumentation used to collect data during and after the pilot-scale grout production.

2.2.1 Process Instrumentation

The process instrumentation is depicted in Figure 2.8. The data from the process were collected and recorded by a data logger with two multiplexer cards, each capable of recording 32 differential measurements. Process temperatures, DSSF and grout flow rates, grout pump discharge pressure, speed and amperage, dry blend feed rate, receiver vessel temperatures, and strain in the cured grout were recorded. The data logger sent the data to a computer, which recorded the data on disk, and to a printer. A basic program was used for data manipulation. The data logger sampled the process variables every 5 min.

Temperatures at the DSSF storage tank, the inlet to the mixer, the grout surge tank, and the grout in the pipe before it entered the receiver vessel were obtained during grout production. All temperatures were recorded by the data logger.

Liquid flow measurements included measurements of the DSSF flow rate, the grout flow rate, and the flush water flow rate. The DSSF flow rate was measured using a magnetic flow meter, which provided a signal to the flow controller. The flow controller drove a pneumatic valve actuator, displayed the flow rate and setpoint, and output a mA signal across a precision resistor to provide a voltage representing DSSF flow rate for the data logger. A rotometer in the DSSF supply line was not useful during production because the finely divided black solids made the waste opaque, thus making it impossible to read the rotometer. The grout flow rate was also measured using a magnetic flow meter. A display located on the flow meter identified percent of full scale, and also provided a current output that was converted to a voltage and transmitted to the data logger. The flush water line was equipped with a turbine flow meter that provided a display in gpm at the control station. This flow meter was equipped with a totalizer to identify the total amount of flush water delivered to the vessel during the run. The volume of flush water was not recorded by the data logger.

A	Amps	HLS	High Level Switch	R	Rotameter
AV	Air Actuated Valve	K	Thermal Conductivity Probe	S	Solenoid Valve
CR	Chart Recorder	LLS	Low Level Switch	SG	Strain Gage
DL	Data Logger	LW	Low Weight Switch	SGC	Strain Gage Conditioner
EV	Electrical Valve Actuator	M	Manual Valve	T	Tachometer
F	Flowmeter	MT	Manual Three Way Valve	TC	Thermocouples
FEC	Feeder Control	P	Pressure Gage	TI	Timer On/Off
FLC	Flow Control	PT	Pressure Transducer	W	Weight Transducer
FT	Flow Totalizer	PUC	Pump Control		

38802-134.3M

FIGURE 2.8. Process Instrumentation

The grout pump speed, discharge pressure, and amperage were all displayed at the control station and recorded by the data logger. The feeder controller was located at the control station and allowed feeder parameters such as motor speed, bin weight, and current feed rate to be displayed. The instantaneous feed rate was transmitted to the data logger. If the feeder bin was refilling and operating at constant speed, the feed rate recorded by the data logger was determined by the motor speed. Data collected by the data logger while the feeder was refilling may be of lower accuracy.

The rheology of the grout was determined from samples taken at the outlet of the mixer and the bottom of the drop chute. The apparent viscosity was measured at different shear rates using a Fann® viscometer. This data was used to determine if the grout produced by the pilot-scale equipment was similar to the grout produced in the laboratory.

2.2.2 Receiver Vessel Instrumentation

The instrumentation of the vessel included equipment to measure temperature, strain in the cured grout, thermal conductivity, acoustic properties of the grout, and the transmitted pressure of separated liquid pressure to the bottom of the vessel. Some of the instrumentation installed in the vessel is depicted in Figure 2.9.

The temperature was measured using 40 thermocouples specially arranged throughout the grout monolith to gather information on the temperature profiles (Figure 2.10). The identity numbers and elevation of the thermocouples are listed in Table 2.1. The thermocouples were attached to tubes that were anchored to the floor of the vessel and held in place at the top by the penetration of the vessel lid. Each thermocouple was bent 6 in. away from the tube to minimize the effect of the tube on the temperature profile being measured. An example of a thermocouple tree is shown in Figure 2.9. More thermocouples were placed near the center of the vessel to provide a better approximation of the maximum temperature rise caused by hydration. Thermocouples were also placed in the air space between the vessel lid and the

® Fann Instrument Corporation, Houston, Texas.

FIGURE 2.9. Instrumentation Inside Receiver Vessel

grout, in the air space of the tent, and in the ambient air, and two thermocouples were placed halfway into the insulation on the east and west side of the vessel. Twelve days after the grout was poured an additional thermocouple was added in the air space between the vessel stand and the ground. Temperatures were recorded hourly for the first 338 hr after production, then every 8 hr until 1400 hr after production.

In situ thermal conductivity measurements were obtained after the grout set using four probes that were positioned in the receiver vessel prior to grout production. These probes allowed the thermal conductivity of the grout to be measured in situ at different stages of curing. Each probe consisted of

38802-134.1M

FIGURE 2.10. Thermocouple Placement in Receiver Vessel

TABLE 2.1. Thermocouple Identity Numbers and Elevations

Series #	Angle (°)	Identity Number of Thermocouples	Placement Heights from Bottom (in.)
A	0	1-8	3, 15, 27, 39, 51, 63, 75, 84
B	180	9-16	9, 21, 33, 45, 57, 69, 81, 87
C	45	17-20	21, 45, 69, 87
D	135	21-24	3, 27, 51, 75
E	225	25-28	15, 39, 63, 84
F	315	29-32	9, 33, 57, 81
G	90	33-36	15, 39, 63, 87
H	270	37-40	3, 27, 51, 75

a 12-in.-long, 0.375-in.-dia. cartridge heater with a 0.063-in.-dia. type K thermocouple attached at the midpoint of the heater sheath. Each assembly was held in place on the end of a pipe that extended downward into the vessel. The measurement technique is a variant of the line-heat-source method, where

the temperature rise versus $\ln(\text{time})$ for a constant rate of heat generation is related to the thermal conductivity of the medium. A thermal conductivity probe is shown along the left edge of Figure 2.9. The actual measurement probe extends out of the bottom of the supporting pipe.

An acoustic set detector was installed in the vessel to measure changes in the acoustic properties of the grout as curing progressed. This type of measurement is being considered as a remote method of confirming that grout has set as expected in a full-scale vault. The device was suspended from the vessel lid with a chain, as shown in Figure 2.9. The wave speed and attenuation of both longitudinal and transverse waves was measured. A description of the tests with this equipment is described separately.^(a)

Two Eaton® embedable strain gages, number CG-2-155-01F(10)-10-6S, were wired into place in the receiver vessel before the grout was poured. One of the strain gages is shown in the lower right corner of Figure 2.9. The strain gage circuit was excited by a Vishay System 2100® strain gage conditioner and amplifier system. The circuit was wired in a three-wire configuration to minimize the influence of the lead resistance on the measurement. The strain gages were 350-ohm gages with a measured lead wire resistance between the conditioner and gage of 2.2 ohm. The first strain gage was oriented to measure strain in the vertical direction at 28 in. from the center of the vessel and 51 in. from the floor; the second strain gage was oriented to measure strain in the radial direction at 26 in. from the center of the vessel and 46 in. from the floor. As an additional check on the strain, markers were placed in the grout before it set and the distance between them was periodically measured using a tape measure. Changes in grout volume were too small to be detected by this method.

(a) Schuster, G. J. and M. S. Good. 1988. Acoustic Wave Speed Attenuation in Grout. Letter Report to Westinghouse Hanford Company by Pacific Northwest Laboratory, Richland, Washington.

® Eaton Corporation, Automation Products Division, Los Angeles, California.

® Vishay Intertechnology Inc., Instruments Division, Malvern, Pennsylvania.

The amount of separated liquid was checked by visual inspection using three hatches on the top of the vessel. Also, four taps were placed around the base of the vessel. The taps were pipes that penetrated the vessel wall. The end of each pipe was covered with geotextile to prevent the grout solids from entering the taps. Two of the taps were connected to pressure gages with a range of 0-100 in. of water column. The other two taps were connected to tubes that allowed the pressure to be measured by the height of the liquid in the tube. Pressure was periodically measured after the grout had been poured.

2.3 SAMPLING

During the pilot-scale test, the dry blend and simulated DSSF were sampled once per hour. The dry blend sample was taken at the feeder active bin, and the time of the sample was occasionally delayed by several minutes to avoid sampling while the feeder cycle was close to beginning a transfer from the storage bin. The DSSF sample was taken just upstream from the flow meter and control valve. To avoid upsetting the flow controller, the sample was slowly bled from the supply line for about a minute. The waste and dry blend samples were used to make laboratory grouts. Waste samples were also used for chemical analysis.

The mixed grout was sampled every 30 min at the mixer outlet and the bottom of the drop chute. These grouts were immediately used for rheology measurements, then cast for curing in the laboratory. The curing took place in an oven that was periodically adjusted to match the maximum vessel temperature. All of the cured grout samples were used for measurements of compressive strength, density, and corrosivity. The cured grout originating from grout samples taken each hour was used for determining EP toxicity, ANS 16.1 leachability, microstructure, and thermal conductivity. The cured grout in the receiver vessel was sampled by drilling three cores at different radial positions in the grout.

A cold joint in the grout may exist where fresh grout flowed over grout that had already begun to set. To study this possibility, graduated cylinders

were partially filled with samples of grout obtained at the start of the run. At the end of the run, these cylinders were filled the rest of the way with fresh grout.

Plans called for measuring the quantity of separated liquid in the receiver vessel and analyzing samples of the liquid; however, no separated liquid was observed in the pour at the top of the grout. Just after pouring there was only a sheen of liquid on the surface, and as the grout started to cure the liquid was quickly absorbed.

The analysis of the properties of laboratory-mixed and cored grout and the cold joint samples, plus the chemical analysis of the DSSF samples, are presented in Lokken et al. (1992).

2.4 TEST MATERIALS

The dry blend used in this pilot-scale test consisted of a blend of 47% blast furnace slag (Blaine fineness 599 m²/kg, grade 120), 47% Class F fly ash (from Centralia Power Plant), and 6% type I,II portland cement. The materials were weighed, placed in a cement mixer, and mixed by turning the mixer 900 revolutions. The blended, dry materials were then placed in hoppers dedicated to dry blend storage. Two batches of dry blend were mixed, and Westinghouse analyzed a sample from the second batch using quantitative Fourier transform infrared (FTIR) spectrometry. The results of the analysis, which are based on three measurements, indicate that the dry blend was well mixed (Table 2.2).

TABLE 2.2. Analysis of Dry Blend Samples

<u>Component</u>	<u>Weight Percent</u>	
Blast furnace slag	47.03	±0.30
Fly ash	46.65	±0.18
Cement	5.97	±0.88
Moisture	0.73	±0.02

The waste makeup procedure for the simulated DSSF used in the run is provided in Appendix A. The procedure was modified from the procedure provided by Westinghouse for making DSSF in the laboratory, in that the addition of $\text{Al}(\text{NO}_3)_3$ was delayed until after the addition of part of the NaOH . This modification was necessary because the solution gels as the acidic solution is neutralized.

Table 2.3 lists the target concentrations and the analyzed concentrations. The majority of the concentrations were determined using inductively coupled plasma (ICP) spectroscopy and ion chromatography.

TABLE 2.3. Measured and Target Waste Concentrations^(a)

<u>Species</u>	<u>Analysis, g/L</u>	<u>Target, g/L</u>
Al	22.4	20.3
B	0.136	0.105
Ba	0.60	0.623
Ca	0.573	0.200
Cr	1.26	1.15
Fe	1.49	1.41
K	11.5	9.72
Mg	0.320	0.00
Mn	3.01	2.75
Mo	0.068	0.049
Na	122	121.8
P	2.02	1.84
Si	0.502	0.56
Zn	2.93	1.63
Cl-	5.36	3.86
NO_2^-	27.2	23.0
PO_4^{3-}	5.4	5.65
NO_3^-	186	154.4
SO_4^{2-}	5.1	5.05
TOC (ppm)	1556.00	1,280.00 ^(b)

(a) DSSF makeup recipe from Claghorn (1987).

(b) Calculated from EDTA and citric acid additions.

The results from analysis of the waste agreed fairly well with the target value for the waste for most constituents. The disagreement for Ca and Mg are believed to be due to the mineral concentrations in the process water used in the makeup. The reason for deviations in the B, Mo, Zn, and Cl concentrations is not known, but they may be due to impurities in other chemicals. Most constituents tended to be slightly higher in concentration than the target.

3.0 RESULTS

This chapter describes the results obtained from the pilot-scale production of simulated DSSF grout. The results are presented in the following order: grout processing results, post-run equipment observations, and post-run receiver vessel observations.

3.1 GROUT PROCESSING

The pilot-scale production of DSSF grout occurred between approximately 9:30 a.m. and 3:00 p.m. on November 14, 1988. During this 5.5-hr period the actual time of grout production was 4.5 hr, as determined from the operational time for the dry blend feeder. Grout processing was very successful and for most of the processing time operation was smooth. The feed rates were accurately maintained at the desired levels during processing.

The only problems occurred between 70 and 150 min of run time, when data being collected by the data logger were lost. On one occasion the printer was accidentally turned off, which created an error that halted execution of the program being used to print the data on the printer and record data on disk.

The second problem occurred at a run time of 80 min, when the inlet supplying both the primary and back-up DSSF delivery pumps became partially clogged. This forced an unplanned shutdown of 47 min while the line was back-flushed and the operability of the pumps was confirmed. When the process was restarted, a valving error resulted in the delivery of water instead of DSSF to the process for about 6 min. When this error was noticed, the remaining grout in the surge tank was sent to the alternate receiver bin. An estimated 40 gal of water grout was delivered to the receiver vessel.

The instantaneous mix ratio is plotted in Figure 3.1. The blank portion of the plot indicates the time the data logger was accidentally turned off. This period includes the time the process was shut down. Note that for the vast majority of time the mix ratio was maintained within a deviation of only ± 0.3 lb/gal. In all cases the deviation was less than ± 0.5 lb/gal, which is

FIGURE 3.1. Grout Processing Mix Ratio

the deviation that the grout formulation must be able to tolerate for processing in the TGE.^(a) The average of the instantaneous readings for mix ratio was 9.08 lb dry blend/gal waste, compared to the target mix ratio of 9.00 lb/gal.

The processing temperatures are plotted in Figure 3.2. The data logger had a problem in the ROM software used for determining thermocouple temperatures. Occasionally, the measurement of the cold junction temperature jumped by one degree, either positive or negative, which moved all temperatures one degree. This jump can be seen in Figure 3.2 at 220 minutes, where all temperatures jump 1°C and then jump back down. It appears that before the shutdown, the temperatures may have been at their "high" level (0 to 70 min) and assumed the "low" level after the data logger was restarted (140 to 310 min). It is

(a) Riddelle, J. G. 1987. *Formulation Criterion for the Hanford Grout Disposal Program*. Internal Report SD-WM-SS-004, Westinghouse Hanford Company, Richland, Washington.

FIGURE 3.2. Process Temperatures During Production

also possible that flushing the DSSF pump inlet caused an approximate 1°C drop in the DSSF temperature due to mixing the cold process water with the warm simulated waste.

The grout pumping parameters, discharge pressure, pump speed, and grout flow rate are shown in Figure 3.3. The data are presented in relative magnitude to the average of the data over the run. The average of the instantaneous readings over the run are pump speed = 249 rpm, grout flow rate = 10.44 gpm, and discharge pressure = 75 psi. Therefore, a reading of 6% deviation for pressure in Figure 3.3 would indicate a pressure of $1.06(75) = 79.5$ psi. The fluctuations in discharge pressure do not indicate changes in the process. To maintain the level of grout in the surge tank the speed of the grout pump was manually adjusted, and the large initial variations were caused by changing the pump speed by larger increments than necessary. After the process had been run for a while it was determined that the grout level in

FIGURE 3.3. Grout Pumping Parameters

the surge tank was slightly decreasing, but nearly steady, at 250 rpm. The pump was left at 250 rpm and occasionally dropped to 230 rpm to restore the level after the level had decreased significantly.

The density of the grout is directly related to the mix ratio, with higher mix ratios producing a denser grout slurry. Samples of the grout were taken from the process at the exit of the mixer and at the discharge from the grout pipe. The density and rheological properties were measured for these samples. Table 3.1 presents the average of the pilot-scale densities, as compared to the average of the densities determined by mixing the components in more controlled laboratory conditions. Results are listed as an average for all the samples, and as an average with two sample points eliminated. One of the points eliminated was for a sample taken at the very beginning of the run, which is believed to have been influenced by the start-up procedure where the waste is started through the mixer before the dry blend to avoid plugging the mixer. This waste collects in the surge tank and results in initial

TABLE 3.1. Comparison of Laboratory and Pilot-Scale Grout Densities

<u>Type of Grout</u>	<u>Average Density, (lb/gal)</u>	<u>Standard Deviation</u>
Laboratory	13.89	$\sigma=0.07$, $n=8$
Pilot-scale	13.80	$\sigma=0.20$, $n=18$
Pilot-scale (2 points deleted)	13.85	$\sigma=0.08$, $n=16$

production of thin grout. The second point deleted was for a sample taken during the 6 min of water grout production. With those two data points eliminated, the pilot-scale densities are in excellent agreement with the densities of samples mixed in the laboratory. The standard deviation of the pilot-scale grout density with the two points eliminated is surprisingly small.

The grout pipe was a 3/4-in., schedule 40, steel pipe that was 170-ft long and contained 14 4-in. radius bends. The rheology of grout samples taken at the entrance and exit of the grout pipe was measured using a Fann viscometer. The rheology is characterized as a flow consistency index (K' lb_f-sec ^{n'} /ft²) and a flow behavior index (n' unitless). These two parameters describe the fluid behavior in a power-law fluid model (Smith 1976).

$$S_s = K' (S_r)^{n'}$$

Where: S_s = shear stress (lb_f/ ft²)
 S_r = shear rate (sec⁻¹)
 K' = flow consistency index (lb_f-sec ^{n'} /ft²)
 n' = flow behavior index (unitless)

K' is analogous to the viscosity for a Newtonian fluid; n' indicates the deviation from Newtonian behavior. For Newtonian fluids $n'=1.0$ and if $n' < 1.0$, then the fluid is pseudoplastic and the apparent viscosity is lower

at higher shear rates. A discussion of the calculations involved in the rheology is found in Dodge and Metzner (1959), Slagle (1962), and Smith (1976).

In this pilot-scale test it was found that the grout was more viscous at the exit of the mixer than at the discharge from the grout pipe. Typical rheological parameters for laboratory-mixed grouts are $n' = 0.7973$ and $K' = 0.0020$; for pilot-scale grout at the mixer discharge, $n' = 0.6594$ and $K' = 0.0112$; and for pilot-scale grout at the grout pipe exit, $n' = 0.8346$ and $K' = 0.0016$. The expected pressure drop in the grout pipe was calculated based on the rheological properties of samples obtained at the mixer discharge and the grout pipe exit. To predict the pressure drop, the non-Newtonian Reynolds number was calculated (Smith 1976; p. 109) and the friction factor was obtained from Figure 12 in Dodge and Metzner (1959). The predicted pressure drop for each grout sample was compared to the measured pressure drop recorded the closest in time to the sample that was taken. Pressure measurements were taken every 5 min, so the greatest difference between the rheology sample time and the pressure measurement was 2.5 min. The comparison is shown in Figure 3.4. Note that in almost all cases the pressure drop is underestimated by the rheology at the pipe discharge point, and overestimated by the rheology at the mixer discharge. This trend suggests the rheology of the grout is time-dependent and tends to thin the longer it is sheared.

The data in Dodge and Metzner (1959) were obtained using smooth brass pipes. The effect of pipe roughness on pressure drop for non-Newtonian fluids in pipe flow is not known. However, in Newtonian fluids, the friction factor has little dependence on pipe roughness up to a Reynolds number of 3000. In addition, the dependence on pipe roughness is less at low Reynolds numbers than at high Reynolds numbers. Therefore, application data from Dodge and Metzner (1959) to calculate the pressure drop in the grout pipeline may only slightly underpredict pressure drops due to the differences in pipe roughness.

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FIGURE 3.4. Measured Pressure Drops Compared to Predictions. Measurements based on rheology of grout slurry at surge tank and grout pump exit.

It should be noted that a friction factor of .008 for pressure drop calculations, as recommended in the grout formulation criteria,^(a) is not conservative. For a grout with a flow consistency index of 0.8, the friction factor is 0.0094 at a Reynolds number of 3200 (Dodge and Metzner 1959).

The second article on which the grout formulation criteria is based (Slagle 1962) is probably less applicable, since the data were gathered using bentonite water suspensions and emulsions to simulate cements with n' values between 0.19 and 0.42. The grout slurry in the pilot-scale test had a n' of 0.66 at the surge tank and 0.84 at the grout pipe exit. Therefore, the grout

(a) Riddelle, J. G. 1987. Formulation Criterion for the Hanford Grout Disposal Program. Internal Report SD-WM-SS-004, Westinghouse Hanford Company, Richland, Washington.

had a more Newtonian (i.e., less pseudoplastic) behavior than the slurries discussed in Slagle (1962). Slagle states that the slurries departed from laminar flow at a Reynolds number of 2100. This finding disagrees with the finding by Dodge and Metzner (1959) that the onset of turbulence is delayed in non-Newtonian fluid flow. The critical flow rate for transition to turbulent flow has been assumed to be at a Reynolds number of 2100 for purposes of this report. This may be nonconservative if the onset of turbulence is delayed due to non-Newtonian effects. The turbulent flow is required to prevent buildup in the grout pipe.

The implications of the pilot-scale rheology are shown in Figures 3.5, 3.6, and 3.7. These figures show the predicted pressure drop and Reynolds number as a function of the grout production rate. The constraints on the plot show the requirement of achieving turbulent flow (designated F_c in the plots) in the grout pipe and the maximum allowable pressure drop (designated F_p in the plots). The flow rate between these constraints is labeled "allowable operating range." The GTF grout pump is designed to operate at flow rates between 50 and 70 gpm.^(a) However, the grout pump can operate over a wider flow range. It should be noted that the GTF may not be able to operate on a continuous basis above 75 gpm due to the maximum production capacity of the DMF.

Figure 3.5 shows the prediction of GTF performance based on laboratory-mixed grout samples. Figure 3.6 shows the same predictions based on the rheology determined from samples taken at the grout pipe exit. These two plots are very similar, and indicate that there is a wide range of grout flow rates over which the GTF could be operated. Figure 3.7 shows the same information based on the rheology determined from the pilot-scale mixer discharge samples. With this rheology the allowable operating range is very narrow. The grout must be produced at a rate of 65 gpm to produce turbulent flow, and cannot exceed 73 gpm and remain below the pressure drop criteria of

(a) Riddelle, J. G. 1987. Formulation Criterion for the Hanford Grout Disposal Program. Internal Report SD-WM-SS-004, Westinghouse Hanford Company, Richland, Washington.

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FIGURE 3.5. Predicted GTF Operating Constraints Based on Rheology of Laboratory-Mixed Grout Samples. F_c is minimum flow to achieve turbulent flow and F_p is maximum flow to stay within pressure drop criteria.

11.2 psi/100 ft, as established in the grout formulation criteria. However, in the GTF the surge tank is agitated, and the agitation may make the grout less viscous before its entry into the grout pump, thus reducing the critical flow rate and widening the allowable operating range.

Also, some relief from the pressure criteria may be possible depending on the vault location and the length of the grout pipe. However, 70 to 75 gpm is the approximate maximum production capacity of the GTF.

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FIGURE 3.6. Predicted GTF Operating Constraints Based on Rheology of Pilot-Scale Grout Samples Taken at the Grout Pipe Exit. F_c is minimum flow to achieve turbulent flow and F_p is maximum flow to stay within pressure drop criteria.

FIGURE 3.7. Predicted GTF Operating Constraints Based on Rheology of Pilot-Scale Grout Samples Taken at the Surge Tank. F_c is minimum flow to achieve turbulent flow and F_p is maximum flow to stay within pressure drop criteria.

3.2 POST-RUN PROCESS EQUIPMENT OBSERVATIONS

The day following completion of the run the process equipment was inspected to determine if any problems were evident from the condition of the equipment. Increased wear was noted on the tip of the first mixer paddle in the grout mixer, where the dry blend and waste first begin to mix (Figure 3.8). The amount of wear was quantified by measuring the increase in the width of the worn tip on the mixer. Prior to this pilot-scale test the maximum width of the worn section was 0.24 in., which represented the wear that resulted from the previous pilot-scale production of PSW grout (Fow et al. 1987). After the DSSF grout production, the width of this worn portion had increased to 0.31 inches. The mixer to be used in the full-scale operation has stellite tips, which will be more resistant to wear; however, this problem may still be of concern since the wear during this pilot-scale

FIGURE 3.8. Wear on Paddle After Production

test occurred in only 4.5 hr. As the tip wore down, a ridge of grout accumulated in the increased clearance. This ridge is shown more clearly in Figure 3.9, which is a view of the removable top of the mixer. The ridge of grout in Figure 3.9 corresponds to the location of the worn paddle in Figure 3.8.

Substantial buildup of grout was noticed in the discharge chute of the mixer (Figure 3.10). In the GTF, this type of buildup may cause problems over a long period of time. An occasional water flush of this area may be advisable. A similar but less severe buildup was noticed on the surge tank wall next to the discharge pipe (Figure 3.11). This buildup was caused by splashing of the grout as it entered the tank, and could also cause a substantial mass of grout to accumulate over a long period of time. A non-splashing grout entry or periodic flush of this area may be useful. The bottom of the surge tank, which slopes at about 30° , was kept clear by stirring the tank bottom approximately every 15 min. Buildup could be felt in the bottom of the tank during this procedure. The surge tank in the GTF is agitated and slopes at about 70° . The grout has a lower residence time in the

FIGURE 3.9. Grout Ridge on the Inside of the Mixer Lid. Ridge corresponds to the location of the worn paddle.

FIGURE 3.10. Buildup in the Discharge Chute of the Grout Mixer

FIGURE 3.11. Buildup on the Surge Tank Wall Above Liquid Level

GTF than it does in the pilot-scale surge tank. Agitating the GTF surge tank will likely prevent buildup of grout in the tank bottom.

At the conclusion of the grout run the surge tank was pumped dry, and the mixer, surge tank, and piping were flushed with about 10 gal of water. The flush water was applied in three approximately equal batches. The first batch was sent through the mixer while the mixer was running, the second was introduced as a spray to clean the surge tank, and the third was a repeat of the spray of the surge tank, which resulted in relatively clean water. After each batch of flush water, the surge tank was pumped dry at a pump speed of 250 rpm (~ 10 gpm). The flush proved to be insufficient. Figure 3.12 is a view into the end of the grout pipe approximately one-third the distance to the vessel. The walls of the pipe are clean, indicating that the grout was not building on the walls during the run; however, the bottom of the pipe contains grout solids that settled in the pipe after the flush was complete.

FIGURE 3.12. Grout Pipe the Day After Production. Note the small amount of grout in the bottom of the pipe.

The pipe was reassembled and was not flushed further. Approximately 4 months later, difficulties were encountered when pumping water through the pipe. Pressures rose above 114 psi, which tripped the high-pressure cutout relay and shut off the grout pump. Violently banging on the pipe with pipe wrenches cleared the pipe, although it occasionally replugged and produced sustained pressures of 90 psi at a flow of 8 gpm. Finally, the grout pipe had to be replaced. Examination of the pipe showed that the maximum deposits of grout ($\approx 1/8$ in.) occurred near the middle of the group pipe. Most of the pipe did not have any deposits. The severe effect of the small amount of grout in the line was unexpected. It is speculated that pieces of grout broke loose from the pipe bottom and bridged the pipe opening.

The proper method for scaling the amount of flush water required is not well defined. It seems reasonable that the flush should be performed at a flow rate that will produce well-established turbulent flow. One way to scale the flush water is to add flush water equal to 1 pipe volume, plus some subsequent duration of flow at or above a certain Reynolds number. The 1 pipe volume removes the grout plug from the pipe, and the duration of flow removes any material remaining on the walls of the pipe. This method is preferable to

scaling by a factor of 2 or 3 pipe volumes, since as the pipe gets longer the multiple pipe-volume method results in more and more flushing of the line. The flush used in the pilot-scale test was nominally equal to 1 pipe volume plus 30 sec of flush at a Reynolds number of 38,000 (assuming rheology of water). It should be noted that the flush of the pilot-scale pipe was considered insufficient to clean the pipe. Without more data, a flush of 1 pipe volume plus 60 sec of flow at a Reynolds number of 38,000 is recommended for the GTE.

The progressive cavity grout pump was disassembled several months after the run to check for buildup of grout in the pump inlet and the pump exit. Grout was found in the bottom of the inlet. The majority of this material was extremely friable and was swept away when lightly brushed. Underneath the first layer of very friable grout was another layer of harder material, which was partially chipped away to show the depth (Figure 3.13). It is believed that the majority of the grout did not build during the run. It is possible that after the flush was complete, some grout remained on the walls of the 4-in. hose leading to the grout pump suction. This grout may have flowed on

FIGURE 3.13. Grout Pump Inlet Disassembled Months After Run

the walls of the tube after the flush and collected in the pump suction area. The grout pump discharges into a 2.5-in. pipe, which quickly reduces to a 3/4-in. pipe. No buildup was seen in the exit of the grout pump.

3.3 POST-RUN RECEIVER VESSEL OBSERVATIONS

This section discusses the observations of the grout after it was placed in the vessel. The discussion focuses on separated liquid, strength development, temperature rise during curing, dimensional changes, and thermal conductivity in hardened grout.

3.3.1 Separated Liquid

The top of the grout was inspected for the presence of separated liquid. At the conclusion of the run at 4 p.m., the surface of the grout was covered by a very thin film of liquid. At 9 a.m. the next morning, liquid was not present on top of the grout.

Around the base of the vessel were four wall penetrations covered with geotextile and connected to either a tube or gauge to determine the pressure of the liquid in the vessel (see section 2.2.2 for description). During the run, the level in the tubes followed the level of the grout fairly closely so that at the conclusion of the run, the level in the tubes was approximately 85 in. By 9:30 a.m. the following day, the level was down to only 25.5 in. in the tubes and 26.6 in. water column on the gauges. Note that the specific gravity of the DSSF waste is 1.26. Therefore, the equivalent tube reading in inches of water column would be $(25.5 \text{ in.}) \times 1.26 = 32 \text{ in.}$ By 11:00 a.m. the second day after the run, both the tubes and the gauges read zero.

Three months after the grout was poured, samples of the grout were taken by core drilling for subsequent characterization. As part of the coring procedure, water was used on top of the vessel. At the time of coring, the tubes had been cut off and were open to drain any liquid that may have entered the pipe penetrating the wall. The water pressure from the water on top of the grout was transmitted to the pressure gauges in pressures up to 90 in. water. The available flow volume with the valve open was on the order of a few milliliters. When the valve was closed, the pressure built in only a few

minutes. It appears that there may be some cracks in the grout that are transmitting the water to the pressure gauges, although the cracks are not sufficient to drain the water to the tubes.

3.3.2 Strength Development

After a few hours in the receiver vessel the grout was like a non-hardened paste. Markers could be easily pushed into the grout. A concrete penetrometer failed to register a reading, meaning the strength was less than 50 psi. Twenty hours after production, the grout had achieved a penetration resistance of 575 psi. Twenty-five hours after production the penetration resistance was greater than 750 psi, which is the maximum measurement on the penetrometer.

3.3.3 Temperature Rise During Curing

The grout in this pilot-scale test produced a temperature rise due to hydration that was greater than expected. Figure 3.14 shows the actual temperature near the center of the mass and a predicted adiabatic temperature (predicted by heat loss calculations) at the center of the vessel. Appendix B provides details on the heat loss calculation used to predict the adiabatic temperature rise. Plots providing the temperature distribution within the grout are provided in Appendix C.

Previous calorimetry runs indicated that the adiabatic temperature rise was on the order of 50°C, although the adiabatic temperature rise in the pilot-scale test is estimated to have been 83°C. The reason for the disagreement is that the heat generated over long periods of time is due to a significant portion of the total heat evolved, and the calorimetry was not precise enough to accurately measure the evolution of heat after it slowed to a low generation rate.

3.3.4 Dimensional Changes in Hardened Grout

The dimensional changes in the grout were small and varied between 0.027% increase and 0.11% decrease. The dimensional stability of the grout was monitored by placing strain gages in the grout. Markers were also pushed into the surface of the grout and the distance between markers monitored using

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FIGURE 3.14. Predicted Adiabatic Temperature Rise and Actual Observed Temperature in Pilot-Scale Monolith

a tape measure. However, volume changes were too small to be detected by this method. The two strain gages were oriented so that one measured the deformation in the vertical direction and the other measured deformation in the radial direction. The deformation in the grout was essentially the same in both orientations. A loose connection in the output of the strain gage conditioner introduced some uncertainty in strain data at early times, but the grout did show a maximum increase in dimension of approximately 270 microstrains, or 0.027% at 166 hours. The dimensional increase is smaller than might be expected from thermal expansion due to the rising temperature of the grout. After the small expansion, the grout shrank until it reached 1100 microstrains shrinkage (0.11%). This maximum shrinkage was recorded when the grout near the strain gages had cooled to approximately 24°C. The maximum shrinkage would amount to less than 1/2-in. subsidence over a 34-ft-high vault. Since this volume change occurred over a fairly short time period, the fraction of subsidence not due to temperature changes would be compensated

during placement of the cold grout cap. The measurements of strain reported here were taken over a period of about 3 months.

3.3.5 Thermal Conductivity

The thermal conductivity measurements are summarized in Table 3.2. Based on an accuracy of $\pm 10\%$, the measurements did not show any correlation to time (temperature and stage of curing) or position. The average measured thermal conductivity was 0.44 BTU/hr ft F° (0.76 W/mK).

3.3.6 Acoustic Properties

Acoustic measurements were successful in measuring increases in sonic velocity as the grout set, and also provided data on the attenuation of the signal over distance. Transverse waves could be transmitted through the cured grout but were not transmitted through the grout slurry. Results indicated that the acoustic measurements are a possible method of determining that the grout has set. The results of this work are discussed in detail in a separate report.^(a)

3.3.7 Restart Pressures

Several months after the pilot-scale grout production test an additional test was performed to measure grout restart pressures using simulated waste that remained from the initial test. The restart pressures were measured in

TABLE 3.2. In Situ Thermal Conductivity Measurements (BTU/hr ft F°)

<u>Probe Number</u>	<u>Day 2</u>	<u>Day 4</u>	<u>Day 7</u>	<u>Day 14</u>
1	0.45	0.45	0.44	0.43
2	0.45	0.44	0.46	0.46
3	0.43	0.43	0.44	0.42
4	0.44	0.45	0.45	0.39

(a) Schuster, G. J. and M. S. Good. 1988. Acoustic Wave Speed Attenuation in Grout. Letter Report to Westinghouse Hanford Company by Pacific Northwest Laboratory, Richland, Washington.

a new 110-ft long, 3/4-in., schedule 40 steel pipe. The simulated waste was delivered at 34°C, which is below the temperature of 43°C for the previous run. It should be noted that the gel strength will be dependent on temperature. Three data points were obtained and are summarized in Table 3.3.

In the first data point, a sample was taken from the pipe exit for rheology and gel strength measurement. The grout pump was then shut down. After 10 min the grout pump was restarted at 200 rpm (~8.3 gpm) and the maximum pressure of 41 psi was observed. For the second measurement the same procedure was used, although the grout was allowed to sit in the pipe for 20 min instead of 10 min. The gel strength measurement also corresponds to 20 min. For the third measurement the grout was allowed to sit in the pipe for 10 min as in the first test, but the flow was restarted. Pressure was applied by very slowly opening a flush water valve.

As shown in Table 3.3 the pressure required to clear the line is highly dependent on the flow rate that is used. Also, the gel strength and pressure required to clear the line increases substantially between 10 and 20 min. The equation used to predict the restart pressure appears to underestimate the

TABLE 3.3. Restart Pressure Data from Second Pilot-Scale Production Test

Number	Gel Strength (a) (lb ^f /100 ft ² - minutes)	Predicted Pressure (b) (pilot) (psi)	Measured Pressure (c) (pilot) (psi)	Restart Method (d) (Pump/water)	GTF Predicted (e) (Equation, psi)	GTF (f) Predicted (Pilot result, psi)
#1	37 - 10 min	16.4	41	pump	71.6	179
#2	135 - 20 min	59.9	95	pump	261	415
#3	85 - 10 min	37.7	15	water	165	66

- (a) Gel strength as measured using a Fann viscometer after allowing the grout to stand for the time listed.
- (b) Pressure predicted from gel strength for pilot-scale restart using equation presented in grout formulation criteria documentation.
- (c) Actual maximum pressure observed in pilot-scale test.
- (d) pump = grout pump was restarted at 200 rpm to restart flow water = flush water pressure was slowly applied to line until flow obtained.
- (e) Pressure predicted for GTF restart based on gel strength measurement and using equation for 1200 ft of 2-in. schedule 40 pipe.
- (f) Pressure predicted for GTF from pilot-scale restart pressure, taking into account the surface and cross-sectional area relationships for the two pipes. Based on 1200 ft of 2-in. schedule 40 pipe.

pressure when flow is started quickly and overestimate the pressure when flow is started very slowly. For comparison, two measurements of laboratory-mixed pilot-scale materials with a waste temperature of 40°C - 45°C produced 10-min gel strengths of 30 and 65 lb_f/100 ft². Sample #3 had a higher gel strength and higher apparent viscosity than expected. The reason for this is not known.

Based on these results it is believed that restart pressures after 10 min will be below the 500 psi limit for the GTF grout pump. Also, it is advisable to start the pump at a slower speed and then accelerate to avoid excessive pressures.

4.0 CONCLUSIONS AND RECOMMENDATIONS

This section summarizes the conclusions and recommendations from the pilot-scale test of simulated DSSF grout.

4.1 RESULTS

The objectives of the pilot-scale test were successfully met. A rise in temperature of 61°C was observed in the grout, and an adiabatic rise of 83°C was estimated. These temperatures would correspond to heats of hydration of 79.3 cal/g dry blend and 107.9 cal/g dry blend, respectively. Measurements of thermal conductivity showed no definite change over time and averaged 0.76 W/mK (0.44 BTU/hr ft F°). After becoming solid the grout showed only very small dimensional changes. Maximum linear change was a 0.11% shrinkage. Short-term volume changes are not a problem. The grout gained sufficient strength within 20 hr of production to meet waste form criteria when poured at 40°C. The time required will be dependent on temperature. The grout at the mixer discharge was considerably thicker than at the pipe discharge. No particular effect on the grout as a result of the 34-ft fall in the drop chute was noted; however, further analyses of the cured grout properties are required. The grout formulation produced essentially no separated liquid. Therefore, little if any liquid will need to be recovered from a vault for reprocessing. The required samples were obtained for characterization of the grout waste form, and the results of the characterization will be presented in a separate report. Acoustic measurements were successful in detecting changes as the grout set.

If the grout pump in the GTF shuts down for 10 min with grout in the grout pipe, it should be able to reinitiate flow in 1200 ft of 2-in. schedule 40 pipe without exceeding the 500 psi pressure limit.

A combination of grout buildup and wear on the first mixer paddle in the grout mixer is a cause for concern, since excessive wear developed after only 4.5 hr of operation. The mixer in the GTF has stellite tips, which will reduce wear. There is not a clear impact on grout processing from substantial

wear in the mixer. Minor reductions in mixing efficiency should be compensated by the agitation of the surge tank.

4.2 RECOMMENDATIONS

The rheology measurements indicated that the grout became less viscous as it was pumped through the grout pipe. This thinning presumably is due to shear experienced during flow of the grout in the pipe. It is possible that the agitator in the GTF surge tank will produce sufficient shearing to thin the grout to near the consistency measured at the grout pipe exit in the pilot-scale test. If the agitator significantly thins the grout, the allowable operating range will be very broad (30 to 75 gpm of grout). However, if the agitator does not thin the grout, the allowable operating range will be very narrow (65 to 73 gpm of grout). Since the effect of the agitator cannot be verified without further testing, it is recommended that the GTF pump grout at 65 to 70 gpm to ensure turbulent flow and that the surge tank agitator be operated.

The temperature rise during curing of the grout was measured at 61°C. The adiabatic temperature rise during curing has been estimated at 83°C. Since the grout will be poured in vaults that are 125 ft X 50 ft X 34 ft, the conditions will be close to adiabatic near the center of the vault. The highest expected feed temperature is 45°C, which would result in an adiabatic grout temperature of 128°C. However, the temperature at the grout surface probably would not exceed 105°C due to evaporation of the pore fluid. Currently, the maximum allowable grout temperature is 90°C. To reduce the temperature of the grout during curing, a heat exchanger could be installed either at the waste feed tank, the waste feed line to the mixer, or in the grout pipe prior to entering the vault. A heat exchanger for the DSSF may be able to drop the temperature to approximately 20°C to 25°C. Ventilating the grout vault during filling will remove additional heat by promoting evaporation of water from the grout surface. Formulation changes may also be required.

Further testing is recommended to accurately determine the flush required to clear the line. In the absence of further testing, the

recommended flush of the grout pipe is at least 1 pipe volume plus 60 sec of flow at a minimum Reynolds number of 38,000 (based on water). Higher flushing flow rates are desirable. This recommendation is based on a pilot-scale flush (1 pipe volume plus 30 sec flow at a Reynolds number of 38,000) that proved insufficient, so it is not known if this flush will produce the desired result.

Grout buildup was noticed in the discharge of the mixer. A periodic flush of this area is recommended. A non-splashing entry is recommended for the surge tank. Agitation of the surge tank is required to prevent buildup in the bottom of the tank.

When restarting the grout pump after a shutdown, it is recommended that the pump be started at a low speed and then accelerated to avoid high restart pressures.

The hardened grout in the pilot-scale receiver vessel should be broken apart and examined. Variations in the properties of grout in different locations within the receiver vessel should be measured. Samples should be taken during this procedure and archived for possible future tests.

5.0 REFERENCES

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APPENDIX A

MAKEUP PROCEDURE FOR SIMULATED DSSF

APPENDIX A

MAKEUP PROCEDURE FOR SIMULATED DSSF

The simulated waste was made in two batches. The first batch of 200 gal was made in a small tank to test the procedure and ensure that problems would not be encountered when making the larger batch. For convenience, small-quantity chemicals were omitted from this batch. The second batch consisted of 2500 gal. The small-quantity chemicals were added to this batch to make up for their omission from the small batch. The 200-gal batch was added to the 2500-gal batch after the makeup was complete.

The procedure used is the same as that used to produce the simulant in the laboratory, except that the AlNO_3 solution was added after the solution had been made caustic to prevent gelling of the solution during neutralization. The data sheets for each of the batches are presented on the following pages.

Simulated DSSF Waste Makeup Procedure (10-9-88 revision)

200 gallon batch

<u>DATE</u>	<u>TIME</u>	<u>SIG</u>	<u>INGREDIENTS/DIRECTIONS</u>	<u>IDENTIFICATION (MFR., GRADE, LOT#)</u>
_____	_____	_____	1) 36.6 gallons (305 lb) process water to tank 70.	
_____	_____	_____	2) Turn on the agitator and add the following chemicals.	
_____	_____	_____	7.9 kg/ 17.4 lbs $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	_____
_____	_____	_____	13.5 kg/ 29.8 lbs $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	_____
_____	_____	_____	11.2 kg/ 24.7 lbs $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (50%)	_____
_____	_____	_____	1.3 kg/ 2.8 lbs $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	_____

CONTINUED, PAGE 1 OF 3

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

200 gallon batch

DATE TIME SIG INGREDIENTS/DIRECTIONS IDENTIFICATION (MFR., GRADE, LOT#)

____ 3) add 10 gallons (83.5 lb) water and slowly add the
following chemicals

____ 5.17 kg/ 11.4 lbs H_3PO_4 _____

____ 4.20 kg/ 9.26 lbs H_2SO_4 _____

____ 1.31 kg/ 2.9 lbs KF _____

____ 2.53 kg/ 5.6 lbs $Na_2Cr_2O_7 \cdot 2H_2O$ _____

____ 4) add 10 gallons (83.5 lb) water and dissolve the
following chemicals

____ 4.42 kg/ 9.7 lbs NaCl _____

CONTINUED, PAGE 2 OF 3

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

200 gallon batch

DATE TIME SIG INGREDIENTS/DIRECTIONS IDENTIFICATION (MFR., GRADE, LOT#)

____ 5) Slowly add the following chemicals to tank 70. Monitor the temperature and when it reaches 95°C discontinue addition. Restart addition when temperature falls to 90°C.

____ 10.93 kg/ 24.1 lbs KOH _____

____ 135.5 kg/ 298.7 lbs NaOH _____

____ 6) Slowly add the aluminum nitrate solution.

____ 371.8 kg/ 819.6 lbs $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (57.2%) _____

____ 7) Add the following chemicals to tank 70.

____ 26.76 kg/ 59.00 lbs NaNO_2 _____

____ 12.01 kg/ 26.5 lbs Na_2CO_3 _____

____ 2.75 kg/ 6.1 lbs EDTA
(tetra sodium salt trihydrate) _____

____ 1.56 kg/ 3.4 lbs citric acid
(tri sodium salt dihydrate) _____

____ 3.00 kg/ 6.6 lbs colloidal silica (HS-30) _____

____ 11) Adjust the volume to the 200 gallon level. Record the weight of water added.

____ 13) After the solution has reached 113°F readjust the volume to 200 gallons by adding additional water. Leave the agitator on low to maintain solids in suspension.

SCALE USED

CALIBRATION #

EXPIRATION DATE

PAGE 3 OF 3

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

FINAL REVISION, INCLUDES ADDITIONS FOR 200 GALLON BATCH CHEMICALS

Simulated DSSF Waste Makeup Procedure (10-13-88 revision)

2500 gallon batch

DATE TIME SIG INGREDIENTS/DIRECTIONS IDENTIFICATION (MFR., GRADE, LOT#)

_____	_____	_____	1) 457 gallons (3,814 lb) process water to tank 69.
_____	_____	_____	2) Turn on circulation pump, and agitator.
_____	_____	_____	3) Add the following chemicals to tank 69 (all should be soluble).
_____	_____	_____	222.1 g $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ _____
_____	_____	_____	42.0 g $\text{Pb}(\text{NO}_3)_2$ (98%) _____
_____	_____	_____	51.8 g $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ _____
_____	_____	_____	2.61 kg (5.75 lb) AgNO_3 _____
_____	_____	_____	98.4 kg (216.9 lb) $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ _____
_____	_____	_____	169.1 kg (372.9 lb) $\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ _____

PAGE 1 OF 6

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

FINAL REVISION ALL QUANTITIES ADDED FROM SMALL BATCH

Simulated DSSF Waste Makeup Procedure (10-13-88 revision)

2500 gallon batch

DATE TIME SIG INGREDIENTS/DIRECTIONS IDENTIFICATION (MFR., GRADE, LOT#)

____ 140.0 kg (308.65) $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (50%) _____

____ 1.50 kg (3.31 lb) $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ _____

____ 16.1 kg (35.6 lb) $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ _____

____ 9.45 kg (20.83 lb) $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ _____

____ 4) in tank 70, add 125 gallons (1041 lb) water and add the following chemicals

____ 64.6 kg (142.5 lb) H_3PO_4 _____

____ 52.5 kg (115.7 lb) H_2SO_4 _____

____ 0.960 g As_2O_5 _____

____ 62.35 g SeO_2 _____

____ 747.8 g MoO_3 _____

PAGE 2 OF 6

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

FINAL REVISION ALL QUANTITIES ADDED FROM SMALL BATCH

Simulated DSSF Waste Makeup Procedure (10-13-88 revision)

2500 gallon batch

DATE TIME SIG INGREDIENTS/DIRECTIONS IDENTIFICATION (MFR., GRADE, LOT#)

_____	_____	_____	5) Transfer contents of tank 70 to tank 69.
_____	_____	_____	6) Add the following chemicals to tank 69.
_____	_____	_____	16.4 kg (36.11 lb) KF _____
_____	_____	_____	163.4 g CuSO ₄ _____
_____	_____	_____	31.6 kg (69.7 lb) Na ₂ Cr ₂ O ₇ *2H ₂ O _____
_____	_____	_____	7) in tank 70, add 125 gallons (1041 lb) water and dissolve the following chemicals
_____	_____	_____	10.43 kg (23.0 lb) BaCl ₂ _____
_____	_____	_____	55.3 kg (121.8 lb) NaCl _____
_____	_____	_____	8) Transfer tank 70 contents to tank 69.

PAGE 3 OF 6

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

FINAL REVISION ALL QUANTITIES ADDED FROM SMALL BATCH

Simulated DSSF Waste Makeup Procedure (10-13-88 revision)

2500 gallon batch

DATE **TIME** **SIG** **INGREDIENTS/DIRECTIONS IDENTIFICATION (MFR., GRADE, LOT#)**

____	____	____	9) slowly add the following chemicals to tank 69 (precipitates will form)
____	____	____	136.6 kg (301.2 lb) KOH _____
____	____	____	1693.4 kg (3733.3 lb) NaOH _____
____	____	____	11) Slowly add 4647.1 kg (10,245 lb) Al_2NO_3 to tank 69 while agitating the tank. When the temperature reaches 95°C add the small 200 gallon batch. Then continue.
____	____	____	10) Add the following chemicals to tank 69.
____	____	____	361.3 kg (796.5 lb) $NaNO_2$ _____
____	____	____	162.1 kg (357.5 lb) Na_2CO_3 _____

PAGE 4 OF 6

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

FINAL REVISION ALL QUANTITIES ADDED FROM SMALL BATCH

Simulated DSSF Waste Makeup Procedure (10-13-88 revision)

2500 gallon batch

DATE TIME SIG INGREDIENTS/DIRECTIONS IDENTIFICATION (MFR., GRADE, LOT#)

____ 37.2 kg (81.9 lb) EDTA
(tetra sodium salt trihydrate) _____

____ 21.2 kg (46.6 lb) citric acid
(tri sodium salt dihydrate) _____

____ 40.5 kg (89.3 lb) colloidal silica
(HS-30) _____

PAGE 5 OF 6

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

FINAL REVISION ALL QUANTITIES ADDED FROM SMALL BATCH

Simulated DSSF Waste Makeup Procedure (10-13-88 revision)

2500 gallon batch

DATE **TIME** **SIG** **INGREDIENTS/DIRECTIONS** **IDENTIFICATION (MFR., GRADE, LOT#)**

____ 11) Adjust the volume to the 2700 gallon level. (To do this take a sample and measure the density. From the total weight of solution determine the volume of solution. Measure the depth in the tank and calculate a volume/ inch height number for the tank. Determine how many inches high the waste should be for 2700 gallons.) Turn on the heater and set the setpoint at 113°F. Allow the contents to reach the setpoint.

____ 13) After the solution has reached 113°F readjust the volume to 2738 gallons by adding additional water. Leave the agitator on low to maintain solids in suspension.

SCALE USED **CALIBRATION #** **EXPIRATION DATE**

_____	_____	_____
_____	_____	_____
_____	_____	_____

PAGE 6 OF 6

Makeup Performed By: _____

Data Sheet Checked By: _____

FULL SIGNATURE AND DATE

APPENDIX B

PREDICTION OF ADIABATIC TEMPERATURE RISE

APPENDIX B

PREDICTION OF ADIABATIC TEMPERATURE RISE

To estimate the adiabatic temperature rise for the grout a heat balance was performed on a 12 in. X 12 in. cylinder centered around the actual location of the maximum temperature within the vessel. The thermal conductivity of the grout was determined from in-situ thermal conductivity probes. Temperature data for the monolith was recorded on an hourly basis for the first 338 hours and every 8 hours after that for a total of 1403 hours. For each set of temperature data, the amount of heat loss occurring during the sample interval is calculated and totaled. The total heat loss is converted to a temperature change using the heat capacity of the grout. This temperature is then added to the observed temperature to obtain the predicted adiabatic temperature for that sample period. The total heat loss continually increases as more heat is lost, but if there is no generation of heat the increase in the term added to the temperature to obtain the adiabatic rise should equal the actual change in temperature due to heat loss. This results in a steady unchanging adiabatic temperature. Because there may be small errors in the estimation of the gradients and in the values of the thermal properties used the problem must be "balanced" at long times when it can be assumed that there is no further heat generation. This is done by adjusting the heat capacity of the grout to obtain an approximately steady adiabatic temperature at long times. The calculation was balanced at 1400 hours for a specific heat of 0.5. The actual specific heat is believed to be closer to 0.6. This error is most likely due to inaccuracies in the method used to determine the radial gradient. The fact that the adiabatic temperature stays flat as the measured temperature is falling between 700 and 1400 hours (29 to 58 days) indicates that the balanced calculation is accurately predicting the case where there is no heat generation (see Figure 3.14). The presence of unusual fluctuations and especially decreases in the adiabatic temperature would have indicated a problem.

The 12 in. X 12 in. cylinder over which the heat balance was centered was located at a height of 54 in. from the floor. This height was chosen

since this is the location where the peak temperatures occurred. This is slightly above the center of the vessel. The center of the cylinder for the heat balance is equidistant from thermocouples 5 and 13 (see Figure B.1). The top of the cylinder is equidistant between thermocouples 13 and 6 and the bottom of the cylinder is equidistant between thermocouples 5 and 12. For purposes of this calculation the temperature profile was assumed to be symmetric in the radial direction. Plots of temperature profiles tended to support the assumption of a radially symmetric temperature distribution (see Appendix C). Therefore, the temperature profiles at 6 in. (2.54 cm) radius in any direction from the vessel centerline are the same. If the problem was not symmetric about the center the two thermocouple trees would not plot into a single line.

FIGURE B.1 Cylinder of Grout Inside the Monolith Over Which the Heat Balance was Taken

The equations used in calculating the adiabatic temperature rise are provided below. A summary of nomenclature is provided at the end of this appendix for convenience. The volume of the cylinder in Figure B.1 was calculated as:

$$V = \frac{\pi D^2 H}{4} \quad (B.1)$$

Where D and H are the diameter (30.48 cm) and height (30.48 cm) of the cylinder. This results in a volume of 22240 cm³. The heat capacity of the cylinder was calculated as 17792 Cal/°C based on the volume of the cylinder (cm³) the density of the grout (1.6 g/cm³) and an assumed specific heat (0.5 cal/g °C), which was determined by adjusting the problem to indicate no heat generation at very long times.

$$C_{p, \text{cylinder}} = V \rho C_p \quad (B.2)$$

At each time for which temperature data was logged, the predicted temperature change from heat loss through each surface of the cylinder was calculated from:

$$\Delta T_{\text{cylinder}} = \frac{k \left[\left(A \frac{\Delta T}{\Delta y} \right)_{\text{top}} + \left(A \frac{\Delta T}{\Delta y} \right)_{\text{bot.}} + \left(A \frac{\Delta T}{\Delta x} \right)_{\text{side}} \right]}{C_{p, \text{cylinder}}} \Delta t \quad (B.3)$$

Here, K is the thermal conductivity of the grout determined from thermal conductivity probes inserted into the grout to be 6.539 cal/(h cm°C). The "A" represents the area of the cylinder surface for which the top or bottom equals 729.7 cm² and the side area equals 2918.7 cm². The thermal gradient is estimated from temperature data. The gradient at the top of the vessel is determined by the difference between temperatures measured by thermocouples

#6 and #13 divided by the 15.24 cm vertical distance between them. Similarly, the gradient at the bottom of the cylinder is calculated using thermocouples #5 and #12.

$$\left(\frac{\Delta T}{\Delta y} \right)_{\text{top}} = \frac{T_{13} - T_{06}}{15.24} \quad (\text{B.4})$$

$$\left(\frac{\Delta T}{\Delta y} \right)_{\text{bot.}} = \frac{T_{05} - T_{12}}{15.24} \quad (\text{B.5})$$

The vertical gradient determined at the edge of the cylinder is assumed to be uniform across the area of the cylinder top or bottom.

To determine the gradient in the radial direction at the surface of the cylinder, a second-order finite difference approximation was used to represent the radial gradient. A linear coordinate system was defined with $x = 0$ at one side of the cylinder, $x = 30.48$ cm on the opposite side, and $x = 91.44$ cm at the next available set of radial thermocouples. The temperature (T_0) at the center of the cylinder side surface is taken as the average of temperatures indicated by thermocouples #5 and #13. The temperature (T_1) at the same elevation but at a position of 91.44 cm (note: this is the same as a radial position of 30 in. (76.2 cm)) is taken as the average of thermocouples #23 and #31. The second-order finite difference approximation provides the following equations for the temperature ($T, ^\circ\text{C}$) and temperature gradient ($dT/dx, ^\circ\text{C}/\text{cm}$) as a function of position (x, cm).

$$T = T_0 + \frac{T_1 - T_0}{2787} \left(\frac{x^2}{2} - 15.24x \right) \quad (\text{B.6})$$

$$\frac{dT}{dx} = \frac{T_1 - T_0}{2787} (x - 15.24) \quad (B.7)$$

Evaluating the temperature gradient at the cylinder surface $x = 30.48$ cm (a radial position 15.24 cm from the centerline) by substituting the temperatures from thermocouples to estimate T_0 and T_1 provides a temperature gradient in °C/cm of:

$$\left(\frac{\Delta T}{\Delta x} \right)_{\text{side}} = \frac{\frac{T_{23} + T_{31}}{2} - \frac{T_{35} + T_{13}}{2}}{182.74} \quad (B.8)$$

The use of a second-order finite difference approximation and two equal temperatures at equal radial positions forces the temperature profile to be radially symmetric with a zero gradient at the vessel centerline. The temperature gradient determined for the center of the cylinder side is assumed to be uniform along the vertical dimension of the cylinder.

The expected temperature change due to heat loss (or gain) during each period is calculated by inserting the gradients determined in equations 4, 5, and 8 into equation 3. The heat loss (or gain) over time is integrated and at each time can be added to the actual observed temperature to determine the adiabatic temperature rise at that point. After heat generation due to curing has stopped, the adiabatic temperature becomes constant because the estimated heat loss matches the observed temperature change in the cylinder.

The error has not been quantitatively estimated based on uncertainties in input parameters. However, it is expected that the calculation of adiabatic temperature rise is accurate to within 5°C or roughly 20% of the estimated adiabatic temperature rise in excess of the maximum observed temperature.

The problem encountered in the ROM software (section 3.1) of the data-logger did not affect the calculated gradients since all thermocouples used in calculating the gradient were attached to the same multiplexer card. If all the temperatures on a card read 1° off the true value, the differences are still accurate. The temperature data was recorded directly on disk and transferred to a Lotus 123 spreadsheet program for calculations.

NOMENCLATURE

- A = area, cm^2
- C_p = heat capacity of grout, $\frac{\text{cal}}{\text{g } ^\circ\text{C}}$, adjusted to balance calculation at long times
- $C_{p,\text{cylinder}}$ = heat capacity of cylinder, $\frac{\text{cal}}{^\circ\text{C}}$
- D = diameter of cylinder, cm
- H = height of cylinder, cm
- k = thermal conductivity of grout $\frac{\text{cal}}{\text{h cm } ^\circ\text{C}}$
- T = temperature, $^\circ\text{C}$
- T_0 = temperature at surface of cylinder side, midway between ends, $^\circ\text{C}$
- T_1 = temperature at elevation of cylinder but at $x = 91.44$ cm or radial position of 76.2 cm
- T_{aa} = measured thermocouple temperatures, $T_{05,06,12,13,23,31,35}$
- $\Delta T_{\text{cylinder}}$ = temperature change, $^\circ\text{C}$, due to heat loss (gain) from cylinder during time Δt
- Δt = time between logged temperature data points, hours
- V = volume of cylinder, cm^3 , for which heat balance is calculated
- x = horizontal distance, cm, cylinder sides located at $x = 0$ and $x = 30.48$ cm
- y = vertical distance, cm
- ρ = density of grout, $\frac{\text{g}}{\text{cm}^3}$

APPENDIX C

TEMPERATURE DISTRIBUTION IN CURING GROUT

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TEMPERATURE DISTRIBUTION IN CURING GROUT

Figure 3.14 provided the temperature of the grout over time near the center of the grout mass where the highest temperatures were observed. This appendix provides additional information on the temperature distribution within the curing grout at three selected times.

Figure C.1 provides the vertical temperature profile within the grout at each of three radial positions measured 100 hours after production. This plot provides a check for the assumption of radial symmetry in the temperature profile within the curing grout. The group of data plotted for each distance from the vessel centerline consists of temperatures measured at multiple radial positions. The thermocouple locations were provided in the document in Figure 2.10. For the temperatures measured 30 inches from the vessel centerline, the data is combined from four thermocouples spaced every 90°. For radial distances of 6 inches and 45 inches, each curve represents data from two thermocouple trees spaced at 180° from each other. The data appears smooth for any given distance from the vessel centerline, indicating that the assumption of radial symmetry in the temperature distribution is valid.

Figure C.2 shows temperature contours within the grout mass at 100 hours. The vertical axis is located at the vessel centerline and provides the distance from the floor of the grout vessel. The horizontal axis corresponds to the horizontal distance from the vessel centerline. The data plotted in Figure C.2 are the same data used to generate the contours in Figure C.1. The temperature data for this plot was assembled by assuming radial symmetry throughout the vessel and combining the thermocouples into a single plane based on their radial position and height in the vessel. Temperature data is only available at radial positions of 6 and 30 inches (6-inch height intervals) and at a radial position of 45 inches (12-inch height intervals). Therefore, there is significant interpolation in the contour plot and the contours should not be over-interpreted. However, the gentle radial temperature gradient (indicated by very few contour lines)

FIGURE C.1. Vertical Temperature Profiles at 100 Hours. The smoothness of the data in the curves indicates that the assumption of radial symmetry in the temperature profile is valid.

FIGURE C.2. Temperature (°C) Contours In Curing Grout 100 Hours After Production

does indicate that the most severe heat loss was from the bottom of the vessel and to a lesser extent, the top of the vessel. Significantly less heat was lost from the sides of the vessel.

Figures C.3 and C.4 provide similar temperature profiles and contours as Figures C.1 and C.2 except that the data is provided at an elapsed time of 260 hours after production, which approximately corresponds to the time at which maximum temperatures were observed. Figures C.5 and C.6 provide similar temperature profiles and contours as Figure C.1 and C.2 at 499 hours after production. At this point, the grout has begun to cool and the heat generation due to grout curing is nearly complete.

FIGURE C.3. Vertical Temperature Profiles at 260 Hours. The smoothness of the data in the curves indicates that the assumption of radial symmetry in the temperature profile is valid.

FIGURE C.4. Temperature (°C) Contours In Curing Grout 260 Hours After Production

FIGURE C.5. Vertical Temperature Profiles at 499 Hours. The smoothness of the data in the curves indicates that the assumption of radial symmetry in the temperature profile is valid.

FIGURE C.6. Temperature (°C) Contours In Curing Grout at 499 Hours

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