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106-AN GROUT PILOT-SCALE TEST
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SUMMARY

The Grout Treatment Facility (GTF) at Hanford, Washington will process the low-level fraction of selected double-shell tank (DST) wastes into a cementitious waste form. This facility, which is operated by Westinghouse Hanford Company (WHC), mixes liquid waste with cementitious materials to produce a waste form that immobilizes hazardous constituents through chemical reactions and/or microencapsulation. Over 1,000,000 gal of Phosphate/Sulfate Waste were solidified in the first production campaign with this facility.

The next tank scheduled for treatment is 106-AN. After conducting laboratory studies to select the grout formulation, part of the normal formulation verification process is to conduct tests using the 1/4-scale pilot facilities at the Pacific Northwest Laboratory (PNL).[a] The major objectives of these pilot-scale tests were to determine if the proposed grout formulation could be processed in the pilot-scale equipment and to collect thermal information to help determine the best way to manage the grout hydration heat.

Two molds were constructed to accomplish these goals. The first mold was designed to produce a temperature gradient in the grout, as it cured, in order to determine the effects of curing temperature on the final properties of the grout. The second mold was designed to simulate pouring grout in 2-ft lifts. The main grout production run filled the first mold and the first of four lifts in the second mold. Over 2000 gal of grout were processed during 3.5 hours of production time during the main pour. Three additional lift pours, each consisting of 350 gal, were conducted at 1-week intervals to complete the second mold. The grout in the main run and the second and third lift pours was produced by mixing simulated 106-AN tank waste with dry-blend consisting of 14 wt% attapulgite clay, 20 wt% cement, and 66 wt% class F fly ash at a mix ratio of 8.7 lb per gal (lb/gal). A dry-blend composition of 11 wt% attapulgite, 20.7 wt% cement, and 68.3 wt% class F fly ash with a mix ratio of 8.4 lb/gal was used for the fourth lift.

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

The major conclusions from the grout production portion of the pilot-scale tests are listed below:

- The grout produced with a dry-blend formulation consisting of 14 wt% attapulgite clay, 20 wt% cement, and 66 wt% class F fly ash showed significant shear thickening and had calculated critical flow rates (CFR) at the pipe discharge that were above the criterion value of 60 gpm. Slight modification of the dry-blend formulation to 11 wt% attapulgite, 20.7 wt% cement, and 68.3 wt% class F fly ash reduced the critical flow rate to below 40 gpm. Other than the critical flow rate concerns, both formulations tested were readily processed by the pilot-scale equipment.

- The Dry Materials Facility (DMF) handled the dry ingredients of the proposed production formulation and mixed dry-blend product within the desired tolerances.

- The restart pressure tests showed that process interruptions as long as 20 minutes did not pose a problem in the pilot-scale equipment. These tests indicated that interruptions of 30 minutes or greater should not be allowed without flushing the system.

- No significant wear was seen on the stellite feed screws and stellite-tipped paddles installed in the grout mixer.

- A 7.5 pipe-volume flush of the pilot-scale grout pipe at 10 gpm was sufficient to prevent buildup.

- Grout buildup in the equipment was similar to that seen in other pilot-scale runs. Buildup in the area of the dry-blend mixer inlet was a concern and may have interfered with grout production if it had not been cleaned between runs. Buildup in other areas did not interfere with grout production but might present decontamination problems.

- The dimensional changes of the grout over the first 7 weeks of curing were small (0.06% shrinkage).

- The thermal conductivity of the cured grout was 0.81 watts per meter°K (W/m°K).

- Neither the original 14 wt% attapulgite clay formulation nor the modified 11 wt% attapulgite clay formulation had free liquids when poured at 40°C.

Since the completion of the first full-scale production campaign, concerns over the effects of high grout temperatures on the long-term grout properties have arisen. Increased airflows to increase evaporative cooling in the grout vault combined with pouring in lifts has been suggested as a means to remove hydration heat and reduce grout curing temperatures. These pilot-scale tests were used to study the effects of increased airflows and pouring in lifts on the final grout temperatures. The major conclusions are listed below:

- The calculated adiabatic temperature rise of the grout poured in the gradient mold was 57°C.
- Comparing calculated heat conduction rates through grout to the experimentally-determined airflow heat removal rates from water/salt solutions showed that conduction of heat through the grout controls the heat removal rate when using the airflow rates planned for the production vault. As a result, increased airflows (e.g. larger blowers) would not significantly increase the heat removal rates.
- An airflow of 13 scfm in the lift mold (which simulated a 3600 scfm airflow in the production vault) kept the maximum short-term grout temperatures below 70°C for all four of the 2-ft lifts poured and maintained average grout surface temperatures below 30°C.
- The net temperature reduction obtained by cooling the surface of a 2-ft lift for 1 week was approximately 30°C.
- Heat removal rates throughout the week between pours were not significantly different for lifts with and without free-standing liquid.
- The lift mold thermal profiles after 1 week of cooling showed a general tendency, as lifts were added, for the peak temperatures to be higher and located farther below the surface with each subsequent lift.

ACKNOWLEDGMENTS

The author would like to thank William A. Sliger for his valuable contributions in preparing the equipment and conducting the tests. The author also acknowledges the Westinghouse Dry Materials Facility for the timely delivery of two separate batches of dry-blend material. The efforts of Paul F. Martin and Ryan O. Lokken in planning and conducting the various grout properties' measurements are also appreciated. The author would also like to thank Greg A. Whyatt for his valuable input while planning and conducting the tests.

The work described in this report was performed as part of the Hanford Grout Technology Program (HGTP), managed by Joseph H. Westsik, Jr. at the Pacific Northwest Laboratory and was funded by the Grout Disposal Program at Westinghouse Hanford.

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1.0 INTRODUCTION

The Grout Treatment Facility (GTF) at Hanford, Washington will process the low-level fraction of selected double-shell tank (DST) wastes into a cementitious waste form. This facility, which is operated by Westinghouse Hanford Company (WHC), includes the Dry Materials Facility (DMF), the Grout Processing Facility (GPF), and the grout disposal vaults. The DMF receives, stores, and blends the individual dry materials for use in the grouting operation. Semitrailer trucks transport the dry materials to the GPF where these materials are mixed with the low-level waste in a continuous process at rates up to 70 gal of grout per minute. The grout slurry is then pumped into near-surface concrete vaults where it hardens and immobilizes the hazardous and radioactive constituents through chemical reactions and/or microencapsulation.

Pacific Northwest Laboratory (PNL) has a pilot-scale grout processing facility capable of grout outputs of up to 25% of the GPF. In 1986, the first major pilot-scale test demonstrated the effectiveness of the proposed grouting equipment and showed that grouting was an effective technique for immobilizing a simulated phosphate/sulfate decontamination waste (PSW). The grout dry-blend formulation used in this first test was a mixture of 41 wt% type I/II portland cement, 40 wt% class F fly ash, 11 wt% attapulgite-150 drilling clay, and 8 wt% indian red pottery clay. Based on this pilot-scale test, a successful production campaign that solidified over 1,000,000 gal of PSW was conducted during 1988 and 1989 (Cline et al. 1989).

In 1988, PNL conducted a second major pilot-scale test to demonstrate the processing of a simulated double-shell slurry feed (DSSF) waste and to provide information for scale-up. The dry-blend for this run was a mixture of 47 wt% class F fly ash, 47 wt% blast furnace slag, and 6 wt% type I/II portland cement. Information obtained during this second run led to concerns about the amount of heat generated during curing and the ultimate temperature of the grout. These tests indicated that the temperature of this second formulation climbed well above the allowed 90°C maximum and degradation of the grout properties was possible (Lokken 1992).

Oak Ridge National Laboratory (ORNL), WHC, and PNL collaborated to investigate approaches to reduce the grout temperatures. The first approach was to formulate a grout mixture that generated less heat and resulted in a lower ultimate temperature while meeting all other formulation criteria. These studies focused on the waste in tank 106-AN because this waste would be the feed source for the next campaign. However, formulation studies conducted by ORNL and PNL determined it was not possible through formulation adjustments alone to have a grout that remains below acceptable temperatures while maintaining all other minimum product criteria. Therefore, increased convective/evaporative cooling is necessary to prevent excessive temperatures in the grout vault. Thus, a formulation that maximized the grout leachability index (ANSI 1986) was chosen for the pilot-scale tests. This formulation had a dry-blend that consisted of 66 wt% class F fly ash, 20 wt% type II portland cement, and 14 wt% attapulgite clay. A dry-blend that consisted of 11 wt% attapulgite clay, 20.7 wt% type II portland cement, and 68.3 wt% class F fly ash was also examined.

The equipment currently being considered to supply the additional air flow for cooling the grout will be capable of supplying 3600 cubic ft per minute (cfm).

1.1 OBJECTIVES

The main objectives of the pilot-scale test are listed below.

1. Confirm that a dry-blend consisting of 66 wt% class F fly-ash, 20 wt% type II portland cement, and 14 wt% attapulgite clay mixed with a simulated 106-AN waste at 8.7 lb/gal can be adequately processed in pilot-scale equipment.

2. Determine the amount of heat that might be removed from a large grout casting through convective/evaporative cooling of its surface.

3. Determine the temperature profiles obtained when an 8-ft section of grout is poured in four 2-ft lifts to support thermal model validation.

4. Determine the rheological properties of the grout slurry at different points in the grout process. Determine the minimum grout production rate that will maintain turbulent flow in the grout transfer pipes.

5. Determine the expected restart pressures for an upset condition where pump operation is interrupted for up to 30 minutes.

6. Confirm that the dry ingredients of the new formulation can be adequately mixed in the DMF.

7. Monitor the dimensional changes of a large-scale grout casting during curing to determine if the grout expands or contracts.

8. Determine the thermal conductivity of the new grout formulation.

9. Determine if the new grout formulation will significantly impact the operating life of the production mixer and pump.

10. Determine the compressive strength, homogeneity, leachability, corrosivity, microstructure, density, and fish and rat toxicity for core samples taken from large-scale pours and compare to laboratory-prepared specimens.

11. Determine the effects of different curing conditions (e.g. time at temperature) on the final grout properties.

12. Determine if grout properties near a lift interface are significantly different than grout properties away from an interface.

13. Determine the effectiveness of 6N citric acid as a decontamination solution.

The results from the pilot-scale grout runs and the analysis of the thermal information will be presented in this report (objectives 1 through 8). The characterization of the core samples, decontamination solution test results, and wear results (objectives 9 through 13) will be discussed in separate reports.

1.2 SCOPE

The composition of the simulated 106-AN tank waste was based on the latest analyses available from WHC (Hendrickson 1992). The required 3500 gal of simulated waste was mixed in an insulated 4000-gal tank and heated to approximately 45°C before processing. The DMF mixed the required dry-blend and production dry-blend trucks transported the dry-blend to PNL.

The pilot-scale tests were conducted using the grouting facilities located at PNL. The grout production rate was approximately 10 gal per minute (gpm). A 5-in. Teledyne Readco, Twin-Shaft Continuous Mixer was used for mixing. This mixer is the same brand and type of mixer used in the GPF. The mixed grout fell into an agitated, conical surge tank that fed a Roper two-stage progressing cavity pump. From the pump, the grout passed through 100 to 135 ft of 3/4-in. schedule-40, carbon steel pipe into one of two molds.

The first mold was 8-ft dia x 7.5-ft high with plate coils on its outer surface. Water at a target temperature of 38°C was circulated through the plate coils to create a thermal gradient in the grout. Therefore, this mold will be referred to as the gradient mold. Thermocouples were used to measure the actual curing temperatures at various locations in the mold. As a result of the gradient, grout in the center of the mold cured at temperatures significantly higher than grout at the walls of the mold. Thermal information from the center of the mold was also used to calculate a predicted adiabatic temperature rise. After collecting the thermal data, core samples were obtained from different locations in the mold to determine the effects of curing conditions on other grout properties.

The second mold was 3-ft wide x 7.5-ft long x 10-ft deep and was used to study the effects of pouring in lifts and convective/evaporative cooling. This mold will be referred to as the lift mold. The length and width of the lift mold were scaled to match the length/width ratio on the production vault. The lift mold was insulated to reduce heat losses through the walls and was equipped to introduce controlled airflows through the mold. The mold was instrumented to measure the inlet and outlet air temperature and humidity to allow heat-loss calculations resulting from the airflow. Prior to filling with grout, the lift mold was used to study the amount of heat that is removed from a layer of liquid by different airflows. Heat removal rates as a function of liquid temperature for water and a sodium nitrate salt solution were determined. This information was used to determine the appropriate airflows to use in the pilot-scale run.

After conducting the airflow tests, the lift mold was instrumented with thermocouples and filled in four 2-ft lifts. These lifts were poured with 1 week between each lift. The temperature and heat removal information obtained from the lift mold was used to determine the effects of pouring in lifts. After the thermal information was collected, core samples were taken to determine if grout properties at a lift interface were significantly different than properties away from an interface.

2.0 FACILITY DESCRIPTION

The pilot-scale grout facility is located in building 324. A schematic of the system is shown in Figure 2.1.

2.1 SIMULATED WASTE FEED SYSTEM

The simulated 106-AN was mixed and stored in an insulated 4000-gal stainless steel tank. The tank is equipped with a 10-HP, variable speed agitator and a circulation heater for temperature control. For this study, adjusting the speed of the agitator was sufficient to control the waste temperature. A centrifugal pump located below the tank was used to transfer the waste through a 1-in. carbon steel pipe to the grout mixer. A second centrifugal pump was added downstream from the first pump to assure adequate waste feed flow rates. The flow rate of the waste is measured using a magnetic flowmeter and controlled using a digital controller and air-actuated pinch valve. The feed temperature at the storage tank, the feed temperature at the mixer inlet, and the instantaneous feed rate were recorded using the data acquisition system.

2.2 DRY-BLEND FEED

The DMF blended the required dry-blend materials, and production dry-blend transfer trailers transported the material approximately 20 miles to the 324 Building. The dry-blend was transferred from access ports in the top of the trailers through a suction wand to a storage bin with a 27-ft^3 capacity using a Vac-U-Max vacuum pneumatic transfer system. The Vac-U-Max transfer system was able to transfer the dry-blend used in this test at a rate of 175 to 200 lb/min.

The storage bin is positioned above the active bin and automatically fills the active bin on a signal from the feeder controller. The active bin has a total volume of 36.6 ft^3 and an active volume of 30 ft^3. The Vac-U-Max, storage bin, and active bin are shown in Figure 2.2. The feeder is an Acrison gravimetric feeder with a weight rate accuracy of 0.5% of the set point. During transfer of materials from the storage bin to the active bin, the feeder operates on a volumetric basis (i.e. the feed screws turn at a constant speed). Instantaneous dry-blend feed rates are recorded by the data acquisition system. After leaving the feeder, the dry-blend passes through an 18-in. dia Sweco vibrating screen to prevent material greater than 0.20 in. from entering the mixer.

2.3 GROUT MIXER

The grout mixer is a Teledyne Readco 5-in. Twin Shaft Continuous Mixer. Due to wear concerns noted in the earlier pilot-scale runs, the mixer dry-blend feed screws were replaced with stellite feed screws, and the first four pairs of mixer paddles were replaced with stellite-tipped mixer paddles (see Figure 2.3). Stellite mixing components are used in the GPF mixer to minimize wear.

The mixing speed is adjustable from 50 to 270 revolutions per minute (rpm). The mixer speed for this pilot-scale run was 250 rpm and was based on matching the paddle tip speed in the production mixer. The adjustable discharge gate on the mixer was left completely open during all grout runs.

Predetermined quantities of dry-blend and simulated waste were metered to the mixer to obtain the desired grout production rate. The mixed grout discharges to a 17.5 gal, agitated conical surge tank. This surge tank (see Figure 2.4) has the same general geometry as the GPF surge tank with 1/4 the volume. The temperature of the grout in the surge tank was recorded by the data acquisition system.

2.4 GROUT PUMP

The pilot-scale grout pump was a Roper progressing cavity pump (shown in Figure 2.5) with an ethylene-propylene-diene-monomer (EPDM) stator. A water-lubricated packing gland serves as the pump seal. The pump inlet contains a small water jet programmed to flush the inlet section with a 4-second, 15.3-gpm spray (approximately 1 gal) every 10 minutes during production. The pump speed was manually adjusted to maintain a constant level in the surge tank. Pump amperage and frequency are recorded using the data acquisition system. The frequency is converted to rpm by using a calibration curve generated prior to operations.

A pressure gauge, pressure transducer, and flow meter are located at the discharge of the grout pump. The pressure and grout flow are recorded by the data acquisition system. A high-pressure, automatic shut-down feature stops the pump if the grout discharge pressure exceeds the preprogrammed maximum pressure of 125 psi.

2.5 PIPING

The grout pump discharges into a 3/4-in. schedule-40, carbon steel pipe that leads to a series of three-way valves. These valves can direct the grout slurry either to the gradient mold, the lift mold, or a dumpster. The total length of pipe when plumbed to the dumpster was 100.5 ft, when plumbed to the lift mold was 119.25 ft, and when plumbed to the gradient mold was 135.5 ft. The dumpster was used to receive flush water and grout not being used in the testing. A thermocouple measured the grout temperature just prior to the three-way valves.

2.6 GRADIENT MOLD

The gradient mold was constructed from an 8-ft dia x 7.5-ft high, carbon steel tank. This tank was placed on a structural support, and its circumference was surrounded by platecoils. The top and bottom were insulated to minimize the heat loss through those surfaces. Water was continuously circulated through the plate coils with a centrifugal pump. A heated 55-gal drum was part of the circulation loop and was used to help maintain a target temperature of 38°C in the plate coils. The exterior of the plate coils was insulated to reduce the heating requirements and improve temperature uniformity. The stand and plate coils were designed to allow disassembly and reuse for subsequent grout pilot-scale tests. At the completion of the tests, the grout will be disposed of in the carbon steel tank. Figure 2.6 shows the gradient mold in its test location west of the high bay area of 324 Building.

The gradient mold was equipped with a 1-ft diameter, 27.5-ft long, polyvinyl chloride (PVC) pipe through which the grout fell ~35 ft after discharging from the grout pipe. This simulates the maximum drop that the grout will see in the full-scale vault.

The gradient mold had 40 type-T thermocouples to measure the temperature profiles and determine the maximum temperature achieved in the grout. Type-T thermocouples have a lower standard limit of error than either type-K or type-J. The thermocouples were placed to measure the steeper gradients (predicted by modeling) near the mold wall. The radial orientation of each series of thermocouples is shown in Figure 2.7. The heights for each thermocouple measured from the bottom of the mold are shown in Table 2.1.

Each thermocouple was bent away from the series bundle at the desired height in order to minimize the effect of the bundle on the temperature profile being measured. Typical thermocouple trees in the gradient mold are shown in Figure 2.8. The locations listed in Table 2.1 are for the thermocouple tips. Temperature data were recorded by the data acquisition system.

Two TML KM-100HB embedment strain gauges were placed in the mold prior to pouring the grout. These gauges were accurate to ± 1% of rated output. Vishay 2310 signal conditioning amplifiers were used to condition these 350 ohm, full-bridge strain gauges. The strain gauges were used to measure expansion and contraction of the grout during the first 49 days of curing. One gauge was placed in an axial orientation 4.5 ft from the bottom and 2 ft from the center. The second was in a radial orientation 3.5 ft from the bottom of the mold and 2 ft from the center. Data from these strain gauges were recorded by the data acquisition system.

A thermal conductivity probe was also placed in the mold prior to pouring grout. However, interference between the power cable and the thermocouple signal did not allow in situ thermal conductivity measurements. As a result of this problem, samples of grout produced with the pilot-scale system were obtained for laboratory thermal conductivity measurements.

Three 4-in. carbon steel pipes were placed in the gradient mold for WHC nondestructive testing (NDT). The location of these pipes are shown in Figures 2.7 and 2.8.

TABLE 2.1. Thermocouple Placement in Gradient Mold

Series #	Angle	# of Thermocouples	Placement Heights from Bottom, (in.)
GA	0°	3	18, 48, 78
GB	180°	2	36, 60
GC	0°	2	36, 60
GD	180°	3	18, 48, 78
GE	45°	3	18, 48, 78
GF	225°	2	36, 60
GG	45°	2	36, 60
GH	225°	3	18, 48, 78
GI	90°	3	18, 48, 78
GJ	270°	2	36, 60
GK	90°	2	36, 60
GL	270°	3	18, 48, 78
GM	90°	3	18, 48, 78
GN	270°	2	36, 60
GO	90°	2	36, 60
GP	270°	3	18, 48, 78
		40 (TOTAL)	

2.7 LIFT MOLD

The lift mold was constructed with metal-reinforced plywood and had the approximate inner dimensions of 3-ft wide x 7.5-ft long x 10-ft tall. The box had a 20-mil high-density polyethylene liner to contain the grout slurry and 4 in. of foam insulation to reduce heat losses through the sides and top of the box. Two in. of duraboard insulation were placed on the bottom of the mold under the 20-mil liner. Duraboard has the required compressive strength, and this thickness has the same thermal resistance as ~4 ft of concrete. A second 20-mil liner was used outside the insulation as a backup for leaks in the primary liner. Figure 2.5 shows the lift mold in its test location west of building 324 high bay.

The air inlet and outlet were fabricated from 2-in. schedule-40 PVC pipe and placed in the lift mold cover at opposite ends of the box. The inlet and outlet had Vaisala HMP 1304 humidity and temperature probes. These probes had a capacitance type humidity sensor with a platinum resistive temperature device (RTD) temperature sensor. This probe was chosen due to its durability and its ability to recover after being placed in a saturated environment. The temperature accuracy of these probes was ± 0.2°C. The accuracy of the humidity measurements was ± 2% for 0% to 90% RH and ± 3% for 90% to 100% RH when calibrated against salt solutions. The outlet also had a Sierra Model 731 mass flow meter to record the airflow through the mold. The calibrated accuracy of the flow meter was better than 3%. Data from the humidity sensors and the flow meter were recorded by the data logger. A blower and butterfly valve at the outlet were used to generate the desired airflow through the mold. The inlet air for the mold was drawn from the high bay area of 324 Building.

The lift mold had 63 type-T thermocouples to measure the temperature profiles in the grout material. The thermocouples were held in position by a thermocouple tree constructed from 1-in. and 1/2-in. PVC pipe. This tree also helped support the inner liner. The installed PVC thermocouple tree is shown in Figure 2.9. The placements of the thermocouples are given in Table 2.2 and Figure 2.10. As in the gradient mold, each thermocouple was bent away from the series bundle at the desired height. Temperature data were recorded by the data acquisition system.

TABLE 2.2. Thermocouple Placement in Lift Mold

Series	# of Thermocouples	Heights from Bottom (in.)
LA	27	0, 4, 8, 12, 16, 20, 24, 28, 32, 36, 40, 44, 48, 52, 56, 60, 64, 68, 72, 76, 80, 84, 88, 92, 96, 108, one adjustable liquid TC
LB	4	12, 36, 60, 84
LC	4	12, 36, 60, 84
LD	4	12, 36, 60, 84
LE	4	12, 36, 60, 84
LF	4	12, 36, 60, 84
LG	4	12, 36, 60, 84
LH	4	12, 36, 60, 84
LI	4	12, 36, 60, 84
LJ	2	108, one adjustable liquid TC
LK	2	108, one adjustable liquid TC
	63 (TOTAL)	

2.8 DECONTAMINATION/RINSE SYSTEM

The decontamination/rinse system consists of a decon solution/rinse water holding tank, a pump, and a valve/tubing network. The valve/tubing network allowed the decontamination solution or rinse water to be injected at 150 psig into the grout mixer at the dry-blend inlet and the waste feed inlet, into the surge tank at its inlet, and into the grout pump at the pump inlet. A by-pass valve at the grout pump outlet could either recirculate the decontamination solution or rinse water back to the decon tank or allow the rinse water to be pumped through the grout line to the molds. The amount of rinse water that was sent through the grout pipe to the molds was controlled by introducing the desired amount of solution in the holding tank. A schematic of the system is shown in Figure 2.11.

2.9 INSTRUMENTATION/DATA COLLECTION

A schematic of the instrumentation is shown in Figure 2.12. The majority of the data were collected with a Wavetek Model 52 Data Multimeter. This data logger had two channels, each of which could read 64 differential measurements via expansion with multiplexer cards. One hundred and twenty-six of the 128 available slots were used to collect data from the pilot-scale run. The data logger was linked to a computer that controlled the timing of the data collection, stored the information on disk as an ASCII file, and produced a backup hardcopy. The ASCII file was imported into Lotus 123® and analyzed. The thermal profiles were generated with Surfer™, Golden Software, Inc. The balance of the data was collected by hand and recorded on data sheets.

Process Schematic

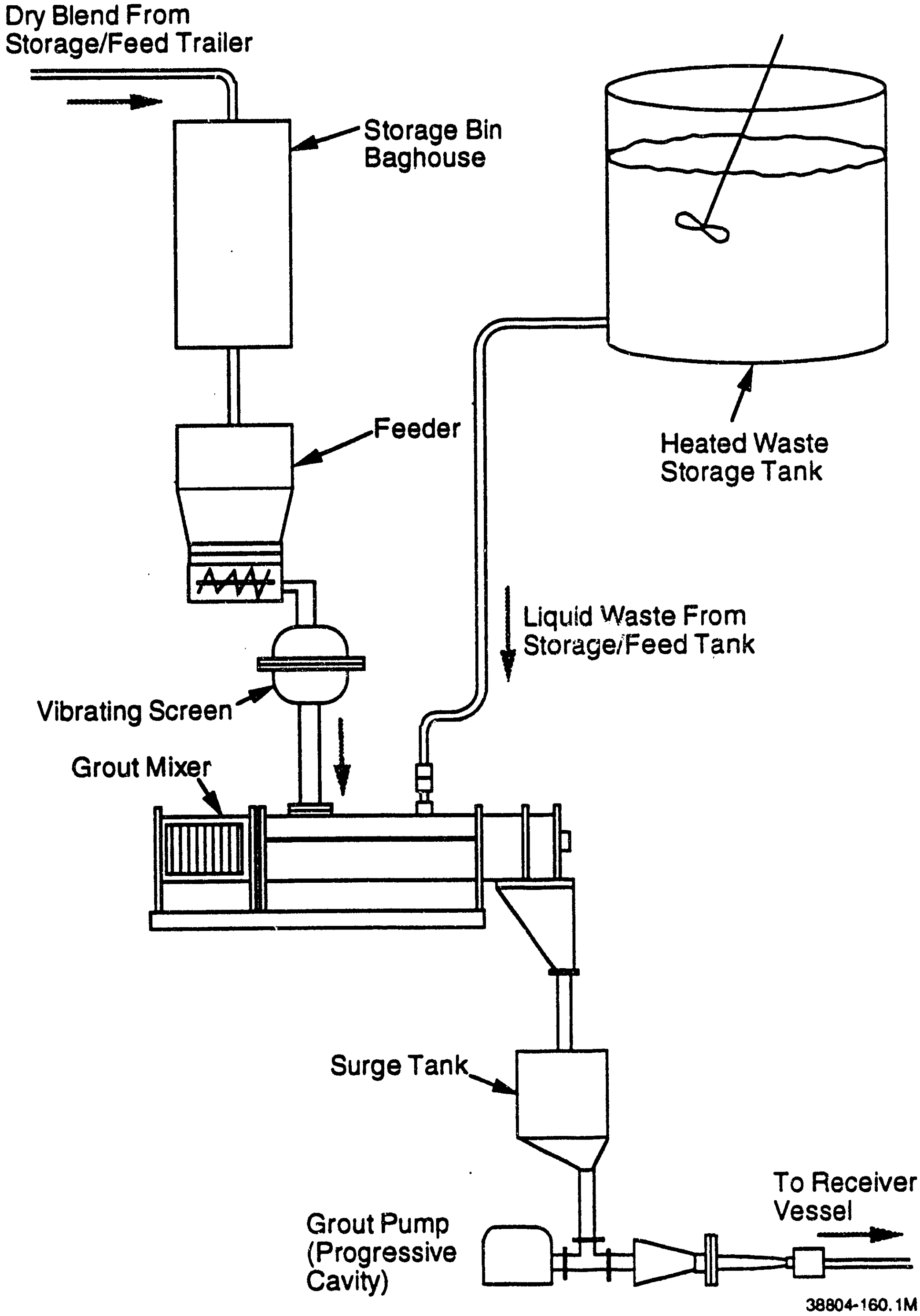


FIGURE 2.1. System Configuration

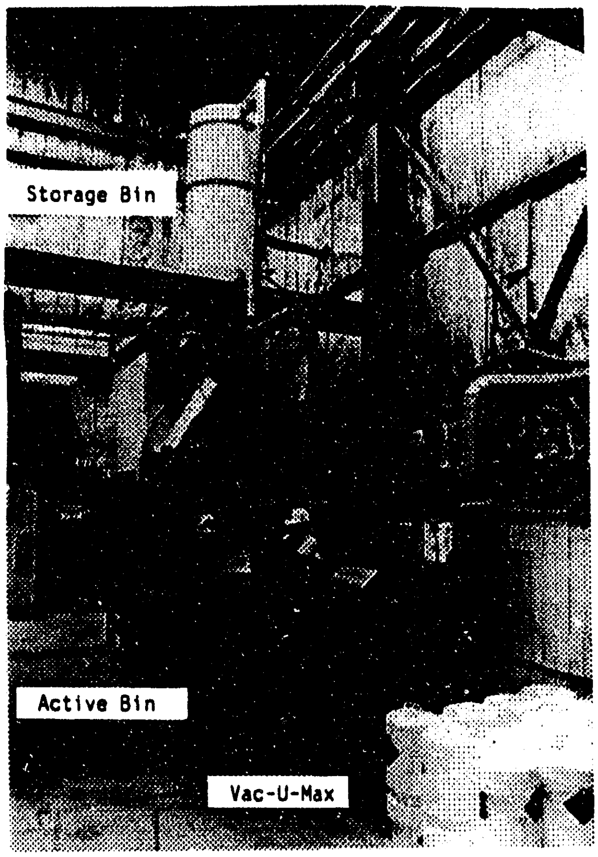


FIGURE 2.2. Dry-Blend Handling Equipment

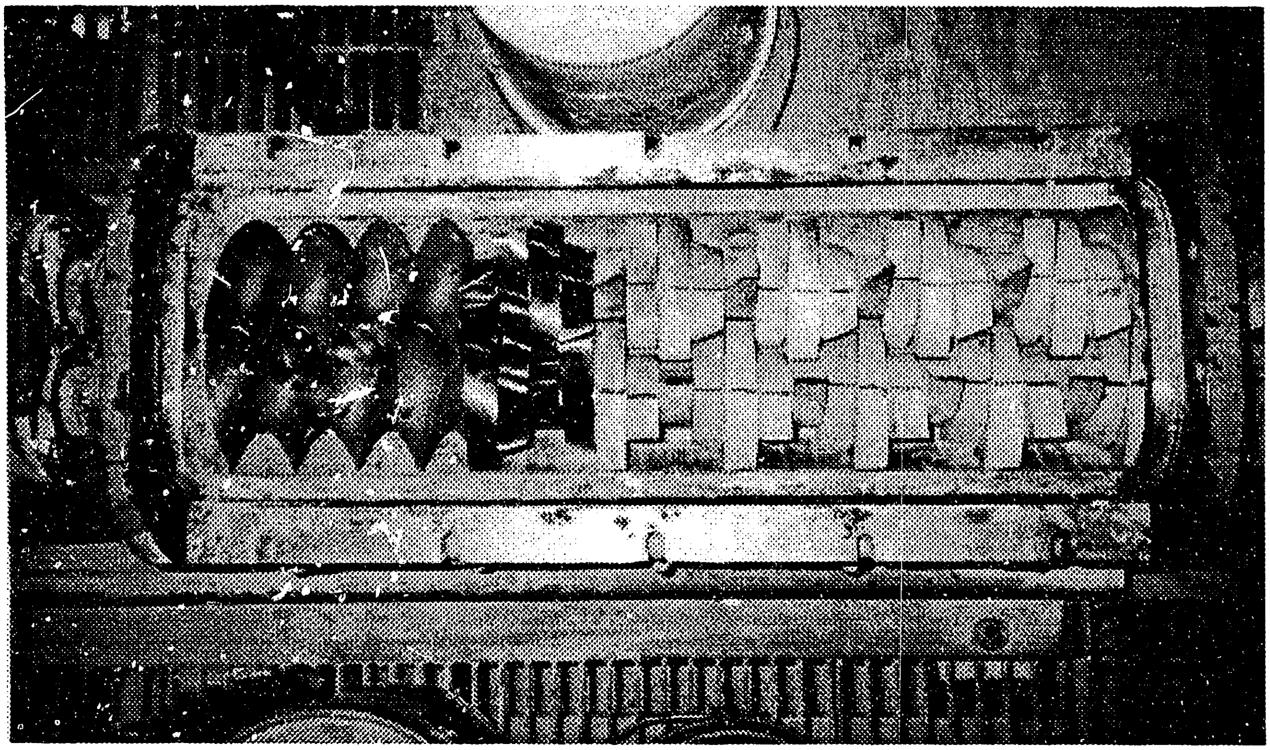


FIGURE 2.3. Internal Mixing Components of Pilot-Scale Grout Mixer (shown with cover removed)

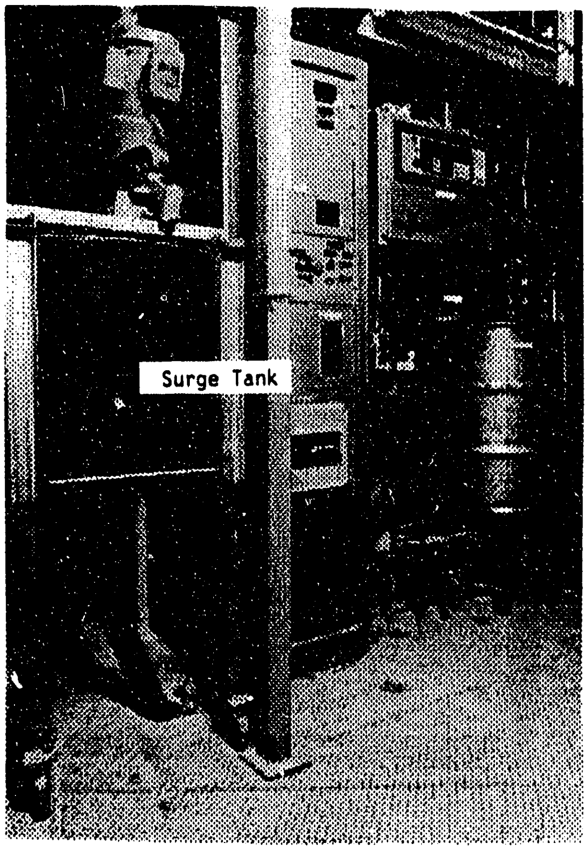


FIGURE 2.4. Pilot-System Control Station

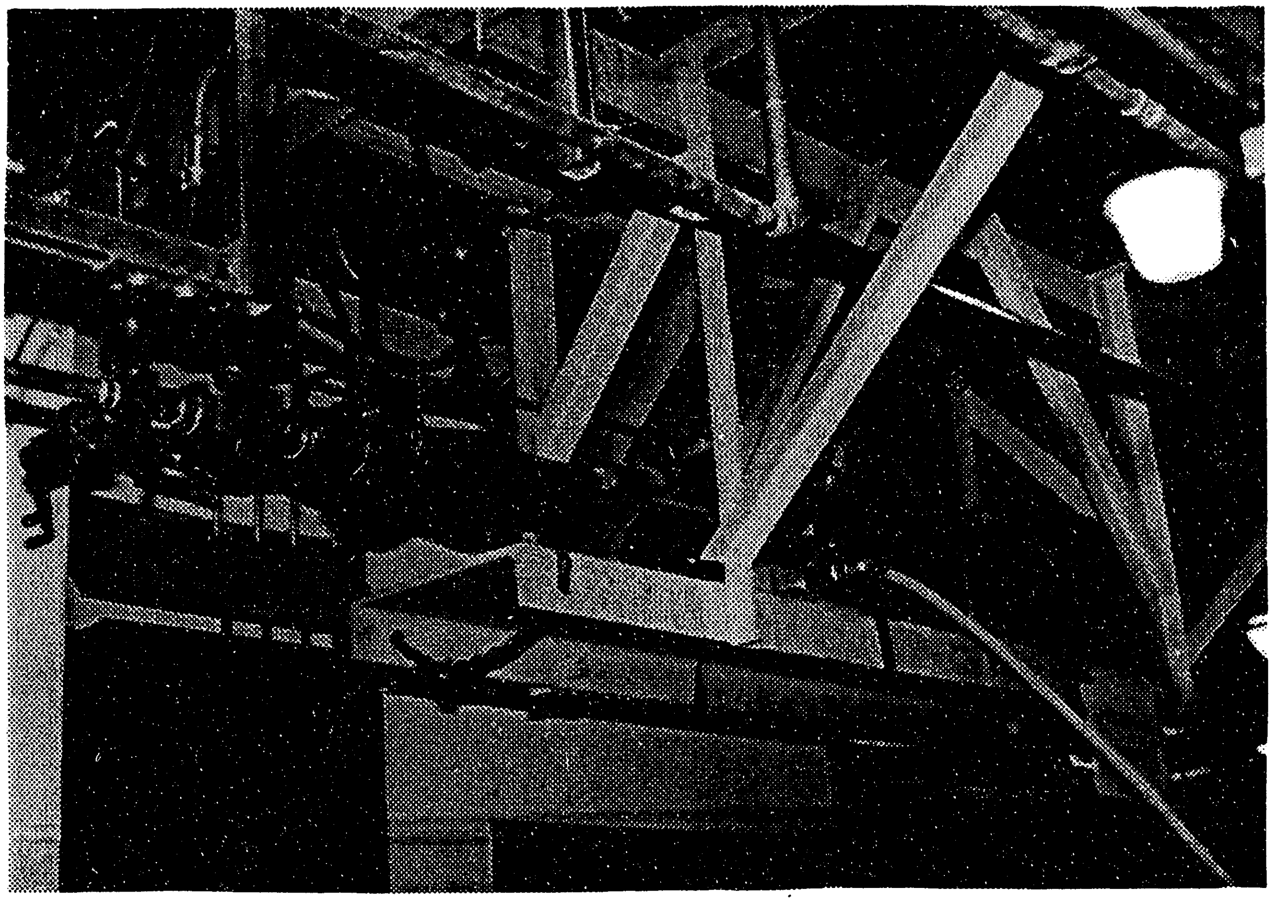


FIGURE 2.5. Pilot-Scale Grout Pump

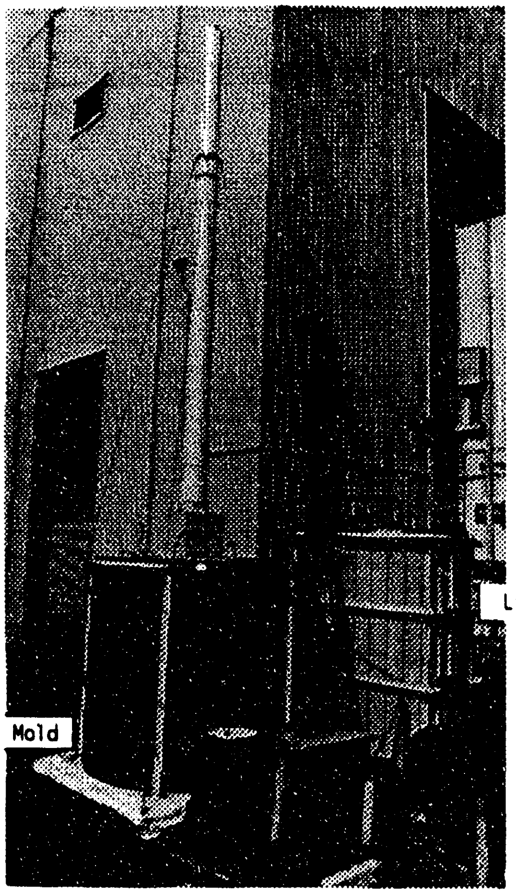


FIGURE 2.6. Gradient Mold and Lift Mold

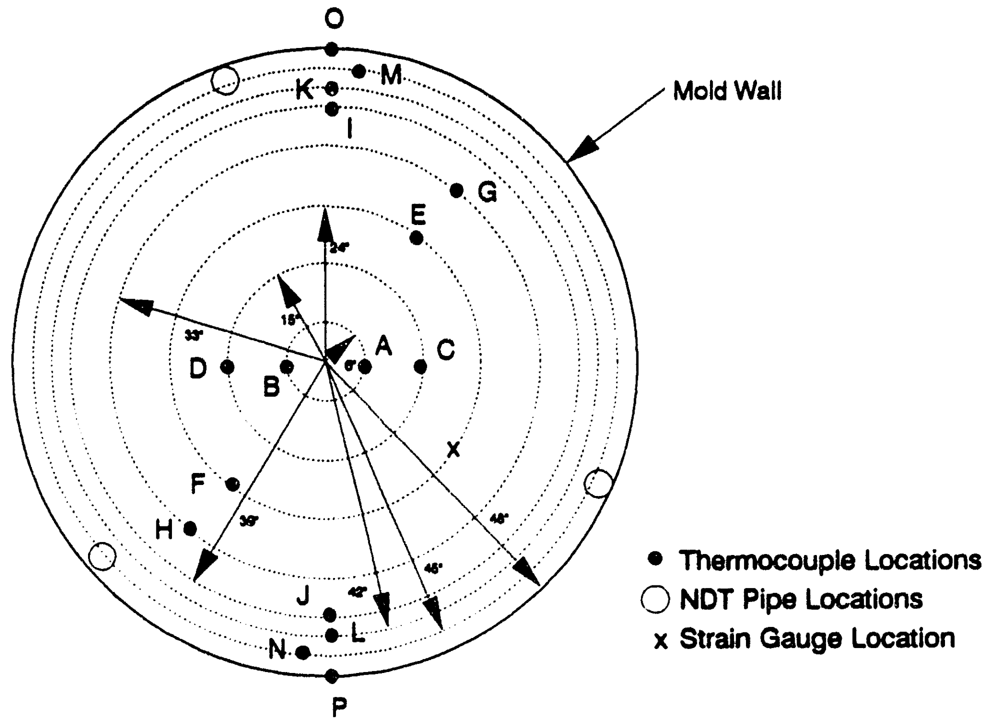


FIGURE 2.7. Thermocouple Placement in Gradient Mold

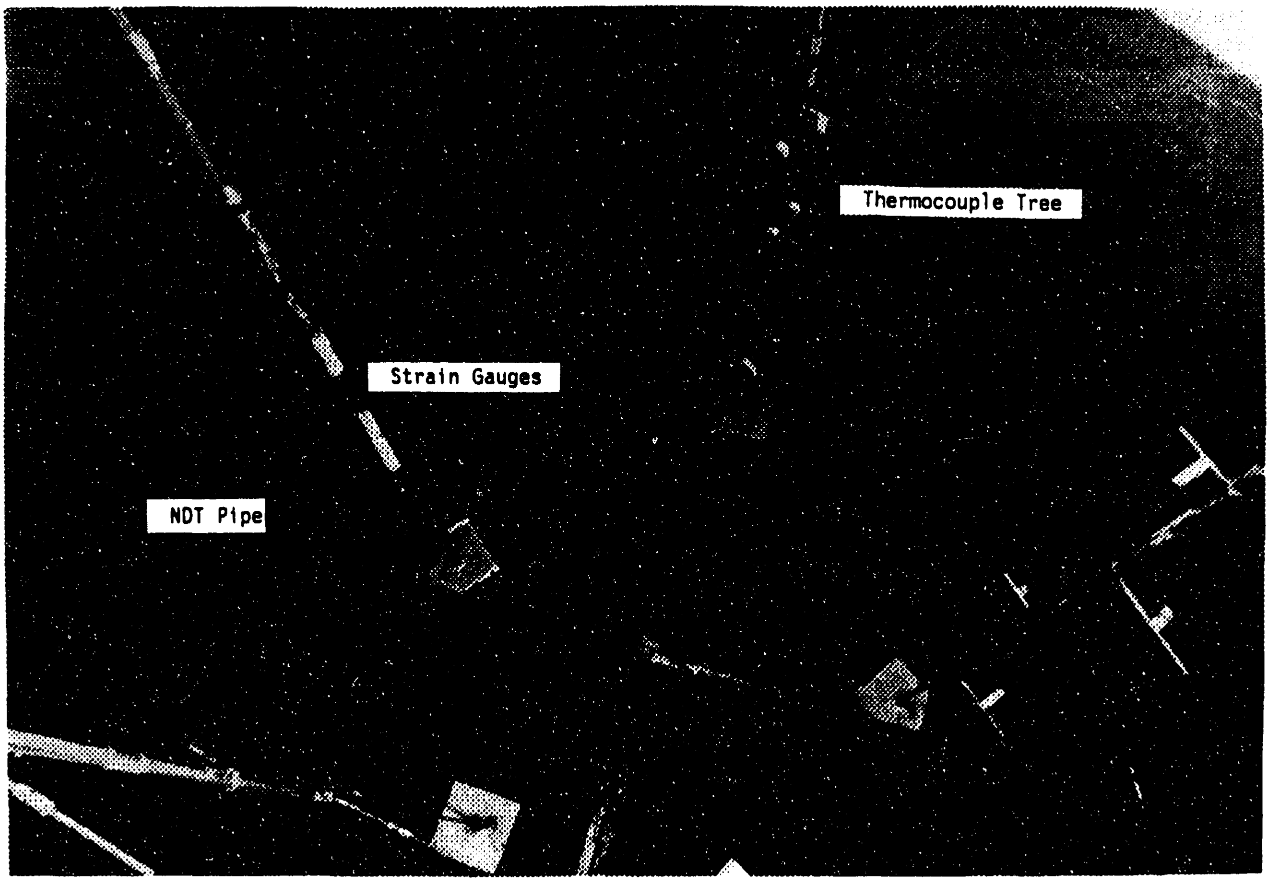


FIGURE 2.8. Instrumentation Inside Gradient Mold

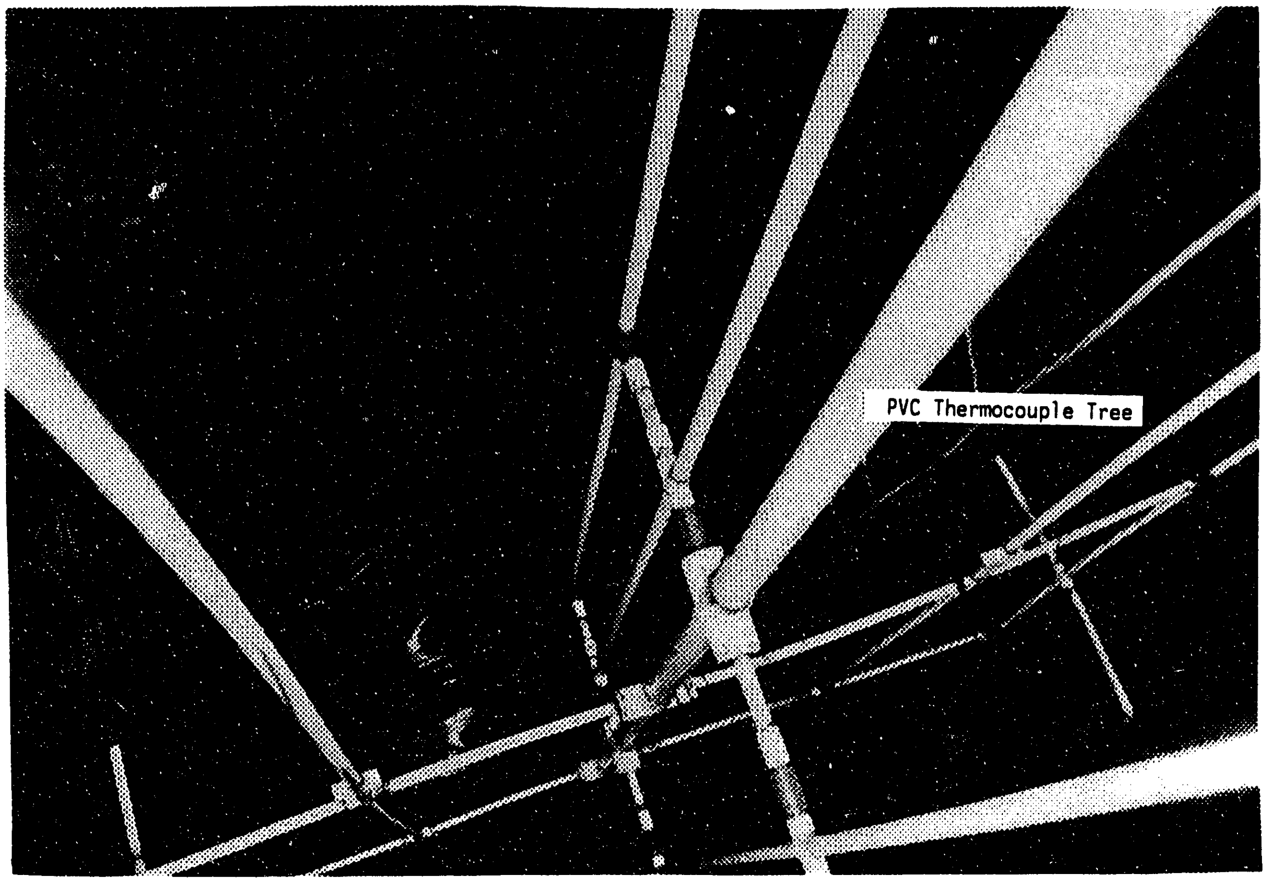


FIGURE 2.9. PVC Thermocouple Tree in Lift Mold

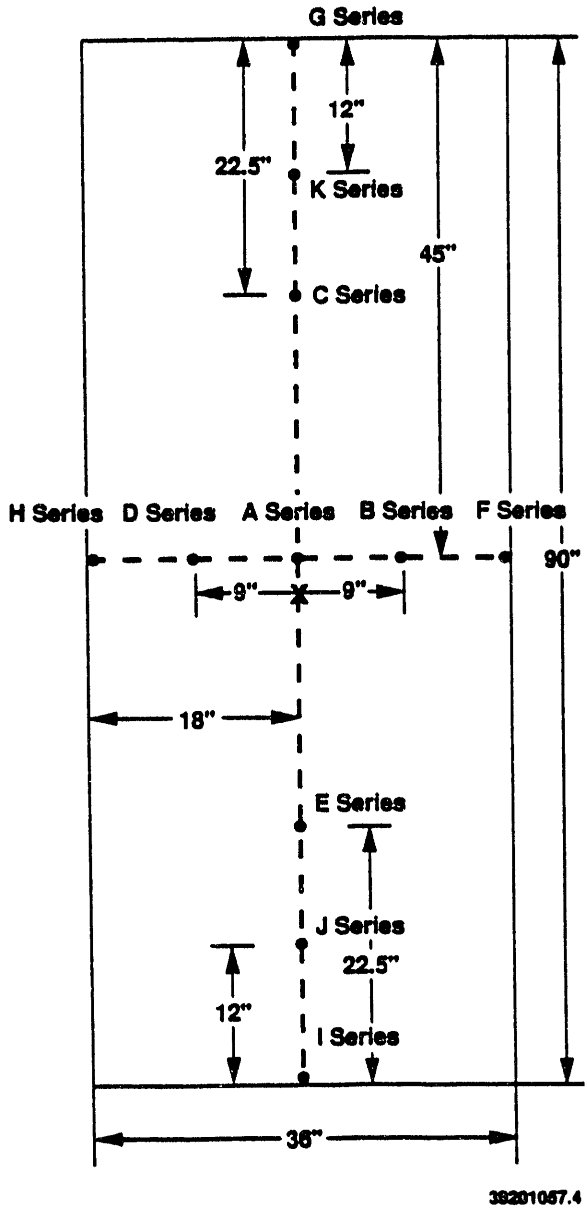


FIGURE 2.10. Thermocouple Placement in Lift Mold (top view)

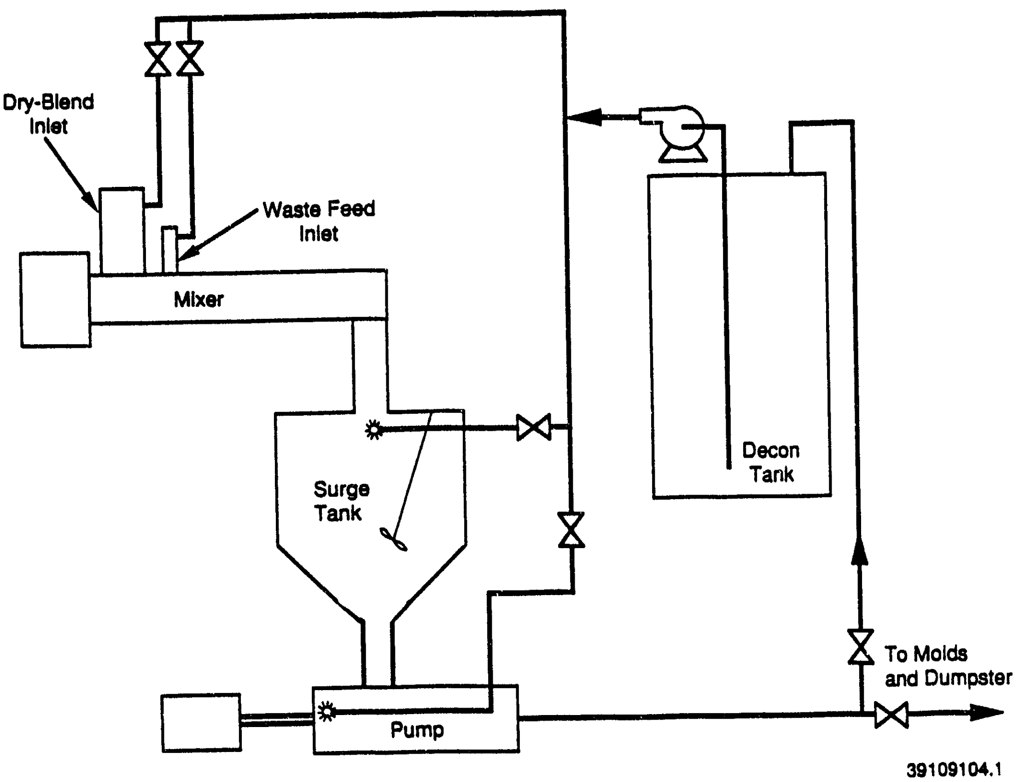
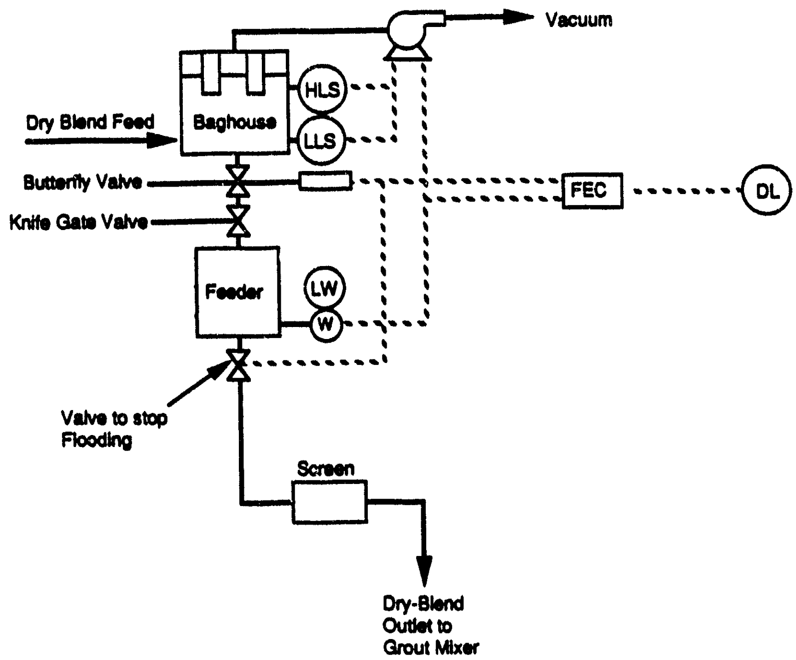


FIGURE 2.11. Decontamination/Rinse System



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

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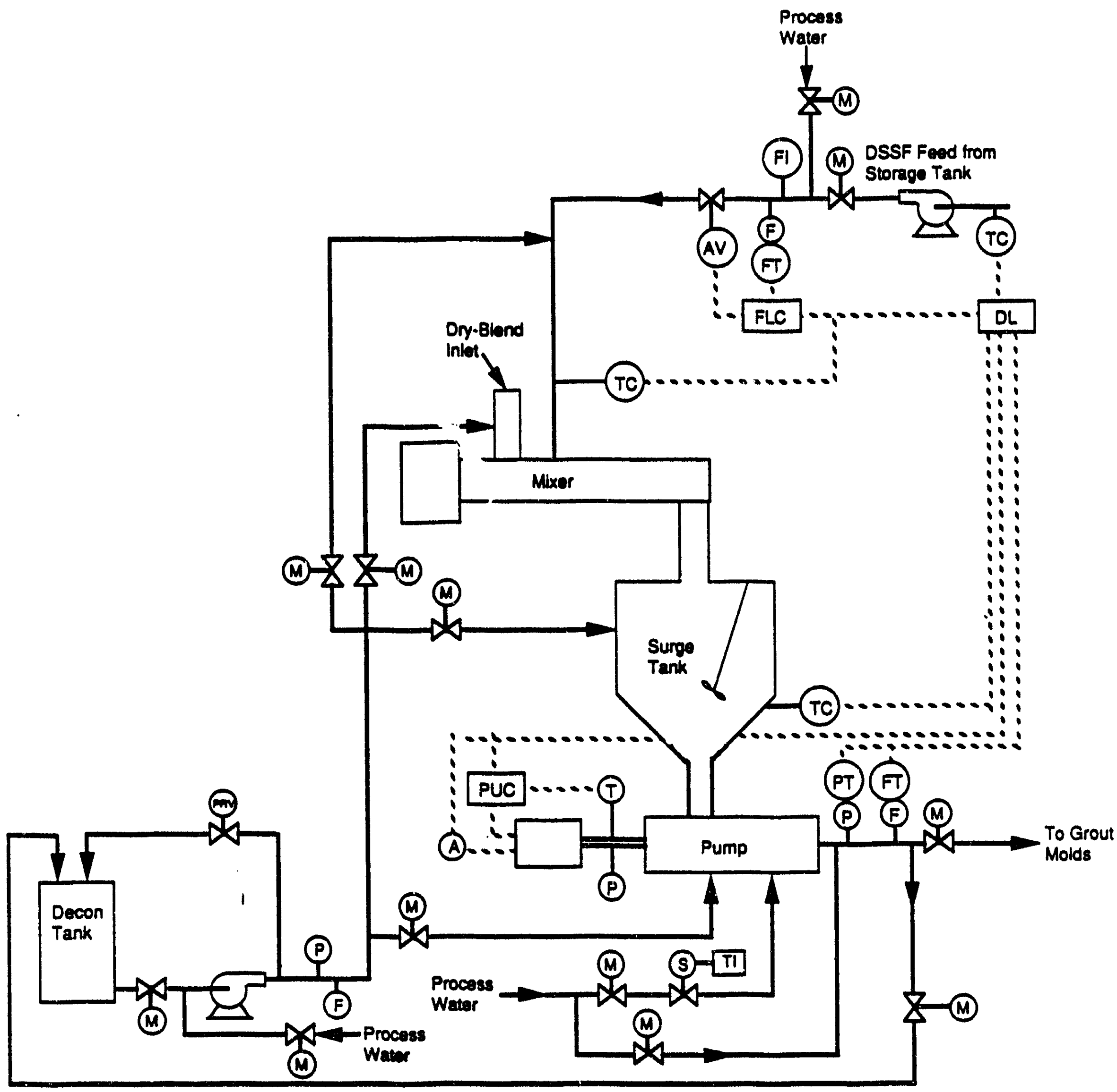
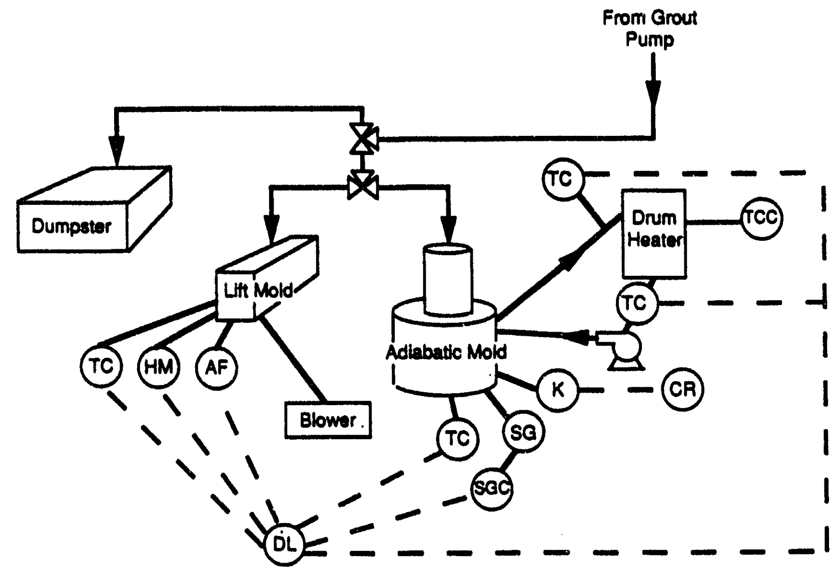
FIGURE 2.12. Process Instrumentation



A	Amps	HLS	High-Level Switch	R	Rotameter
AF	Air Flowmeter	HM	Humidity Meter	S	Solenoid Valve
AV	Air Actuated Valve	K	Thermal Conductivity Probe	SG	Strain Gauge
CR	Chart Recorder	LLS	Low-Level Switch	SGC	Strain Gauge Conditioner
DL	Data Logger	LW	Low-Weight Switch	T	Tachometer
EV	Electrical Valve Actuator	M	Manual Valve	TC	Thermocouples
F	Flowmeter	MT	Manual Three-Way Valve	TCC	Temperature Controller
FEC	Feeder Control	P	Pressure Gauge	TI	Timer On/Off
FI	Flow Indicator	PRV	Pressure Relief Valve	W	Weight Transducer
FLC	Flow Control	PT	Pressure Transducer		39201057.2
FT	Flow Totalizer	PUC	Pump Control		

FIGURE 2.12. Process Instrumentation (Cont)



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

39201057.3

FIGURE 2.12. Process Instrumentation (Cont)

3.0 MATERIALS

Two main materials are mixed to produce the grout waste form. The first material is known as the dry-blend. The dry-blend is comprised of a blended mixture of the various dry materials that control the grout slurry properties and react to form the solidified waste form. The second material is the waste slurry. This material contains the water necessary for the cementitious reactions and the dissolved/suspended waste solids. The specifics of the dry-blend and waste slurry used in this pilot-scale run are described below.

3.1 DRY-BLEND

The two dry-blend formulations used in the pilot-scale grout runs are shown in Table 3.1. The type II portland cement was obtained from Ash Grove Cement in Spokane, WA; the class F fly ash was obtained from Pozzolanic Northwest in Mercer Island, WA; and the attapulgite clay was purchased from Floridin Company in Quincy, FL. A 5-lb sample of each dry-blend ingredient was sent to the WHC Analytical Laboratories. These samples were used to determine the baseline data for the Fourier Transform Infrared (FTIR) spectrometry technique, which was used to confirm the proper quantity of each component in the blended material.

TABLE 3.1. Dry-Blend Formulation

Component	Baseline Formulation Weight% (± 5 relative wt%)	Adjusted Formulation Weight% (± 5 relative wt%)
Class F Fly Ash	66.0	68.3
Type II Portland Cement	20.0	20.7
Attapulgite-150 Clay	14.0	11.0

The DMF was used to mix the dry-blend material required for the pilot-scale tests. Use of the DMF facility was important because the dry-blend mixing method has been shown to be an important consideration when attapulgite clay is one of the dry-blend components (Lokken et al. 1987). The dry-blend for the main portion of the pilot-scale grout run was blended in eight 5000-lb batches. A slightly modified dry-blend formulation that had less clay was used for the fourth lift. This dry-blend was produced in two 4000-lb batches.

A 5-lb sample of each batch of blended material was sent to Teo Rebagay in WHC Analytical Laboratories for FTIR spectrometry to determine the amount of each component. The results of these analyses for the first eight batches are shown in Table 3.2. All eight batches blended for the main portion of the production scale run were within the ± 5 relative wt% tolerance.

The analyses of the samples of the modified formulation are shown in Table 3.3. The analyses show that the cement content was slightly below the allowable ± 5 relative wt% tolerance. However, the seals on the samples sent to the WHC Analytical Laboratories had been broken, and the integrity of these samples was suspect. Therefore, an additional sample of material was taken

TABLE 3.2. Dry-Blend Analyses

Batch #	Attapulgite Clay	Portland Cement	Class F Fly Ash
Target Value	14.0 ± 0.7	20.0 ± 1.0	66.0 ± 3.3
1	14.27	19.93	66.21
2	13.95	20.25	65.23
3	14.18	19.77	63.78
4	13.87	19.66	63.21
5	13.93	19.86	68.44
6	14.14	20.03	66.36
7	13.99	19.88	68.00
8	13.80	20.49	68.03

TABLE 3.3. Modified Dry-Blend Analyses

Batch Number	Attapulgite Clay	Portland Cement	Class F Fly Ash
Target Value	11.0 ± 0.55	20.7 ± 1.04	68.3 ± 3.42
1[a]	10.54	19.44	70.37
2[a]	10.47	19.42	70.02
Sealed Sample	10.87	20.49	70.11

(a) Seals on samples sent to laboratories had been broken.

from a sealed container intended for other studies. Analyses of this sample indicated that all materials fell within the ± 5 relative wt% tolerance. Even if the cement was slightly low, this dry-blend composition was adequate to determine if the modification to the dry-blend formulation would produce the desired results.

The dry-blend was transferred to PNL in production transport trucks and pneumatically transferred with the Vac-U-Max vacuum wand directly from the top of the transport truck to the storage bin of the dry-blend feeder.

3.2 106-AN SIMULATED WASTE

The general reference to 106-AN waste refers to the waste in Tank 241-AN-106. However, before the waste in Tank 241-AN-106 is treated, it will be transferred to an agitated holding tank (102-AP) that already contains the heel of a waste from an earlier PSW grout run. Thus, the pilot-scale run attempted to simulate this combined waste. A total of 3500 gal of simulated waste were required to complete the pilot-scale test.

The pilot-scale run used the best available estimate of the composition in the feed tank after combining the wastes. The information for this estimate (see Table 3.4) was obtained from D. W. Hendrickson, WHC (Hendrickson 1992).

The available analyses for the EPA target toxic metals indicate that silver, barium, cadmium, mercury, and lead were below the detection limit. For the pilot-scale run, it was assumed that these materials were not present. Arsenic and selenium were detected at levels about an order of magnitude below the regulated limit. These chemicals were not added to the simulated waste since they were present in such small quantities. The only toxic metal that was found in significant quantities was chromium (544 mg/liter). The ability of grout waste forms to immobilize these quantities of chromium has been established in earlier laboratory investigations and pilot-scale runs (Lokken 1992). As a result, chromium was not added to the simulated waste as a waste minimization activity.

In formulating the final recipe for the simulated waste, additional OH^- is added as NaOH to compensate for the addition of aluminum as $Al(NO_3)_3 \cdot 9H_2O$, boron as $Na_2B_4O_7 \cdot 10H_2O$, and glycolate as glycolic acid. The sodium concentration is initially allowed to float, and the final concentration results only from the addition of other Na containing compounds. Initial chemical analyses of the waste showed that the Na and PO_4^- were significantly lower than expected. Analyses of the "anhydrous tri-sodium phosphate" showed that hydrated sodium phosphate had been shipped by the vendor. Additional tri-sodium phosphate was procured and added to correct the problem. The simulated waste recipe is shown in Table 3.5.

The target composition and final analyses of the simulated waste are shown in Table 3.4. The total organic carbon was analyzed using a Xertex-Dohrmann Model DC-80 TOC analyzer. The solution was analyzed for anions using a Dionex Series 4000i, Ion Chromatograph (IC) and for cations using a Jarrel-Ash Model 975 Plasma Atomcomp, Inductively Coupled Plasma Spectrometer (ICP). The analyzed values for the main constituents agreed fairly well with the target values. Some of the minor constituents such as boron, calcium, silicon, and magnesium were higher than the target value. This is believed to result from mineral impurities in the process water or from impurities present from earlier tests conducted in the waste feed tank.

TABLE 3.4. Simulated 106-AN Waste Composition

Species	Analysis Moles/Liter (± 5%)	Target Moles/Liter
Al	0.383	0.341
B	0.016	0.0026
Ca	0.004	0.00186
Na	3.77	3.954
Ni	0.0011	0.00104
Si	0.008	0.00158
P	0.211	0.196
K	Not Analyzed	0.246
Mg	0.0010	0.000107
Cl^-	0.093	0.0675
NO_2^-	0.536	0.534
NO_3^-	1.279	1.130
SO_4^{2-}	0.029	0.0273
PO_4^{3-}	0.173	0.196
OH^-	Not Analyzed	0.497
TOC[a]	0.248	0.238
CO_3[b]	0.359	0.341

(a) Calculated from EDTA, HEDTA, glycolate, and citrate additions
(b) Calculated from total carbon and TOC values

TABLE 3.5. Simulated Waste Recipe for Pilot-Scale Test

Chemicals	Batch Size grams/liter	grams/gal	lb/3500 gal	Kg/3500 gal
$NaNO_3$	8.580	32.48	250.66	113.7
NaOH	75.22	284.7	2196.9	996.5
$Al(NO_3)_3 \cdot 9H_2O$ (60wt%)	213.3	807.2	6228.0	2825.0
$Na_3(PO_4) \cdot 12H_2O$[a]	32.10	121.5	937.6	425.3
$Na_3(PO_4)$[a]	18.26	69.13	533.5	242.0
$NaNO_2$	36.89	139.6	1077.4	488.7
Na_2CO_3	36.21	137.1	1057.5	479.7
KCl	1.836	6.949	53.62	24.32
NaCl	2.501	9.466	73.04	33.13
Na_3(Citrate)$\cdot 2H_2O$	2.501	9.466	73.04	33.13
$Na_2B_4O_7 \cdot 10H_2O$	0.248	0.939	7.242	3.285
Na_2SO_4	3.889	14.72	113.6	51.52
$Ni(NO_3)_2 \cdot 6H_2O$	0.302	1.143	8.821	4.001
$Ca(NO_3)_2 \cdot 4H_2O$	0.439	1.662	12.82	5.816
Na_4(EDTA)$\cdot 2H_2O$	1.415	5.356	41.34	18.75
Na_3(HEDTA)$\cdot 2.5H_2O$	5.293	20.03	154.6	70.12
H(Glycolate)(70wt%)	0.924	3.497	26.98	12.24
$Mg(NO_3)_2 \cdot 6H_2O$	0.0276	0.1046	0.807	0.366
COLLOIDAL SiO_2 (40WT%)	0.238	0.900	6.942	3.149

(a) Both hydrated and anhydrous tri-sodium phosphate were added due to the vendor shipping the wrong materials in the initial order.

4.0 GROUT PRODUCTION

The grout in this pilot-scale test was produced in four different runs. The first run produced enough grout to fill the gradient mold and the first lift in the lift mold. This run is referred to as the main production run. The second, third, and fourth runs were short production runs that produced enough grout to complete the second, third, and fourth lifts in the lift mold. These smaller runs are referred to as the lift pours. The observations made and data collected during these runs are discussed in the following sections.

4.1 MAIN PRODUCTION RUN

The main grout run was conducted on April 16, 1992, and started at 9:48 a.m. The plan was to start production, complete the grout pour into the gradient mold, and then pour the first lift into the lift mold. While filling the gradient mold, four 55-gal drums were filled for WHC core drilling tests. Production went as planned except for one shutdown, which occurred after 218 minutes of elapsed run time. This shutdown was caused by a blockage in the waste feed line. When the reduced waste feed flow was first noticed, attempts were made to add process water to the waste feed to avoid a shutdown. However, enough thick grout was produced to clog the mixer. Grout started to backup into the dry-blend inlet, and a shutdown could not be avoided. The shutdown lasted 58 minutes while the mixer was cleaned and the waste line was back-flushed. The rinse water generated from cleaning the mixer and the water from the timed flush of the grout pump inlet were pumped to the dumpster prior to restarting grout production. This shutdown is the reason for the deviations that appear in the run information between 218 and 276 minutes of elapsed run time.

The nominal composition of the grout produced was 8.7 lb of dry-blend per gal of waste. The planned production rate of 10 gpm of grout required a waste flow of 6.94 gpm and a dry-blend feed rate of 3623 lb/hr. An initial water back-flush of the waste line was required before waste flow could be established. After the flush the startup went smoothly, and the nominal composition was being produced within 1 minute. Sampling during the grout run went as planned. Slurry samples from the surge tank and at the grout discharge were taken every 30 minutes. Collection of the slurry samples was timed to investigate the effects of different pumping rates and variations in the mix ratio. Waste feed just before the pinch valve and dry-blend from the active hopper were sampled once every hour.

4.1.1 Grout Pump Speeds and Flow Rates

Once the operating level in the surge tank was reached, the surge tank agitator and the grout pump were started. Figures 4.1 through 4.3 show the pump characteristics obtained during the main run. A pump speed between 204 and 224 RPM was required to balance the mixer and pump outputs and to maintain the proper level in the surge tank. Figure 4.1 shows the measured grout flow rates versus pump speed while producing grout. The linear nature of the data indicates that the deviations in the grout flow were directly related to changes in the pump speed. The average grout production rate while operating was 10.0 gpm.

4.1.2 Grout Pump Amperage

The pump amperage and speed are shown in Figure 4.2. The shapes of these curves match very closely, which indicates that variations in pump power were the direct result of changing the pump speed. No significant trends in the pump power requirements were noted that could be tied to changes in the grout equipment during processing (e.g. stator swelling, stator wear, grout buildup, etc.).

4.1.3 Grout Pump Discharge Pressures

The pump discharge pressure and pump speed are shown in Figure 4.3. The discharge pressure while filling the gradient mold at normal pumping speeds was generally 63 to 78 psi. Deviations outside of this range were the results of changes in pump speed or pressure drops that occurred when the grout flow was diverted to fill the 55-gal drums. While filling the drums, the grout was not pumped to the top of the drop chute. The discharge pressure dropped to 41 to 46 psi while pouring the first lift due to the shorter pipe length and the reduced head loss when no longer pumping to the top of the drop chute.

The predicted pressure drops while pumping to the gradient and lift molds were calculated using Equation (4.1):

$$\Delta P_{TOTAL} = \frac{3.9V^2fl}{D(100)} + \frac{h\rho}{19.24} + 2\left(\frac{Q}{C_V}\right)^2\frac{\rho}{8.34} \qquad (4.1)$$

Where ΔP_{TOTAL} = predicted pressure drops, psi
ρ = grout density, lb/gal (13.0 lb/gal)
V = velocity, ft/sec (9.04 ft/sec)
f = friction factor (0.008)
D = ID of pipe (0.824 in.)
l = pipe length, ft (GRAD - 135.5 ft, LIFT - 109.25 ft)
h = head, ft (GRAD - 25.67 ft, LIFT - 1.67 ft)
Q = grout flow, gpm (10 gpm)
C_V = three-way valve flow coefficient, dimensionless (7)

The first term in this equation accounts for the frictional pressure drop (Riebling 1991). The second two terms account for the pressure drop due to the elevation difference between the pump and the grout pipe outlet and the pressure drop due to the three-way valves (Crane 1988). The calculated pressure drop for the piping to the gradient mold and lift mold was 78.2 psi and 51.5 psi, respectively. The main purpose for this calculation was to determine if the simplified method for calculating the frictional pressure drop in the Grout Formulation Standard Criteria Document (Riebling 1991) would predict a reasonably accurate but conservatively high pressure drop valve. After accounting for the valves and elevation differences, the predicted frictional pressure drop is reasonably accurate and conservatively high.

4.1.4 Process Temperatures

The target temperature for grout entering the molds was 40°C. Experience with laboratory grouts suggested that a waste temperature of 45°C would produce a 40°C grout when mixed with ambient temperature dry-blend. Figures 4.4 through 4.6 show the temperatures at various points in the process. The waste inlet temperature was quite constant over the entire run at 47°C, and the grout in the surge tank had a constant temperature of 43°C.

The temperature of the grout near the pipe discharge was slightly more variable and ranged from a high of 43°C to a low of 37°C, with the average temperature of the grout (excluding down time) being 40.7°C. Part of the temperature variability could be due to the programmed flush, which occurred every 10 minutes during the main grout production run. If the data logger took the data point right after the cold process water was added to the pump inlet, the temperature of the grout at the discharge would dip slightly. The discharge temperature of the grout produced after the shutdown shows significant temperature dips every 10 minutes.

4.1.5 Grout Mix Ratio

Figure 4.7 shows how the grout mix ratio varied during the pilot-scale run. While processing the nominal mix ratio of 8.7 lb/gal, the mix ratio always stayed well within the allowable ± 0.5 lb/gal. Between 144 and 194 minutes of elapsed run time, the grout ratio was changed to investigate grout production in the upper portion of the allowable operating window. Grout processing was not significantly different during this time frame, which indicated that the upper portion of the acceptable operating window could be mixed and pumped without operational problems. The average mix ratio for the entire grout run after accounting for the water added with the timed rinse was 8.79 lb/gal.

4.1.6 Final Rinse Procedure

Twenty-five gal of water were used to rinse the grout mixer, surge tank, grout pump, and the grout pipe leading to the lift mold. The water was pumped through the grout pipe at a rate of 10 gpm. The water from this rinse (minus the 3 to 4 gal that remained in the pipe) was placed on top of the first lift of grout in the lift mold. An additional 10 to 15 gal of water were recirculated through the pilot-scale equipment for 10 minutes using the decontamination/rinse system. This rinse water was pumped to the dumpster.

After rinsing, the drop chute penetration into the gradient mold was plugged with insulation, and the lift mold inspection port was sealed. The lids on the 55-gal drums were also replaced and sealed.

4.2 LIFT POURS

The three pours that completed the lift mold were conducted essentially the same as the main pour. Each 2-ft lift required 350 gal of grout and approximately 35 minutes of production time. The pump characteristics were the same as the main run, and the pressure at the pump discharge ranged between 40 and 55 psi. Minor differences in the runs are discussed below.

4.2.1 Lift 2

The temperature of the grout entering the mold in the second lift was slightly lower (37° to 39°C) than the grout produced in the main pour and the other lift pours. This lower temperature was due to a slightly lower waste feed temperature. Grout samples were taken while pumping at 10 gpm after 10 and 25 minutes of grout production. Approximately 20 gal of rinse water were placed on top of the second lift.

4.2.2 Lift 3

The temperature of the grout entering the mold during the third lift was 39° to 40°C. Grout samples were taken while pumping at 10 gpm after 10 minutes of production. After the first sample was obtained, the grout production rate was slowed to approximately 5.5 gpm to allow a slurry sample to be obtained while pumping at 2.5 gpm. The lower production rate was necessary to stay within the surge tank capacity while pumping at the lower rate for a sufficient time period to assure that the grout collected at the discharge had been pumped through the entire length of the pipe at the lower rate. After the low pumping speed slurry sample was taken, the pump speed was increased and the production rate was returned to 10 gpm for the remainder of the run. At the completion of the third lift, all rinse water was directed to the dumpster, and the liquid remaining on the grout surface was removed.

4.2.3 Lift 4

The fourth lift used a slightly modified dry-blend formulation (see Table 3.1), which reduced the amount of attapulgite clay. Grout was produced at a rate of 10 gpm with a nominal mix ratio of 8.4 lb/gal. No significant changes were noted in the pump characteristics as a result of this formulation change. The average measured mix ratio for the fourth lift pour was 8.46 lb/gal. The grout for the fourth lift entered the mold at 41° to 43°C. Slurry samples were obtained while pumping at 10 gpm after 10 and 25 minutes of production time. Two additional 55-gal drums were poured while completing the fourth lift. At the completion of the run, the drum lids were replaced and sealed to prevent moisture evaporation. These drums were used by WHC for NDT testing, but they were also used to see if the amount of drainable liquid formed increased with the adjusted formulation. Approximately 24 hours after completion of the fourth lift, the drums were checked and found to have no free liquids. Approximately 20 gal of rinse water were placed on top of the fourth lift. After completing the rinse, grout production was restarted and additional slurry samples were obtained for critical flow rate calculations.

4.3 HANDLING DRY-BLEND TRANSFERS

The first attempts to produce grout occurred on April 14, 1992, 2 days before the main production run. These first attempts to produce grout were not successful due to dry-blend flooding problems that occurred during the transfer of dry-blend from the storage hopper to the active hopper. The initial settings for the high and low levels in the active hopper were 80% and 20%. These values are the vendor-recommended levels and the levels used in the DSSF pilot-scale run. When the dry-blend flooding occurred, the grout at the mixer discharge became too thick and would not pass through the 3-in. line to the surge tank. The grout buildup in the discharge required a system shutdown. These extremely thick mixes, however, did not affect mixer performance (e.g. did not cause a shear pin failure).

Three separate startups were attempted, but all were aborted due to the flooding problems. In each case, the flooding occurred near the end of the refill cycle. Similar problems were encountered during the PSW pilot-scale run (Fow et al. 1987) and were thought to be the result of fluidization of the dry-blend by the Vac-U-Max system. Attempts to solve the flooding problems assumed that fluidization of the dry-blend was the cause of the problem. During the last startup attempt, the blower on the Vac-U-Max system was shut off to prevent fluidization of the grout. However, dry-blend flooding still occurred, and it was decided to postpone tests until the problem was solved.

The 450 gal of grout produced on this first day had the desired mix ratio and were placed in the mold under the desired conditions. The level in the gradient mold (approximately 14 in.) was below the first layer of thermocouples, and the additional rinse water required to clear the pilot-scale equipment after the shutdowns was sent to the dumpster. Thus, the aborted startup did not significantly affect the results obtained from the gradient mold.

The dry-blend flooding problem was solved the following day. When this particular dry-blend is transferred from the storage bin, the fall is sufficient to fluidize the material. Measurements of the bulk density show that dry-blend poured into a container has a bulk density of 1.03 g/cc, which increases to 1.28 g/cc when tapped to remove trapped air. The low bulk density material just after pouring is very fluid. In fact, prior to the final lift pour, material was dumped from the storage hopper into an empty active hopper while the rest of the system was deactivated. The dry-blend material was fluid enough to pass through the feed auger, scalping screen, and grout mixer and fill the surge tank.

Apparently, when the dry-blend reached the 20% full level and the system called for more material from the storage hopper, the level was low enough to allow the fluidized material to pass through the augers and flood the mixer. The solution to this problem was to change the high and low hopper levels in the system software to 90% and 70%. This change accomplished three things that might have contributed to solving the problem: 1) It increased the amount of material in the hopper to act as a cushion and plug paths for the fluidized dry-blend to pass through, 2) it reduced the amount of material delivered during a single transfer, and 3) it increased the residence time in the active hopper to allow the air to escape from the dry-blend before reaching the feed augers.

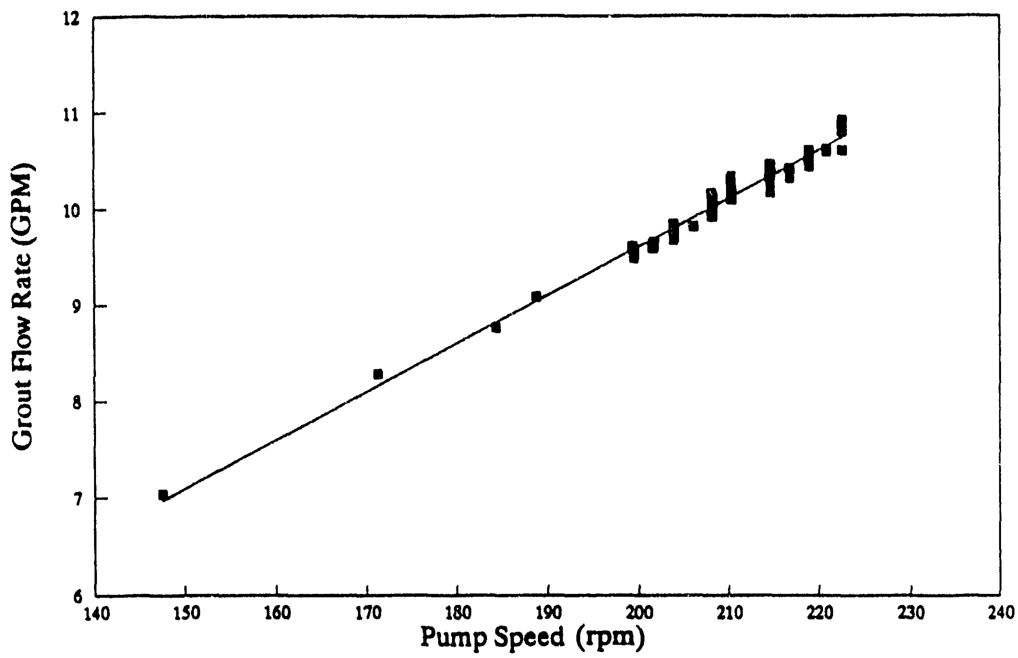


FIGURE 4.1. Grout Pump Speeds and Flow Rates During Main Production Run

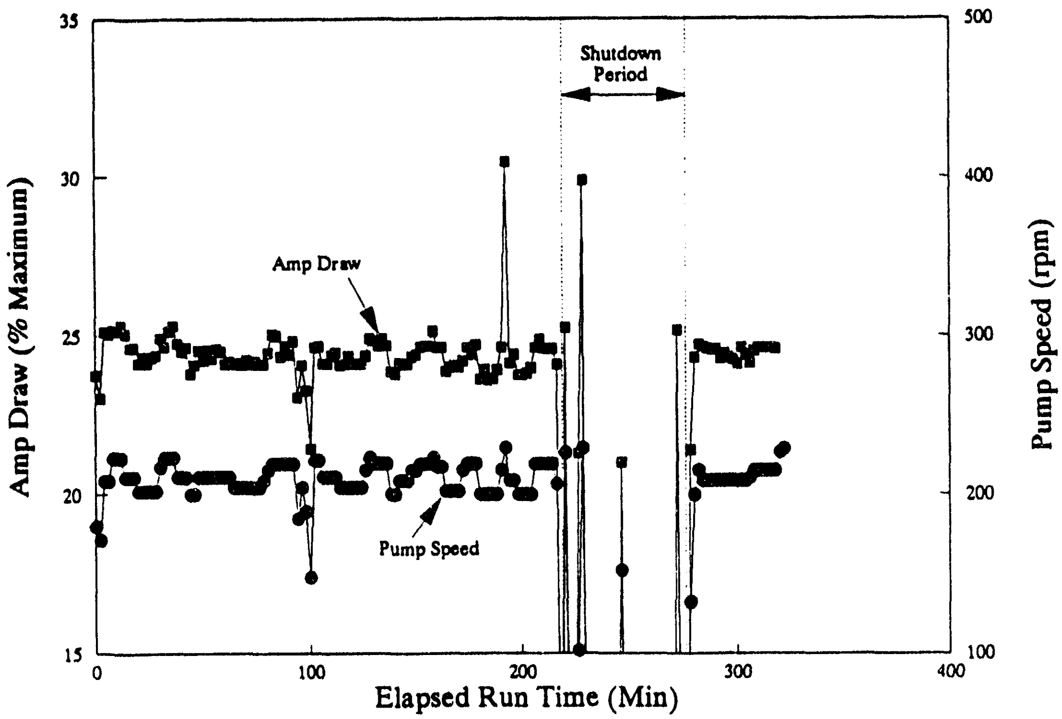


FIGURE 4.2. Grout Pump Speeds and Amp Draw During Main Production Run

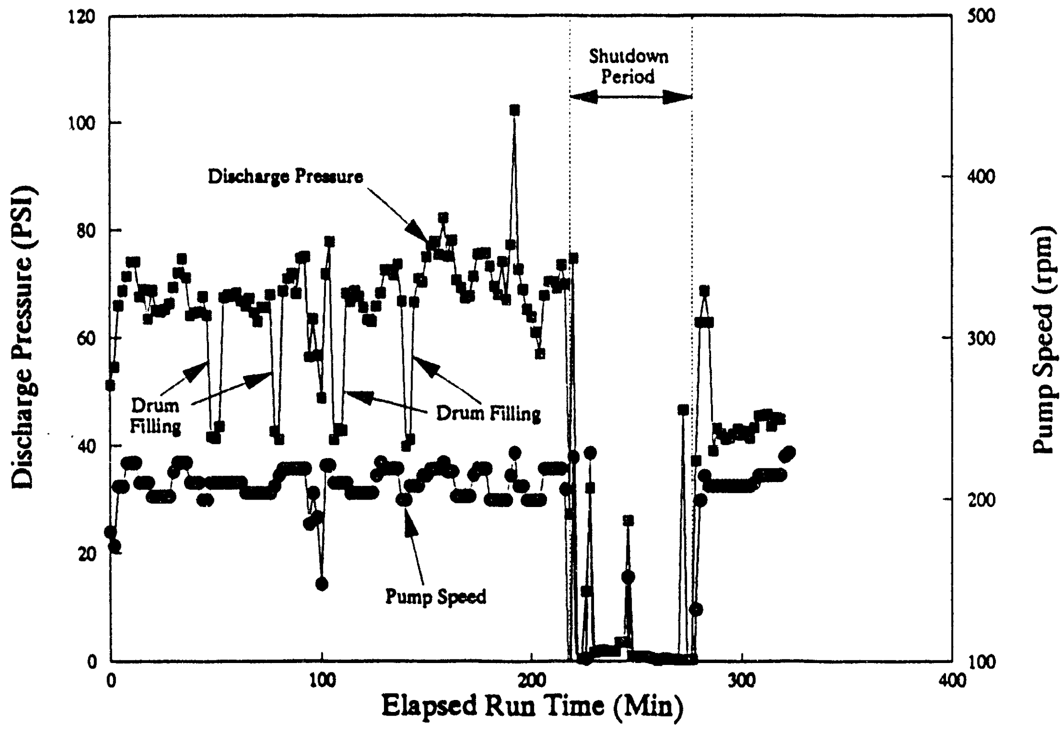


FIGURE 4.3. Grout Pump Speeds and Discharge Pressures During Main Production Run

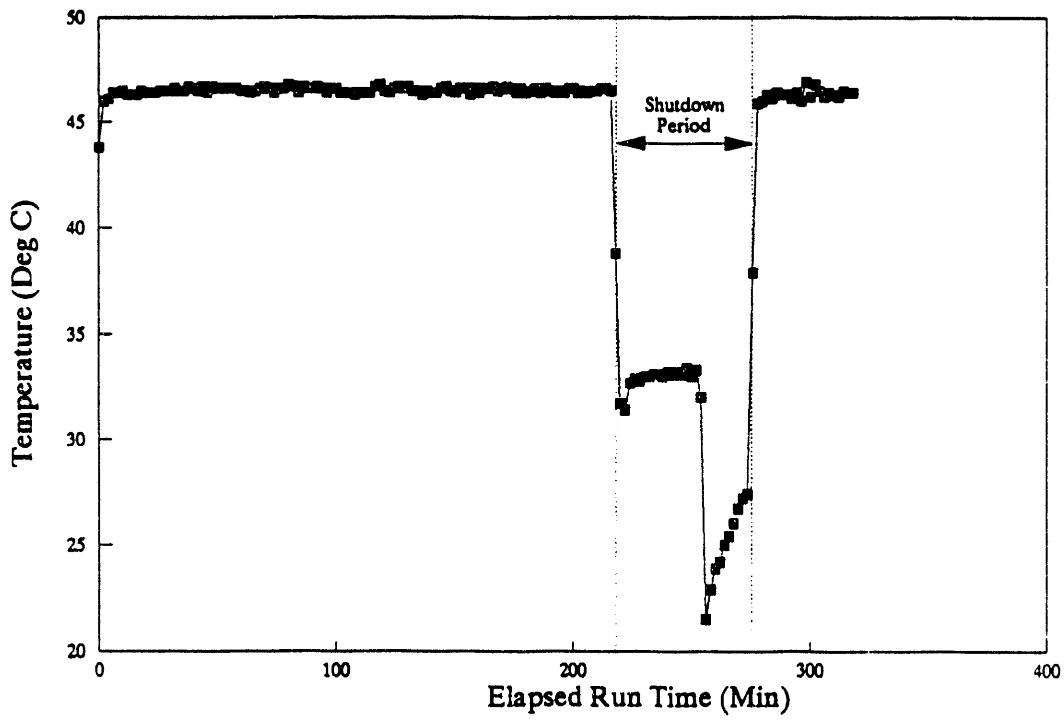


FIGURE 4.4. Waste Inlet Temperature

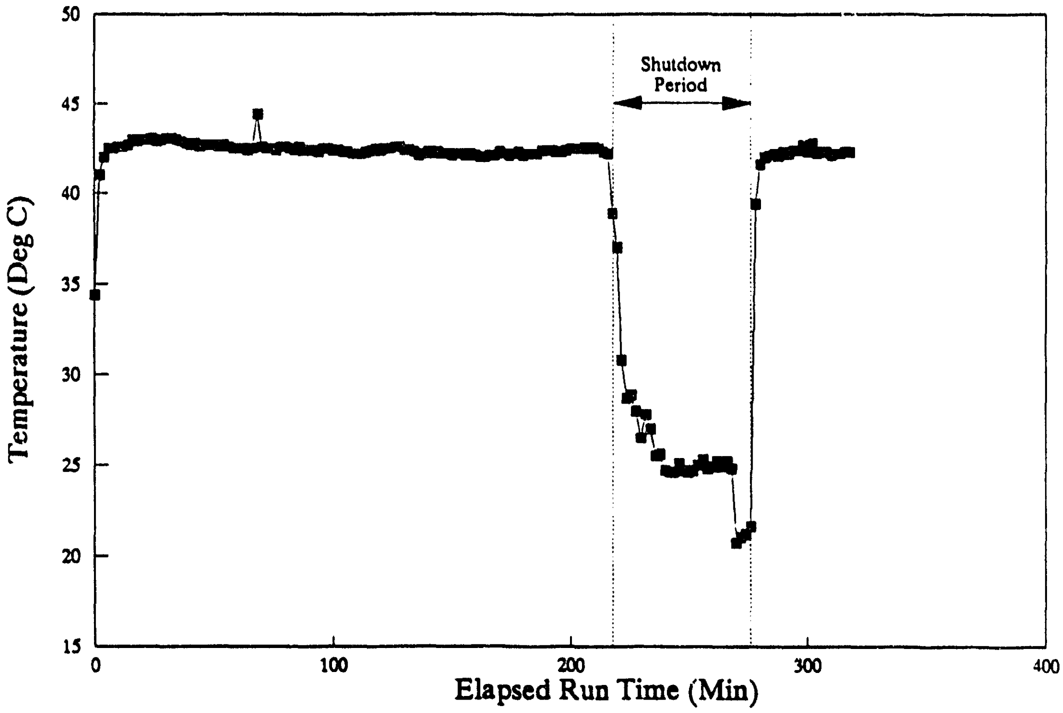


FIGURE 4.5. Temperature of Grout at Surge Tank

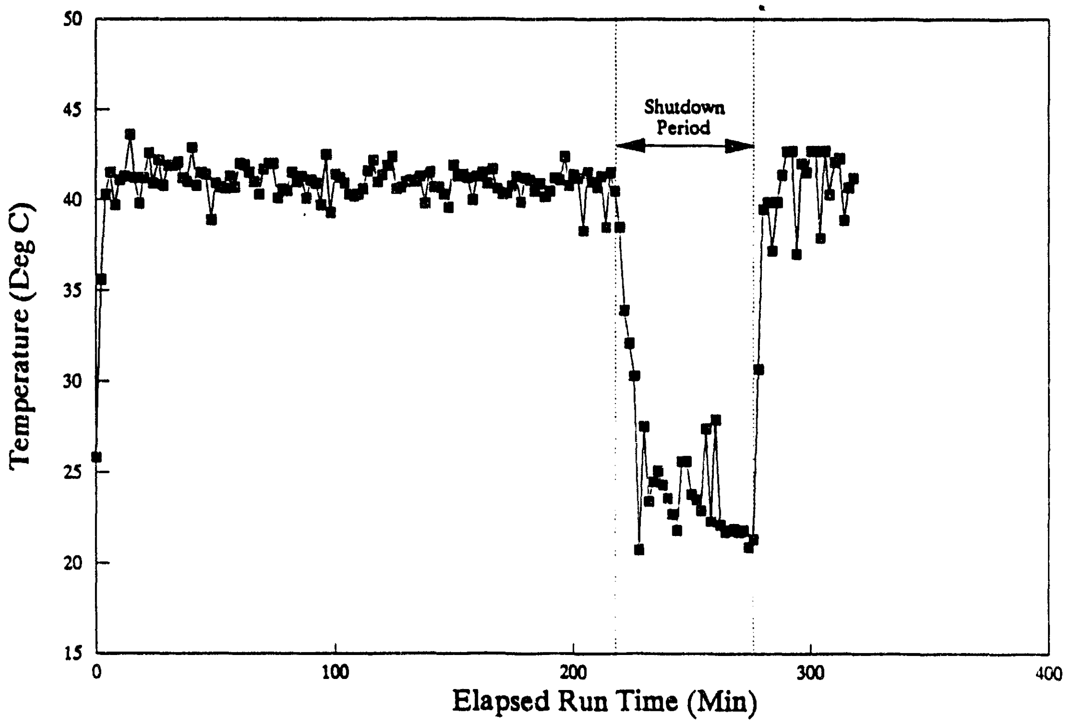


FIGURE 4.6. Temperature of Grout Near Discharge

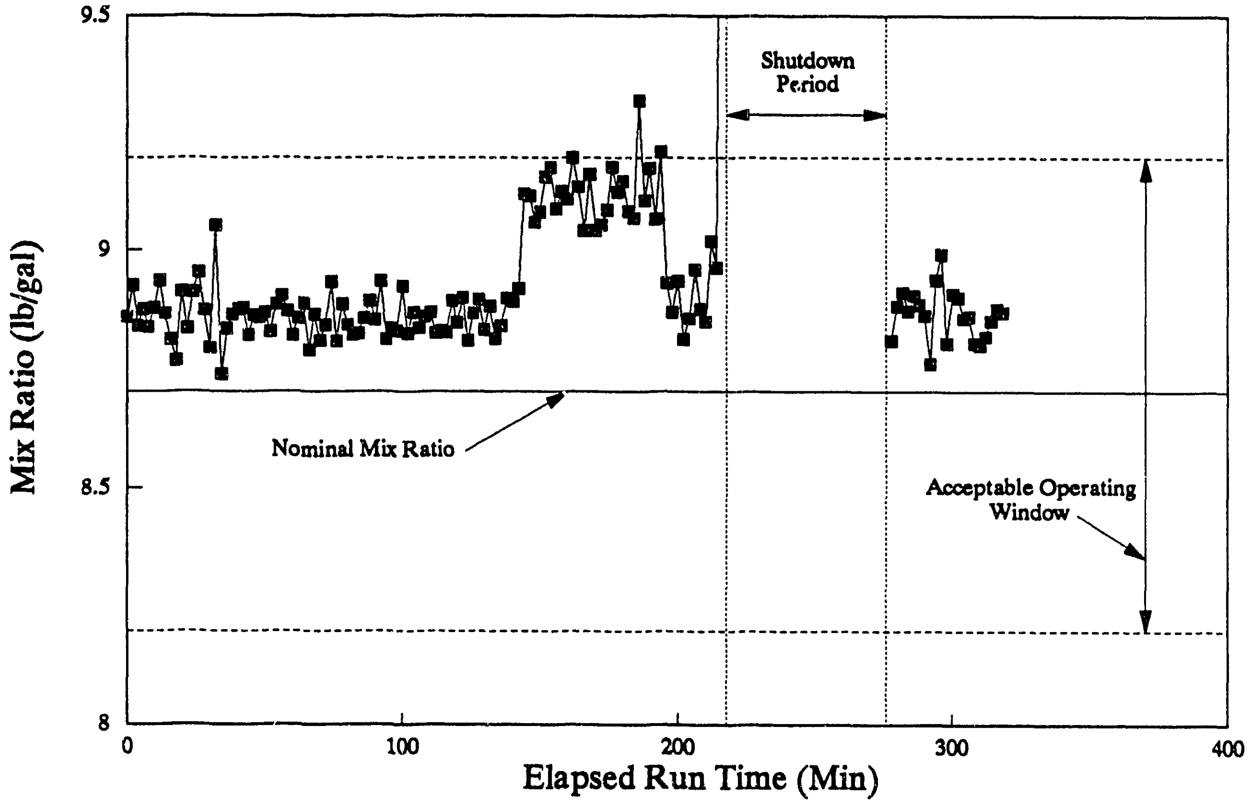


FIGURE 4.7. Grout Mix Ratio During Main Production Run

5.0 GROUT AND GROUT SLURRY PROPERTIES

An important part of the formulation verification process is to determine if the grout formulation, developed with laboratory studies, yields the desired properties when mixed with equipment similar to that used in the production facility. The following sections discuss some of the grout and grout slurry properties obtained from the material produced in the pilot-scale tests. Core samples were also taken from each mold to measure additional properties. The analysis of the core samples will be discussed in a different report.

5.1 RHEOLOGICAL PROPERTIES

The rheological properties of the grout slurry are important for two main reasons. First, after mixing, the grout must be pumped to the disposal vault through several hundred feet of pipe. If turbulent flow is not maintained in the pipe, settling could occur and eventually restrict the flow of grout. The other important slurry property is the grout gel strength. If the grout develops a high gel strength shortly after mixing, the possibility of the grout setting up in the equipment during process interruptions becomes much greater. The rheological properties of the grout produced in the pilot-scale run are described in the following two sections.

5.1.1 Critical Flow Rates

In the production processing scheme, the grout is pumped to the disposal vault through several hundred feet of pipe. The flow through this pipe must be turbulent to assure that settling of the solids in the grout slurry do not restrict the flow of grout. The minimum flow rate that will produce turbulent flow is known as the critical flow rate (CFR). The maximum allowable CFR for a grout formulation that will be processed in the GTF is 60 gpm (Riebling 1991). The critical flow rate is calculated using Equation (5.1).

$$CFR = \frac{\pi D^2}{1.283}\left(\frac{1,129K'\left(\frac{96}{D}\right)^{n'}}{\rho}\right)^{\frac{1}{(2-n')}} \tag{5.1}$$

Where CFR = critical flow rate, gpm

K' = fluid consistency index, lb/ft^2

n' = flow behavior index, dimensionless

ρ = slurry density, lb/gal

D = inside pipe diameter, in. (pilot-scale - 0.824"; production - 1.939")

This equation assumes that a Reynolds number of 2100 is the transition point from laminar to turbulent flow for grout slurries (Smith 1976). The grout slurry samples obtained during the main grout pour and the lift pours were used to determine the densities and rheological properties necessary for the above calculation (see Table 5.1).

Table 5.2 shows the critical flow rates determined from samples taken during pilot-scale testing. Rheological information obtained from samples taken at the surge tank indicated that the grout formulation with 14 wt% attapulgite clay had a critical flow rate of less than 40 gpm. This value was well below the 60 gpm criteria value. These values are also fairly close to the values obtained from laboratory samples prepared with the pilot-scale materials. However, the samples at the pipe discharge yielded critical flow values that were above 60 gpm. This effect, known as shear thickening, was also seen in the PSW pilot-scale run when attapulgite clay was part of the formulation (Fow 1987). The amount of shear thickening observed did not change significantly for different pumping speeds or minor variations in the mix ratio.

The CFR value for the last discharge sample taken during the main pour does not show the shear thickening effect. At first, it was thought that this may be due to the different pipe length when pumping to the lift mold. However, discharge samples taken from the same location in the later pours show significant shear thickening, so the shorter pipe length does not explain the results. The other possibility is that the timed flush activated just prior to taking this last sample, and the grout was slightly diluted with rinse water. The timed flush was not used in the other lift pours to avoid this added variable.

TABLE 5.1. Grout Slurry Properties

Mold	Grout Density (lb/gal)		n′		K′ (lbf/ft^2)	
	Surge Tank Sample	Discharge Sample	Surge Tank Sample	Discharge Sample	Surge Tank Sample	Discharge Sample
Lab 1[a]	13.07		0.8060		2.83E-03	
Lab 2[a]	13.07		0.8050		2.68E-03	
Gradient	12.86	12.62	0.8086	0.4151	1.84E-03	4.47E-02
Gradient	12.93	12.83	0.6956	0.5095	3.99E-03	2.36E-02
Gradient	12.95	12.72	0.6929	0.4954	4.69E-03	2.87E-02
Gradient	12.93	12.70	0.7134	0.4496	3.75E-03	3.38E-02
Gradient	12.94	12.69	0.6746	0.6201	5.20E-03	1.36E-02
Gradient	12.85	12.64	0.6882	0.5188	4.49E-03	2.23E-02
Gradient	12.98	12.86	0.6658	0.5266	5.85E-03	2.49E-02
Gradient	12.97	12.86	0.6890	0.6772	5.08E-03	9.96E-03
Lift 1	13.04	12.93	0.7277	0.5892	4.09E-03	1.03E-02
Lift 2	12.99	13.00	0.8568	0.6285	1.49E-03	9.70E-03
Lift 2	12.88	12.87	0.8466	0.6491	1.46E-03	6.97E-03
Lift 3	13.05	12.97	0.8701	0.7202	1.12E-03	3.62E-03
Lift 3	13.07	13.06	0.7961	0.8390	2.13E-03	1.63E-03
Lift 4	12.95	12.97	0.7864	0.6507	1.72E-03	5.13E-03
Lift 4	12.96	12.92	0.7982	0.6239	1.63E-03	5.92E-03
Lift 4	13.11	12.97	0.7511	0.6253	2.46E-03	7.03E-03
Lift 4	13.00	13.00	0.7743	0.6869	1.81E-03	3.40E-03

(a) Laboratory samples prepared with pilot-scale materials

TABLE 5.2. Calculated Critical Flow Rates

Mold	Wt% Clay In Dry-Blend	Mix Ratio (lb/gal)	Pumping Rate (gpm)	Dry-Blend Age (Days)	Critical Flow Rate (gpm)			
					Pilot-Scale		GTF	
					Surge Tank Sample	Discharge Sample	Surge Tank Sample	Discharge Sample
Lab 1[a]	14	8.70	N/A	6	12.7		39.4	
Lab 2[a]	14	8.70	N/A	6	12.1		37.5	
Gradient	14	8.45	13	7	9.1	13.9	28.2	61.3
Gradient	14	8.70	10	9	9.4	13.8	32.8	57.1
Gradient	14	8.70	10	9	10.4	14.8	36.8	61.9
Gradient	14	8.70	10	9	9.8	13.4	33.6	58.1
Gradient	14	8.70	15	9	10.3	16.2	36.9	61.0
Gradient	14	8.70	10	9	9.9	14.0	35.1	57.5
Gradient	14	8.95	10	9	10.8	15.5	38.9	63.1
Gradient	14	8.95	15	9	10.9	17.3	38.4	62.0
Lift 1	14	8.70	10	9	11.2	11.3	37.9	43.7
Lift 2	14	8.70	10	16	9.9	13.0	28.7	48.6
Lift 2	14	8.70	10	16	9.2	11.4	27.1	41.7
Lift 3	14	8.70	10	23	8.2	9.8	23.6	33.5
Lift 3	14	8.70	2.5	23	9.5	9.6	29.8	28.6
Lift 4	11	8.40	10	2	7.6	9.1	24.1	33.3
Lift 4	11	8.40	10	2	7.7	8.9	24.2	33.4
Lift 4	11	8.40	15	2	8.4	10.1	27.8	38.0
Lift 4	11	8.40	10	2	7.4	7.9	24.0	28.0

(a) Laboratory samples prepared with pilot-scale materials.

Additional slurry samples were obtained while pouring the second and third lifts. These lifts used the dry-blend with 14 wt% clay that had been blended for the main run. As a result, the times between blending and grout production were longer for the second and third lift. The thickening effect of attapulgite clay lessens as the storage time of the mixed dry-blend increases when ingredients are sealed (Lokken 1987). The reduced thickening effect for both surge tank and discharge samples can be seen in Table 5.2.

A second sample from the third lift was taken while pumping at a lower rate of 2.5 gpm. The results of this test showed no shear thickening at the reduced pumping speed. The higher CFR value for the second surge tank sample from the third lift indicates that mixing in the surge tank also results in some shear thickening. Due to the lower pumping rate, the dwell time in the surge tank was higher and allowed more time for the surge tank mixer to shear the grout slurry.

Comparing CFR values for 10 and 15 gpm pumping rates showed that little additional shear thickening occurred at flow rates higher than 10 gpm. Results from 15, 10, and 2.5 gpm pumping rates indicate that the shear thickening is dependent on the shear rate. The low flow rate test showed that until some minimum threshold shear rate is obtained, little shear thickening occurs. At higher shear rates, the shear thickening effect levels out. However, there are currently not enough points to determine the threshold shear rate or how rapidly this thickening effect levels out.

The difference in the pipe diameter between the pilot-scale and production system affects the shear rate. For Newtonian fluids in laminar flow, the shear rate is proportional to the velocity and inversely proportional to the pipe diameter (Carleson 1987). Thus, at constant velocity, the pilot-scale pipe results in approximately double the shear rate. However, the production pipe is longer and will shear the grout slurry at a lower rate for a longer period of time. Therefore, it is difficult to determine the extent of shear thickening expected in the production scale system from the pilot-scale results currently available.

The results of the critical flow rate tests did not conclusively determine that the dry-blend formulation with 14 wt% attapulgite clay would exceed the 60 gpm criteria value for the production equipment. However, the results do show the many variables which can affect the performance of the attapulgite clay and that high critical flow rates are possible with the 14 wt% attapulgite clay formulation. In addition, the main purpose for the clay is to control free liquid generation, but the grout in the gradient mold showed no free liquids. The need for evaporation water for grout cooling also reduces the concerns about free liquids. Therefore, reducing the amount of attapulgite clay would reduce the possibility of exceeding the 60 gpm criteria value without jeopardizing other grout properties.

A slightly modified dry-blend was prepared by the DMF for the fourth lift. This formulation reduced the attapulgite content from 14 to 11 wt% while keeping the fly ash/cement ratio constant. The critical flow rates for this adjusted formulation still showed a shear thickening effect, but the highest CFR obtained for the five samples taken was 38 gpm. Two 55-gal drums of this formulation poured while completing the fourth lift had no free liquids after 24 hours. Therefore, the adjusted formulation with 11 wt% attapulgite clay adequately controlled the free liquid and had critical flow rates well below the criterion value even after significant shear thickening. The formulation verification study will determine if free liquids are a problem for acceptable processing conditions different than those of the pilot-scale run.

5.1.2 Restart Pressure Tests

After completion of the fourth lift, tests were conducted to determine the required restart pressures after leaving grout in the pipe for varying lengths of time. The dry-blend composition and mix ratio used for the fourth lift were used for the restart tests. Grout was produced for at least 10 minutes prior to each restart test to ensure that the system had come to a steady state. Just prior to stopping production, samples of grout were taken from the surge tank and the pipe discharge for gel strength measurements (Riebling 1991).

For all restart tests, the pump was set to the full production speed (204 RPM) and accelerated at the maximum motor controller acceleration rate (92 RPM/sec) since the production facility currently does not plan to ramp the pump during restarts. After the desired interruption time, the pump was restarted and the maximum pressure was taken from the pressure gauge at the grout pump discharge.

The relationship between the restart pressure and the gel strength is given by Equation (5.2).

$$P_{RESTART} = P_{GEL} + P_{ACCEL} = \frac{S_G L}{300D} + P_{ACCEL} \qquad (5.2)$$

Where $P_{RESTART}$ = restart pressure, psi

P_{GEL} = pressure required to overcome gel strength of grout, psi

P_{ACCEL} = pressure required to accelerate grout in pipe, psi (pilot-scale - 5.8 psi; production - 30 psi)

S_G = gel strength, lb/100 ft^2

L = pipe length, ft (pilot-scale - 100.5'; production - 525')

D = pipe diameter, in. (pilot-scale - 0.824"; production - 1.939")

This equation was taken from Riebling 1991 and modified to include the pressure required to accelerate the grout in the pipe up to the pumping speed. It was used to either calculate the anticipated restart pressure from the measured gel strength or to calculate the grout gel strength from the observed restart pressure.

Table 5.3 shows the results of the restart tests. After 10-minute and 20-minute interruptions, the measured gel strengths of both the surge tank and the pipe discharge samples were below the 100-lb force/100 ft^2 criterion value (Riebling 1991). The measured restart pressures for these interruption times were significantly higher than the restart pressures calculated from the measured gel strengths. However, gel strengths, back calculated from the observed pilot-scale data and applied to the production system pipeline for the first vault, gave restart pressures well within the pump clearing capacity of 500 psi, and interruption times of up to 20 minutes should not be a problem. However, the difference in the calculated and observed restart pressures indicated that the methodology for calculating the allowable gel strength should be reviewed.

TABLE 5.3. Restart Pressures

Restart Times (Min)	Sample Location	Gel Strength ($lb/100\ ft^2$)		Restart Pressures (psi)			
				Pilot-Scale		Production	
		Measured with Viscometer	Calculated from Pilot-Scale Restart Pressure Data	Calculated from Measured Gel Strength	Measured	Calculated From Measured Gel Strength Data	Calculated from Pilot-Scale Data
10	Surge Tank	21	91.5	14.3	43	49	113
10	Discharge	19	91.5	13.5	43	47	113
20	Surge Tank	34	194.8	19.6	85	61	206
20	Discharge	33	194.8	19.2	85	60	206
30	Surge Tank	155	>293	68.8	>125	170	>294
30	Discharge	260	>293	110.8	>125	265	>294

After a 30-minute interruption, the measured gel strength jumps significantly and is above the criterion value. The first attempt to restart the system tripped the high pressure cutout switch in the p lot-scale system, which was set at 125 psi. Back calculation yields a production system restart pressure of greater than 277 psi. Both the measured and observed results indicate that 30-minute interruptions would not be acceptable for the production system.

A second attempt to restart the pump after the 30-minute interruption resulted in a maximum pressure of 80 psi. Apparently, enough of the gel breaks up during the first restart attempt to reduce the pressure requirements for the second restart attempt. The high 30-minute gel strengths and the initial difficulties clearing the grout line indicated that longer interruptions should not be tested.

5.2 DIMENSIONAL CHANGES

The dimensional changes of the grout in the gradient mold over the first 7 weeks were monitored with two strain gauges. One gauge was oriented axially while the other gauge was oriented radially. These gauges both show the same general behavior (see Figure 5.1). The grout initially expands 100 to 175 microstrains (0.0100% to 0.0175%). This is less than the amount of expansion that would be expected due to thermal expansion of the grout. After the initial expansion peak, which occurs after 16.67 hours, the grout contracts. This contraction levels out after 7 weeks at 525 to 600 microstrains (0.0525% to 0.0600%).

The differences in the axial and radial expansion/contraction curves are due to the different degrees of freedom of movement in the two directions. As the temperature of the grout increased, the grout was free to expand in the axial direction, but was constrained by the mold in the radial direction and probably plastically deformed. This would account for the 75 microstrain difference in the strain gauges seen at the peak expansion. After the grout starts contracting, this 75 microstrain difference is maintained, and the agreement between gauges is quite good.

Significant dimensional changes in the grout would be a concern. Expansion of the solidified grout could damage the vault walls. Significant contraction could leave a void between the cold cap and the cover blocks that would increase the chances of the blocks breaking when the protective barrier is put in place (Riebling 1991). However, the results of these tests indicated that the magnitude of the dimensional changes for this grout formulation would not be a problem. The small amount of expansion that occurs happens shortly after pouring while the grout still has low strength. The measured contraction of the grout would amount to less than 0.25 in. over a 30-ft depth of grout. The contraction also appears to level out after 7 weeks of curing so that placement of the cold cap several months after completing the last lift in the vault should compensate for any contraction.

5.3 THERMAL CONDUCTIVITY

Interference between the heater cable and the thermocouple wire prevented in situ thermal conductivity measurements. When this was discovered, a sample of grout produced with the pilot-scale equipment was cast around a second thermal conductivity probe. The sample was approximately 8 in. long x 4.5 in. in diameter. The measured thermal conductivity of this sample was 0.81 W/m°K. This value was used for all adiabatic temperature calculations.

Without the in situ probe, changes in thermal conductivity as a function of temperature and stage of curing were not determined. However, thermal conductivity measurements in past pilot-scale tests using a different waste and dry-blend showed that the thermal conductivity was very constant and did not change with stage of curing, temperature, or position (Lokken 1992).

5.4 SEPARATED LIQUIDS

The models generated from the formulation studies conducted by ORNL predicted that the grout formulation used in the pilot-scale run would have a small amount (0.7 vol%) of drainable liquids. Observations of the gradient mold shortly after pouring and 24 hours after pouring showed no drainable liquids. There are several possible reasons for this difference. One reason may be the differences in the dry-blend mixing method. The dry-blend for the experimental tests was mixed for 23 hours in a V-blender while the DMF uses a 5-minute blending time. The ability of attapulgite clay to control drainable liquids is lower for longer blend times (Lokken 1987).

Another possible reason for the lower observed drainable liquids is the difference in the initial curing condition of the laboratory samples and the grout in the gradient mold. The laboratory samples are held at 40°C for the first 24 hours after pouring. This low temperature reduces the hydration reaction rates and allows more settling and drainable liquid formation before the grout gels. The temperature of the grout in the gradient mold increased rapidly and gelation of the grout may have occurred more quickly and prevented settling. The shear thickening of the grout produced with the pilot-scale test may have also reduced the amount of settling.

A fourth reason could be the higher average mix ratio in the pilot-scale grout (see Section 4.1.5). Regardless of the reasons, the laboratory tests were conservative and predicted more drainable liquid formation than was observed in the pilot-scale run.

5.5 GROUT SLURRY DENSITIES

The grout slurry densities determined from samples taken using the pilot-scale grout runs are shown in Table 5.1. These data show that the grout densities were generally quite consistent but that pouring grout through the drop chute and/or the sampling process at the gradient mold resulted in slightly lower slurry densities. Table 5.4 shows the average grout slurry densities for different groupings of samples. The first grouping is all the samples taken from the surge tank while processing grout with a mix ratio of 8.7 lb/gal. The second grouping contains the samples that were taken from the lift mold (i.e. did not fall through the drop chute) while processing a grout with a mix ratio of 8.7 lb/gal. The average densities of these two groupings are very close with small standard deviations. The third group is all the samples that were taken after the grout fell through the drop chute into the gradient mold while processing a grout with a mix ratio of 8.7 lb/gal. Densities of these samples have a small standard deviation but are about 2% lower than the densities of the first two groupings. Apparently, the fall through the drop chute and/or sampling the falling stream entrained air in the slurry and reduced the slurry density.

The last grouping of samples in Table 5.4 shows the densities of samples taken while pouring the fourth lift. These samples are separated since the

TABLE 5.4. Grout Slurry Densities

Sample Grouping	Formulation/ Mix Ratio (lb/gal)	Average Density (lb/gal)	Number of Samples	Standard Deviation
All Surge Tank Samples	Baseline/8.7	12.963	10	0.073
Discharge Samples Taken While Pumping to Lift Mold	Baseline/8.7	12.966	5	0.071
Discharge Samples Taken While Pumping to Gradient Mold (through drop chute)	Baseline/8.7	12.716	5	0.070
All Samples Taken While Pouring Lift 4	Adjusted/8.4	12.985	8	0.057

fourth lift used a modified dry-blend composition and a different mix ratio. The average density of this group of samples had a small standard deviation and an average value that was essentially equal to the grouts made with the original dry-blend formulation.

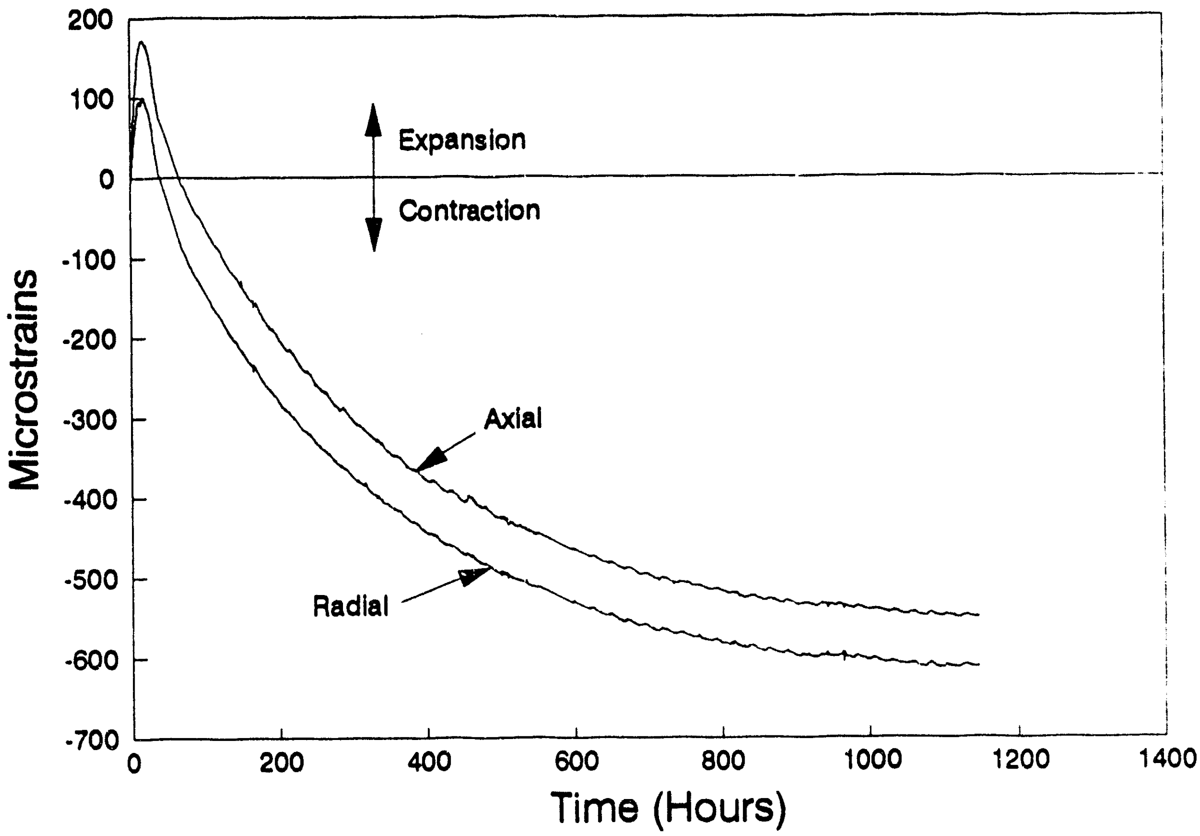


FIGURE 5.1. Grout Dimensional Changes

6.0 THERMAL INFORMATION

The normal frequency used to collect the thermal data for both the gradient mold and the lift mold ranged from once every 10 minutes to once every 60 minutes. However, some data were lost due to problems that occurred with the data collection system. The first problem resulted when a storage disk with data from a previous instrumentation shakedown test was used for the pilot-scale run. These data caused the disk to reach capacity faster than anticipated. Data from hours 43 through 73 were lost due to this error in the first week. When this problem was discovered, the data disk-handling procedure was changed to include a reformatting step for all storage disks just prior to use. A second problem was discovered when an electrostatic precipitator in the general vicinity of the data logger was tested during an off-shift. The discharge from the precipitator interfered with the data collection and in one case caused the program to crash. Once this problem was discovered, data collection was suspended while electrostatic precipitation tests were conducted. A third problem occurred when maintenance personnel turned off the power to the data logger while trying to de-energize a different circuit. This caused the program to crash and 46 hours of information were lost during the week following the fourth lift (hours 554 through 600). The building manager was notified to prevent reoccurrence of this problem.

The amount of data lost compared to the total collected was relatively small. In graphs and data analysis, the gaps in the data are approximated with a straight line.

Thermal information was obtained from both the gradient and the lift molds. The thermal information from the gradient mold is discussed in Sections 6.1 through 6.2. This information was used to determine the different curing conditions of the core samples taken from the mold and to calculate an adiabatic temperature rise.

The thermal information from the lift mold is discussed in Sections 6.3 through 6.6. Section 6.3 discusses the convective/evaporative cooling of the grout in the lift mold and compares the results to those obtained in tests conducted prior to grout production. Section 6.4 looks at the short-term temperature profiles as the different lifts are added. Section 6.5 discusses the ability of this lift scenario to control the early heat release from the grout, while Section 6.6 examines the short-term data and estimates the long-term temperatures in the lift mold.

6.1 GRADIENT MOLD THERMAL PROFILES

Figures 6.1 through 6.4 show temperature profiles of vertical cross sections of the grout in the gradient mold. These profiles were taken at the peak temperature and after 1, 2, and 3 weeks of curing. The peak temperature was 86°C and occurred 74 hours after completing the pour. By the end of the third week, the center of the mold had cooled to temperatures below 55°C.

The main objective of the gradient mold was to have different portions of the grout cure under significantly different curing profiles. The curing temperature of grout 3 in. from the edge of the mold was maintained below 55°C, while the grout at the center of the gradient mold reached temperatures as high as 86°C. Analyses of core samples taken at different radial distances from the mold wall will help determine the effects of different curing profiles on the final properties of the grout. The analyses of the core samples will be discussed in a later report.

6.2 ADIABATIC CALCULATIONS

Thermal information from the center of the gradient mold was used to calculate an adiabatic temperature rise. This calculation uses the thermal conductivity and measured thermal gradients in the grout to determine the amount of heat lost from a specific volume of grout. The heat loss above that necessary to account for the observed temperature drop is assumed to be hydration heat. This heat is added back to the grout to calculate the adiabatic temperature rise. Equations used to calculate the adiabatic temperature rise are shown in the Appendix.

Figure 6.5 shows the observed and calculated adiabatic temperature as a function of time. The calculated adiabatic temperature rise after 7 weeks of curing (assuming a starting temperature of 40°C) is 57°C, and the maximum calculated adiabatic temperature is 97°C.

Assuming the specific heat of the solids in the grout is 0.2 cal/g°C, the mass-weighted, average specific heat of the grout (solids and water) is 0.527 cal/g°C. Using this value as the specific heat and a grout density of 12.72 lb/gal, the calculated heat release from the grout after 7 weeks is 5100 Btu/cubic ft. The total amount of heat generated in this formulation over the first 7 weeks is significantly less than the heat generated from the grout formulation used in the previous DSSF pilot-scale test (Lokken 1992). The measured adiabatic temperature rise shows that this grout formulation will require pouring in lifts and forced convective/evaporative cooling to maintain temperatures below 90°C. However, the total amount of heat that will have to be removed to control the grout temperatures is less than that required for the previous DSSF formulation.

6.3 CONVECTIVE/EVAPORATIVE COOLING

The current philosophy for dealing with the grout hydration heat is to pour the grout in lifts and use increased airflows in the grout vault to increase the rate of heat removal. Current plans call for installation of equipment that will provide 3600 CFM of airflow.

The main goals of the lift mold were to measure how much heat the planned convective/evaporative cooling could remove from the grout surface, and measure the effects of this cooling on the temperatures in the grout. However, it is also important to know how changes in the airflow might affect the heat removed from the grout. Therefore, two sets of heat removal tests were conducted with the lift mold. The first set of tests studied the heat removal for different airflows and liquid surface conditions. In this case, the surface of the grout was simulated by a liquid layer placed in the bottom of the lift mold. These tests are referred to as the convective/evaporative cooling tests.

For the convective/evaporative cooling tests, a target airflow rate of 13 standard cubic feet per minute (scfm) was determined by maintaining the airflow/cooling surface area ratio the same for the lift mold as is planned for the production vault. In other words, a 13 scfm airflow in the pilot-scale tests will supply the same volumetric airflow per unit area of cooling surface as a 3600 scfm airflow in the production system. Higher (24 and 36 scfm) and lower (6 scfm) flow rates were investigated to determine the effects on the heat removal rate. Because the proposed airflows in the production vault are more familiar to most readers, the airflow numbers throughout the report are given as "modeled production airflow (actual pilot-scale airflows)." For example, if an airflow of 13 scfm was tested, the report will list the airflow as 3600 (13) scfm.

The anticipated heat removal rates from the grout can also be calculated by taking the heat removed per unit area of cooling surface in the lift mold and multiplying by the area of cooling surface in the production vault. Again, since the anticipated heat removal rates from the production vault are more familiar to most readers, the heat removal values throughout the report that are calculated from lift mold data will be given as "anticipated" heat removal rate (actual pilot-scale heat removal rate). For example, a heat removal rate of 720 Btu/hr in the pilot-scale lift mold will be reported as 200,000 (720) Btu/hr.

The second set of tests measured the heat removed from the grout surface as the successive lifts were poured into the mold. An airflow of 13 scfm was used for all the grout surface heat removal tests.

6.3.1 Description of Convective/Evaporative Cooling Tests

The experimental setup for these tests are schematically shown in Figure 6.6. An immersion heater was placed in the liquid at the bottom of the mold. At the start of each test, the liquid in the lift mold was heated to a temperature of 48° to 50°C. At this point, the power to the heater was turned off and the butterfly valve was set to obtain the desired airflow rate through the lift mold. The inlet and outlet air conditions, the liquid temperature, and the airflow rate were recorded every 5 minutes as the liquid cooled through the temperature range of 30° to 45°C. These parameters were used to calculate a heat removal rate as a function of liquid temperature. These tests were conducted in the 324 Building highbay to reduce fluxuations in inlet air conditions. However, inlet air conditions still varied between 17° to 27°C and 18% to 32% RH.

Heat removal from water and from a 3.2-M solution of sodium nitrate was investigated. This second solution had a vapor pressure depression of approximately 10%. The vapor pressure depression of this $NaNO_3$ solution approximates the vapor pressure depression measured over a solution of simulated 106 AN waste.

6.3.2 Results of Convective/Evaporative Cooling Tests

Figures 6.7 and 6.8 show that the heat removal was a strong function of liquid temperature with 2 to 3 times more heat removal at 45°C compared to 30°C. The rate of airflow also affected heat removal rates. Increasing the airflow from 1660 (6) scfm to 3600 (13) scfm significantly increased the heat removal rates. The outlet air conditions in the 1660 (6) scfm case were saturated (100% RH for the water, 96.5% RH for the salt solution) for all liquid temperatures tested. This indicates that the potential for evaporation of the water or salt solution was greater than the removal rate of a 1660 (6) scfm airflow. Increasing the airflow to 3600 (13) scfm removed sufficient quantities of water to bring the outlet conditions below saturation for liquid temperatures below 39°C. Thus, at 3600 (13) scfm the evaporation rate from the surface became the limiting process, and cooling was not proportional to airflow.

Increasing the airflows to 6650 (24) and 9970 (36) scfm increased the heat removal at higher liquid temperatures, where the outlet conditions were still saturated in the 3600 (13) scfm case. However, these higher airflows had less effect at the lower liquid temperatures where evaporation was the rate limiting step. An adequate airflow in the production vault is therefore, dependent on the expected surface temperatures. If high surface temperatures are expected, higher airflows in the production vault would be worth pursuing. However, if low surface temperatures are expected, the current plans for using 3600 CFM are adequate.

Figure 6.9 shows that heat removal rates from a $NaNO_3$ solution are lower than the heat removal rates from the water. The crossing of the curves at higher temperatures is thought to be due to experimental startup errors as the system came to a steady state. The lower heat removal rates are probably due to the reduced water vapor pressure at the liquid/air interface. Reduced heat removal rates for the salt solution were found with all four airflows tested.

The relative amounts of heat that were removed due to sensible heat and evaporation were also determined. Over the entire temperature range investigated, 82% to 96% of the heat loss was the due to evaporation. Thus, evaporation water must be available from the free liquid, rinse water, and/or grout pour solution for effective cooling.

6.3.3 Determining Adequate Airflow Rates

Heat removal from the grout can be viewed as a two-step process. First, the heat conducts through the grout to the surface. The heat is then removed by the airflow. If the airflow removes enough heat to make the conduction through the grout, the slower, rate-limiting step, then the flow would be considered adequate. Therefore, it is important to relate these two steps to determine which is likely to limit the heat removal and determine the length of the pour schedule.

The convective/evaporative cooling tests show that the amount of heat removed from a liquid surface increases significantly with increasing liquid temperature. On the other hand, the rate of heat transfer through the grout is controlled by the thermal gradient established between the interior and surface of the grout. As the surface temperature increases, the gradient becomes smaller, and the amount of heat transferred to the surface is reduced. Therefore, the temperature will come to a pseudo-steady state at a point where the heat removed from the grout through conduction is equal to the convective/evaporative heat removed from the liquid at the grout surface. Figure 6.10 graphically shows the relationship between surface cooling and heat conduction through the grout. The surface cooling data shown in this figure are for airflows of 1660 (6), 3600 (13), and 9970 (36) scfm over a solution of $NaNO_3$. The heat conduction rates assume that the grout 1 ft below the surface is at a given temperature and the thermal gradient from that point to the surface is linear. A thermal conductivity of 0.81 W/m°K is assumed for the grout. The pseudo-steady state surface temperatures are where the conduction and convective/evaporative surface cooling curves intersect.

To determine which of the two steps in the heat removal is rate limiting, first examine a case where the airflow is kept constant [3600 (13) scfm] and the grout temperature 1 ft below the surface is varied. If grout 1 ft below the surface is at a temperature of 90°C, Figure 6.10 shows that the surface temperature is about 35°C and the heat removal is about 350,000 Btu/hr. When grout 1 ft below the surface cools to 70°C, the surface temperature falls to 31°C and the heat removal rate drops to about 230,000 Btu/hr. If the grout 1 ft below the surface cools to 50°C, the surface temperature falls to 27°C and the heat removal rate falls to about 110,000 Btu/hr. Thus, the temperature of the grout near the surface has a strong effect on the heat removal rate.

Next consider the case where the temperature of the grout 1 ft below the surface is held at a constant 70°C while the airflow is varied. At an airflow of 3600 (13) scfm, the surface temperature is 31°C and the heat removal rate is about 230,000 Btu/hr. Increasing the airflow 2.75 times to 9970 (36) scfm reduces the surface temperature to 27°C but only increases the heat removal slightly to 250,000 Btu/hr. Reducing the airflow to 1660 (6) scfm increases the surface temperature to 36°C but only reduces the heat removal slightly to about 210,000 Btu/hr. Comparing the two cases shows that adjusting the airflow either up or down does not significantly impact the heat removal rates, while changing the gradient in the grout near the surface has a significant impact. This suggests that the rate limiting step is the conduction of heat to the grout surface. Increasing the planned airflow rates will not remove significantly more heat or shorten the pour schedules.

Figure 6.10 also shows that the pseudo-steady state surface temperatures for the 3600 (13) scfm case are generally quite low. Even if the grout 1 ft below the surface reached a temperature of 90°C, the surface temperature is only 36°C. Since the pseudo-steady state surface temperatures are low, the evaporation rate is the rate-controlling step and higher airflows are not required to remove the evaporated water (i.e. the outlet air conditions will not be saturated).

This analysis indicates that the planned airflow is adequate and that increasing the blower size will not significantly shorten the pour schedule. However, this analysis treats the conduction of heat through the grout in a very simple manner. Current WHC modeling efforts should include test cases that vary the airflows to confirm the conclusions of this analysis.

6.3.4 Cooling of Grout Surfaces

Airflow data were collected for all four lifts, but the analyses generally concentrate on the second and third lifts. The second lift is more representative of the majority of the lifts that will be poured into the vault, since it is poured on top of the cooled first lift and is covered by the third lift 1 week later. The environment for the third lift is similar to the second but did not have the rinse water placed on the surface. Therefore, comparisons between the second and third lift should be the most accurate.

The instantaneous heat removal rates calculated from the changes in the inlet and outlet air conditions are shown for the second lift in Figure 6.11. The highest rate measured was just over 200,000 (720) Btu/hr and was obtained shortly after the completion of the pour. Figure 6.11 also shows the average temperature obtained from three thermocouples placed at the liquid layer/grout surface interface. The 200,000 (720) Btu/hr heat removal obtained for a liquid temperature of 29°C is in agreement with the heat removal rates determined in the airflow tests. The high initial heat removal is due to the early hydration of the grout near the surface and due to the fact that the grout enters the mold at an elevated temperature. A steep thermal gradient is initially established which rapidly transfers heat to the surface. As grout near the surface cools, a less steep gradient is established, and the heat removal rate decreases.

Past the initial heat release, the data show two trends. The first trend is the 24-hour cycling period that corresponds to the day/night temperature cycles. Figure 6.12 shows an expanded 24-hour temperature cycle that also includes the inlet and outlet temperatures. As the inlet temperature increases and peaks, the heat removal drops off and reaches a low. The warmer inlet air and the reduced heat loss from the grout cause the liquid temperature to increase slightly. As the inlet temperature drops, the heat removal rate from the warmer liquid goes up significantly. The increased heat loss from the grout and the cooler inlet air reduce the surface temperature, and the heat removal rate peaks and falls off. The 24-hour pattern repeats when the inlet air temperature increases.

The second trend is the gradual reduction in the heat removal rate over the week. This gradual reduction is due to the reduced steepness of the thermal gradient in the grout as the grout heat production rate slows and the temperatures within the grout are reduced.

The absence of a liquid layer on the third lift did not affect the heat removal rates (see Figure 6.13). However, the surface temperature of the third lift had greater temperature fluctuations than the liquid temperature of the second lift. The trend for the average surface temperature to increase over the week for the third lift was due to a similar trend in the inlet air temperatures.

The expanded 24-hour period of the surface, inlet, and outlet temperatures for the third lift (see Figure 6.14) are similar to that for the second lift. The heat removal rate peaks when the surface temperature peaks. However, the grout surface temperature follows the inlet air temperature more closely than the second lift liquid temperature. This probably explains the greater surface temperature fluctuations seen in the third lift.

The total amount of heat removed and the total quantity of water evaporated for the 1-week cooling period for each lift was calculated from the changes in the inlet and outlet air conditions. These values are shown in Table 6.1. The total amount of water evaporated in the third lift was less than the amount of water evaporated in the second lift, while the heat removed from the third lift was greater. This is the only indication that the grout surface was drier for the third lift than for the other lifts.

The main conclusions that can be drawn from the grout heat removal tests is that an airflow of 3600 (13) scfm is sufficient to cool the grout surface with or without a layer of cooling water. Although there were some indications that the grout surface in the third lift was drying out, this drying did not significantly affect the ability to remove heat with the airflow.

TABLE 6.1. Total Heat and Water Removed With Convective/Evaporative Cooling

Lift Number	Total Heat Removed (Btu ± 6%)[a]	Total Water Evaporated (gal ± 6%)[a]	Lift Temperature Reduction (°C)
1	79,900	8.5	19.3
2	70,800	7.9	17.2
3	77,600	7.0	18.8
4	99,200	9.9	24.0

(a) Calculated from changes in the inlet and outlet air conditions.

6.3.5 Cooling Water/Grout Interactions

One concept for maximizing the cooling effect of increased ventilation in the grout vault was to add additional water to the top of each lift to assure that sufficient water was present for evaporative cooling. In these tests, approximately 1.5 in. of rinse water (20 gal) were added to the first, second, and fourth lifts. This quantity of water was added to assure that the majority of the surface was covered with water during the week between lifts. No rinse water was added to the top of the third lift. The effects of this layer on grout cooling are discussed in Section 6.3.4. However, additional effects were noted that are believed to be due to placing rinse water on top of the uncured grout.

At the completion of the first lift, the layer of liquid at the grout surface was approximately 1.5 in. deep. Just prior to pouring the second lift, half of the grout surface was exposed, while the other half was covered by a thin (approximately 1/4 in.) layer of water. The estimated water remaining on the surface was 1 to 2 gal. The exposed portion had a muddy appearance and may have been poorly solidified.

The water layer, after completing the second lift and adding the rinse water, was again about 1.5 in. Figure 6.15 shows the surface of the second lift after 7 days. The majority of the surface was still covered with a thin (approximately 1/4") layer of liquid. The estimated water remaining on the surface was 3 to 5 gal. The surface has the same appearance as the first lift.

As the third lift was poured, the liquid remaining on the grout surface was pushed to the side of the mold, away from the inlet, and formed a pool in one end of the mold. This 3 to 5 gal of liquid was removed after the completion of the run. Figure 6.16 shows that the surface of the third lift after 7 days is dry and is covered by fine white salt crystals. The grout did not have the muddy surface appearance present in the first two lifts.

The liquid layer, after adding the rinse water at the completion of the fourth lift, was about 2 in. deep. After 24 hours, the liquid at the surface was gone. A small leak in the mold outer liner indicated that this water was lost due to a leak in the inner liner as opposed to evaporation or adsorption by the grout. It was not possible to estimate the quantity of material that leaked through the inner liner by the small quantity (<50 ml) of material that leaked through the outer liner. After 1 week, the top 1 to 2 in. of grout had not solidified. The grout surface after 1 week is shown in Figure 6.17.

As noted above, there appeared to be a poorly solidified layer on the grout surface for those lifts that 1.5 in. of rinse water was placed on top of the freshly poured grout. The surface of the third lift, where the liquid was removed, and the surfaces of the gradient mold and all the 55-gal drums, where no rinse water was added, did not have this layer. The addition of rinse water cools the grout and dilutes the waste near the surface. This might allow increased settling and segregation of the dry-blend components leaving the less dense attapulgite clay and the finer fly ash particles at the surface. The condition of these layers after covering with subsequent lifts is not known. If these layers do not solidify after being covered by the subsequent lifts, they would be a concern.

Flooding the lift surface with 1.5 in. of water was based on two assumptions: 1) a layer of water on the grout surface was required for cooling, and 2) flooding the surface with water would not effect the grout curing. However, the heat removal information presented in Section 6.3.4 indicated that the layer of water was not necessary for effective cooling. The poorly solidified layer on lift 4 indicates that at least the short-term curing of the grout was negatively affected by flooding the surface. Based on these results, the grout surface should not be flooded with 1 to 2 in. of water (4000 to 8000 gal in the production vault) immediately after completion of the pour. Only the water necessary to rinse the grout pipe should be placed on top of the lift. This water, along with the water from the grout pore solution, should be used to supply the evaporation water necessary for cooling. Analyses of core samples taken from the lift mold will concentrate on the lift interface regions and will be discussed in later reports.

Since the pore solution water may become the main source for the evaporation water, the amount of water necessary to cool the grout was calculated and compared to the amount of water that might be available from the grout pore solution. The amount of water required to cool the grout was calculated assuming that 80% of the heat removal was due to evaporation and that the grout would have to be cooled by 20°C. The other 20% of the heat removal is assumed to be due to heating the inlet air. Using these assumptions, 0.016 g of water are required to cool each gram of grout. Considering that the grout is composed of 41.3 wt% water, about 4% of the total water would have to evaporate to cool the grout. This indicates that there may be sufficient water in the grout to supply the evaporation water. However, as water evaporates, the pore solution will become more concentrated. As a result, the vapor pressure depression at the surface may reduce the cooling efficiency of the increased airflow. If this is the case, additional evaporation water could be added after the grout has cured for several days. A better understanding of how the vapor pressure at the grout surface changes as water evaporates would help determine if additional evaporation water was necessary.

6.4 SHORT-TERM TEMPERATURE PROFILES IN LIFT MOLD

The short term temperature profiles observed in the lift mold are shown in Figures 6.18 through 6.25. These profiles show the side view centerline (i.e. 1.5 ft from the front and back inner surfaces of the mold) temperatures of the grout. In the following discussions, the center of a lift refers to the area 1 ft below the surface of the grout block at the completion of the lift. Two profiles were taken for each of the four lifts placed in the mold. One profile was taken when the center of each new lift was at its peak temperature (about 33 hours after completion of the lift pour), and the second profile was taken 1 week after completion of the lift pour.

The peak thermal profile in the first lift (Figure 6.18) shows that the heat loss from the grout is primarily through the top surface with some heat loss through the bottom surface. The heat loss through the side surfaces for this short time period are relatively small. The warmest areas are just below the center of the lift. After 1 week of cooling (Figure 6.19), the warmest temperatures in the mold are 40° to 44°C and below the center of the lift.

The peak thermal profile in the second lift (Figure 6.20) shows that the warmest area after pouring the second lift is near the center of the second lift and shifted slightly towards the cooling air outlet side of the mold. Heat from the second lift is conducted into the cooled first lift and lost to the cooling air at the surface. After cooling for 1 week, the different lifts can no longer be distinguished from the thermal profiles (Figure 6.21). The warmest area (44° to 48°C) is centered about 2 ft below the grout surface and is slightly warmer than the first lift after a week of cooling.

The peak thermal profile in the third lift (Figure 6.22) shows that the warmest area in the third lifts shifts down slightly and is a little below the center of the third lift. The warmest area is again shifted slightly towards the cooling air outlet side of the mold. After 1 week of cooling (Figure 6.23), the different lifts can no longer be distinguished from the thermal profiles. The warmest area is centered about 2 ft below the surface and has again increased in temperature (relative to the second lift) to 44° to 52°C.

The peak thermal profiles for the fourth lift (Figure 6.24) show the warmest areas again shift down and are below the center of the lift. The warmest area is not shifted toward the cooling air outlet side of the mold. After 1 week of cooling (Figure 6.25), the thermal profiles show that all four lifts are acting like a single monolith. The warmest area has shifted down slightly relative to the third lift and is centered about 2.5 ft below the surface. The warmest temperatures in the grout have risen to 52° to 56°C.

Examining the general trends for all four lifts indicates that there is a temperature buildup occurring as the successive lifts are added. The first lift is effectively cooled during the first week because heat is lost through the mold bottom. When the second lift is poured, some of the initial hydration heat from the second lift goes into reheating the first lift. As a result, the peak temperatures in the second lift are low. However, the grout in the first lift also acts as insulation and reduces the amount of heat from the second lift that is lost through the bottom of the mold. Therefore, once the first lift is reheated, most of the heat that is lost from the second lift is lost through the surface. Lower overall heat loss leads to warmer temperatures in the grout when the third lift is poured. As a result, heat loss into the warmer cured grout of the second lift is not as great when the third lift is poured. This increases the maximum peak temperature of the third lift and shifts the warmest area down below the lift center since the proportion of heat lost through the surface is now greater. This pattern continues for the fourth lift.

The buildup of temperature seen in the first four lifts, however, is probably self limiting. As the grout temperatures get warmer, the thermal gradient in the grout conducts more heat to the surface where it can be removed by the airflow. The total amount of heat removed for lifts 2 through 4 (see Table 6.1) shows the beginning of this trend.

Changes in the plenum height as the mold is filled (e.g. reduced residence time) and/or changing the surface conditions of the grout (e.g. removing the cooling-water layer in the third lift) could contribute to the heat buildup if they reduced the cooling effect of the forced airflow. However, Table 6.1 shows that the total heat removed from the upper lifts increases. Thus, heat buildup in the grout as opposed to decreased cooling efficiency is the better explanation for the observed behavior.

Shifting of the warmest areas to the cooling air outlet side of the mold is probably due to enhanced heat loss on the cooling air inlet side (i.e. cooler, drier inlet air is more effective in cooling the grout surface).

6.5 SHORT-TERM PEAK TEMPERATURES IN LIFT MOLD

One of the main concerns when trying to control the temperature of the grout by pouring in lifts is that the initial hydration heat is released so rapidly that the surface cooling does not adequately control the maximum temperatures. There is currently insufficient information for this grout formulation to know if short-duration, high-temperature peaks early in the curing cycle will adversely affect the final grout properties. Table 6.2 shows the slurry-pouring temperature, the highest mold temperatures recorded after the 1-week cooling periods, and the maximum lift temperatures. The maximum lift temperatures ranged from a low of 58°C in the second lift to a high of 69°C in the fourth lift. This variation was explained in Section 6.4 as a gradual buildup of heat as the successive lifts are poured. However, there is also a general trend that relates maximum lift temperatures to the pour temperature. Higher maximum temperatures would be expected for higher pouring temperatures since the starting point for the temperature increase is higher and also because the early hydration reaction kinetics are higher. The maximum short-term temperatures obtained for each lift are probably a combination of both effects.

TABLE 6.2. Observed Lift Temperature Variations

Lift Number	Pouring Temperature (°C)	Range of Maximum Mold Temperatures After 1-Week Cooling Period (°C)	Maximum Lift Temperature (°C)
1	38-43	44-44	66
2	37-39	44-48	58
3	39-40	48-52	62
4	41-43	52-56	69

6.6 ESTIMATION OF LONG-TERM TEMPERATURES IN LIFT MOLD

To look at the long-term temperature profiles of grout poured in lifts, a calculation of the adiabatic temperature of grout near the center of the second lift was conducted. This calculation was similar to the one conducted for the gradient mold (see Appendix) but used a rectangular box instead of a cylinder for the heat balance calculation. The rectangular box was 22.5 in. wide x 9 in. deep x 4 in. thick and had its centroid located at the lift centroid. The surfaces of this box were grouped into the side surfaces and the top and bottom surfaces. For the adiabatic calculation, heat loss from all surfaces was added back to the grout. A second "Calculated Lift Temperature" was determined by adding the heat lost through the sides to the observed temperatures, since these losses would not be present in the center of the vault (e.g. conduction losses through the mold insulation). The heat lost/gained through the top and bottom surfaces were assumed to be "real" losses/gains. This analysis was conducted for both the second and third lifts.

The calculated adiabatic temperature rise for the gradient mold, second lift, and third lift are shown in Table 6.3. There is excellent agreement between the calculated numbers from the gradient mold and the second lift. The calculated numbers from the third lift are 8° to 10°C higher but are still in reasonable agreement with the other calculated numbers.

TABLE 6.3. Comparison of Calculated Adiabatic Temperatures

Adiabatic Calculation	Average Pour Temperature (°C)	Curing Time (Hr)	Calculated Adiabatic Temperature (°C)	Calculated Temperature Rise (°C)
Gradient Mold	40	1150	97	57
Gradient Mold	40	978	96	56
Gradient Mold	40	813	96	56
Lift 2	38	978	97	59
Lift 3	39.5	813	107	67.5

The calculated adiabatic temperature, calculated lift temperature, and actual lift temperature for the second lift are shown in Figure 6.26. The calculated lift temperature shows that the surface cooling and the heat loss into the cooled first lift effectively control the early temperatures in the second lift. However, once the second lift is covered with the third lift, heat from the third lift increases the temperature in the second lift. After the fourth lift is poured, the adiabatic temperature and the calculated lift temperature curves are essentially parallel. The shape of the calculated lift temperature curve indicates that cooling the surface does not significantly affect grout located 5 ft below the surface. Thus, these curves indicate that essentially all the benefits derived from cooling the surface of the lift are obtained in the first week. However, the net gain cannot be assessed until the end of the second week when heat from the lift above has been included. Beyond the second week, the grout is covered by enough new grout that cooling of the surface will have little effect. The net reduction from the adiabatic curing temperature due to surface cooling of the second lift after 2 weeks was on the order of 30°C. Table 6.1 shows that the total heat removed during the week the second lift is cooled is 70,800 Btu, which would account for a 17.2°C temperature reduction. Since the grout at the edges of the mold is cooler than the center portion of the grout, the total loss from the surface will be less than that lost from the middle. Thus, the total heat removed from the grout is in reasonable agreement with the above calculations that indicate a net 30°C temperature reduction.

A similar analysis was conducted on the grout temperatures obtained at the center of the third lift. Figure 6.27 shows that the same general patterns are obtained. The net reduction from the adiabatic curing temperature due to surface cooling of the third lift after 2 weeks was also on the order of 30°C. One notable difference is the widening of the calculated lift temperature and adiabatic curves. This widening indicates that the surface cooling is removing heat from the third lift. However, only four pours were conducted and the center of the third lift was never more than 3 ft from the surface. If the lift sequence had continued beyond the fourth lift, there probably would have been little additional heat removal from this portion of the grout.

Assuming that the net temperature reduction derived from cooling a 2-ft lift for 1 week is on the order of 30°C, and a conservative adiabatic temperature rise of the grout is on the order of 70°C, the total temperature rise of grout in the vault will be on the order of 40°C. If the grout is poured at 40°C, the final grout temperature predicted from the pilot-scale information should be on the order of 80°C. This indicates that the heat removed with the airflow during a 1-week cooling period should prevent grout temperatures from exceeding 90°C in the production vault.

This analysis of the long-term, lift mold temperatures indicates that the amount of heat removed by convective/evaporative cooling from a 2-ft lift during a 1-week cooling period should prevent grout temperatures from exceeding 90°C. However, this may not be the optimum schedule and modeling of the full-scale vault is recommended to determine the best pour schedule. This modeling should include 1) the actual lift scenario, which includes a 24-hour pouring period, 2) heat release from the grout as a function of temperature and extent of reaction, 3) a temperature-dependent heat-removal rate from the surface, and 4) the slower heat removal mechanisms present in the vault.

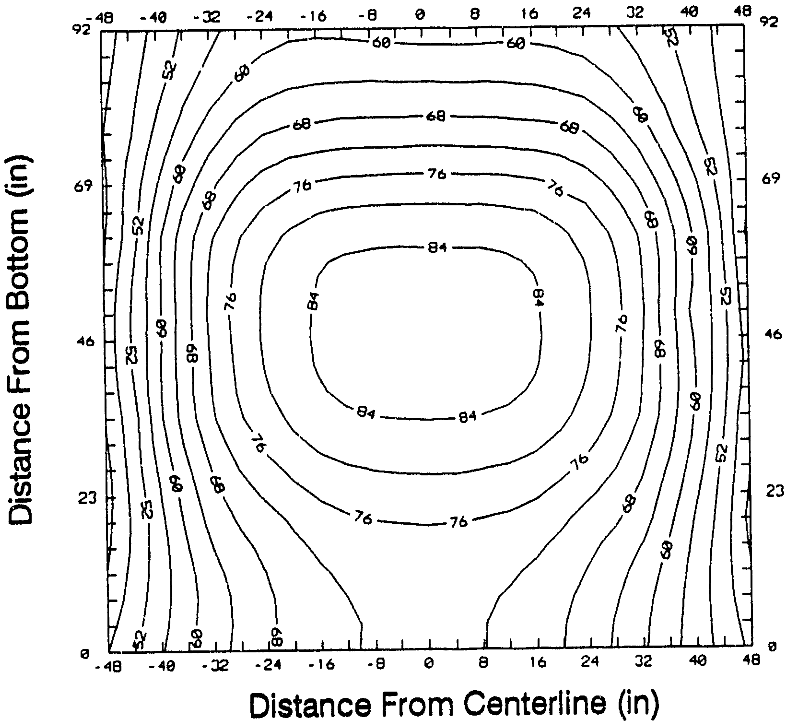


FIGURE 6.1. Thermal Profile of Grout in Gradient Mold After 74 Hours (peak center temperature)

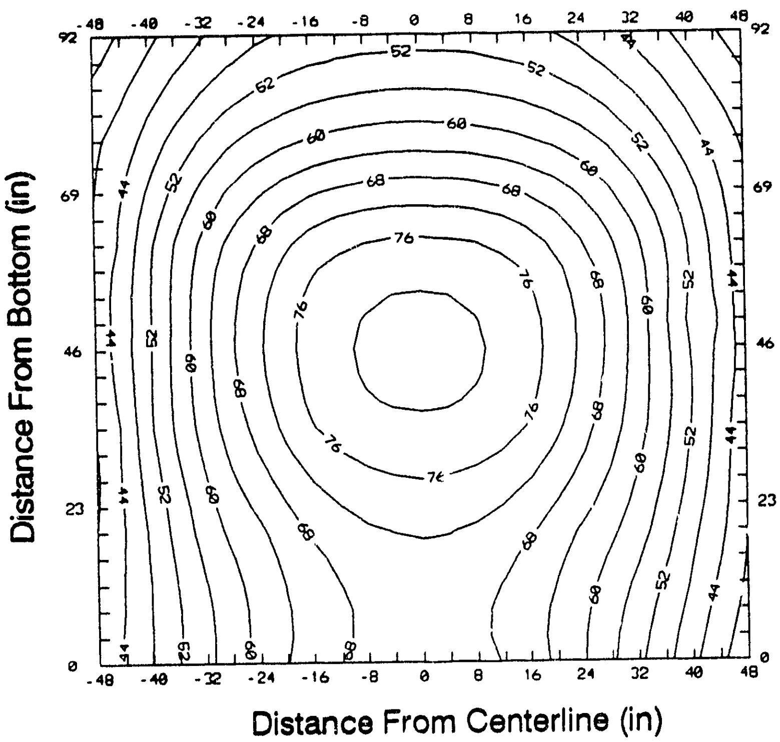


FIGURE 6.2. Thermal Profile of Grout in Gradient Mold After 1 Week

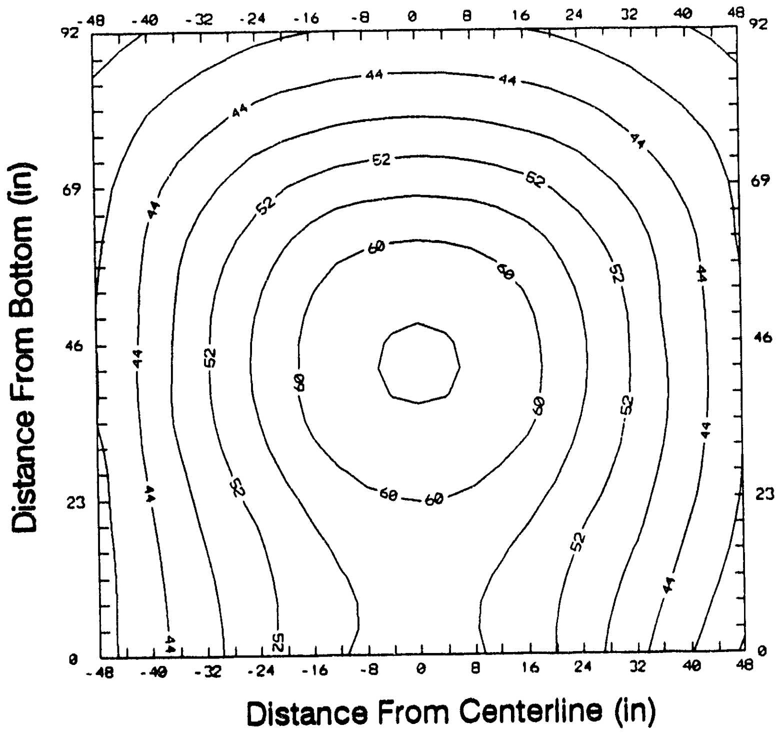


FIGURE 6.3. Thermal Profile of Grout in Gradient Mold After 2 Weeks

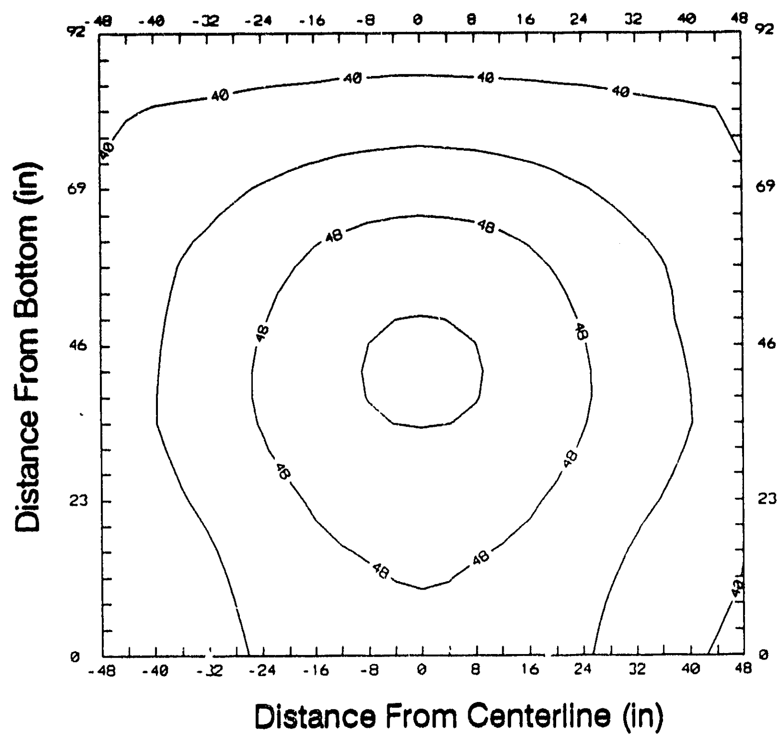


FIGURE 6.4. Thermal Profile of Grout in Gradient Mold After 3 Weeks

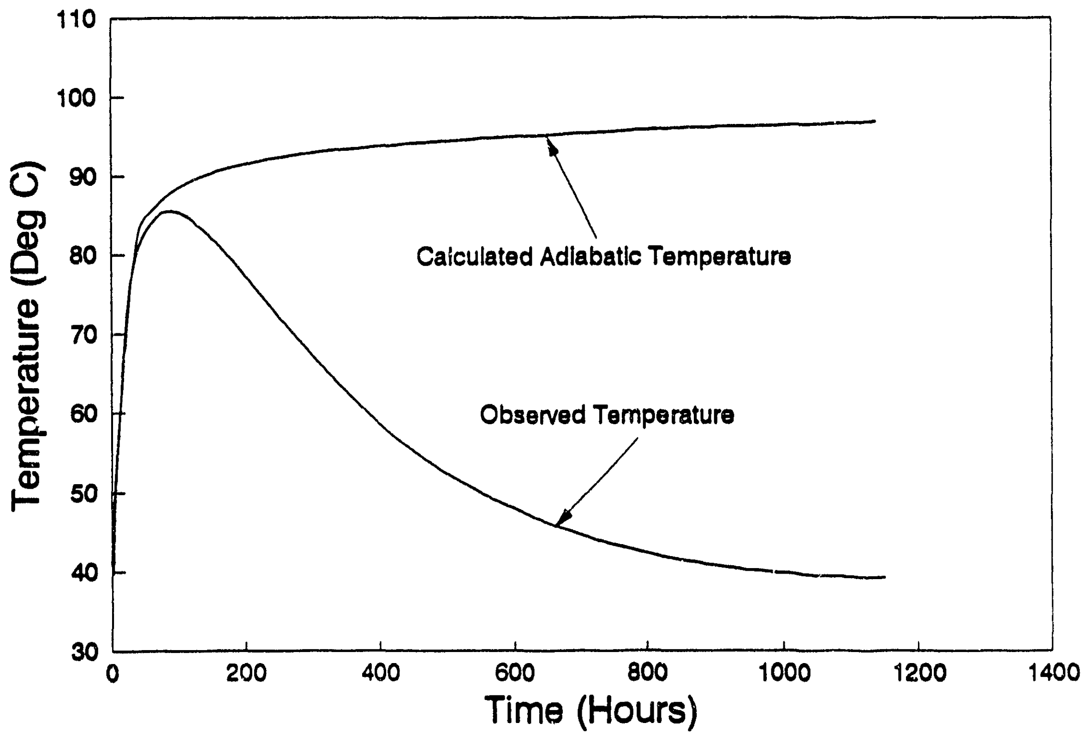


FIGURE 6.5. Calculated Adiabatic Temperature and Observed Temperature in Gradient Mold

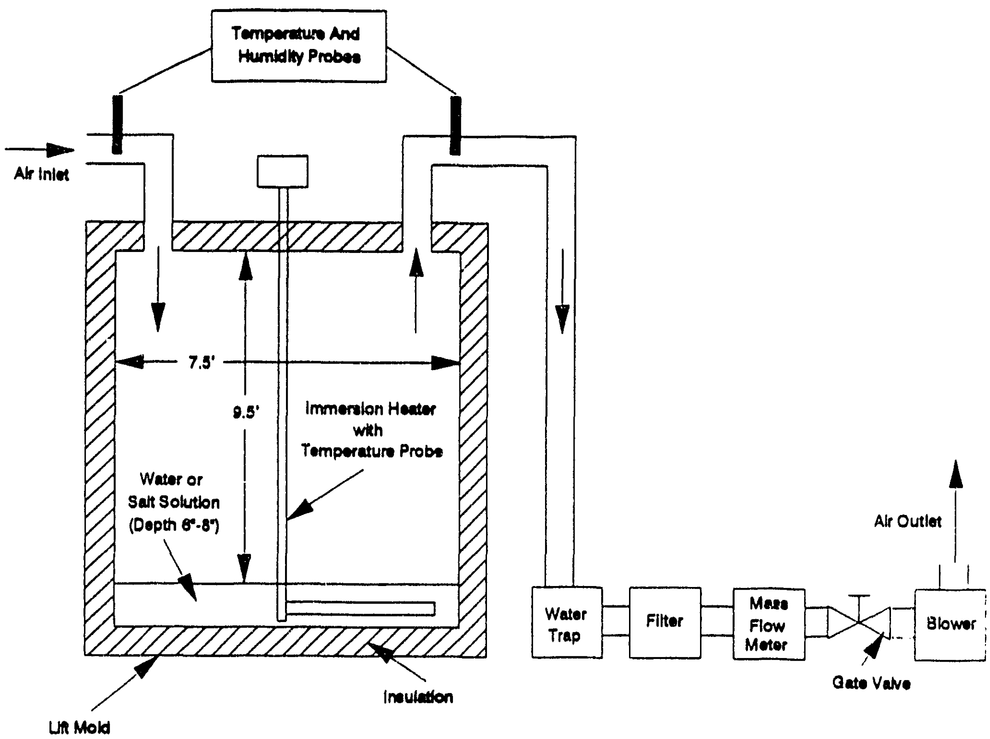


FIGURE 6.6. Experimental Setup for Heat Removal Tests

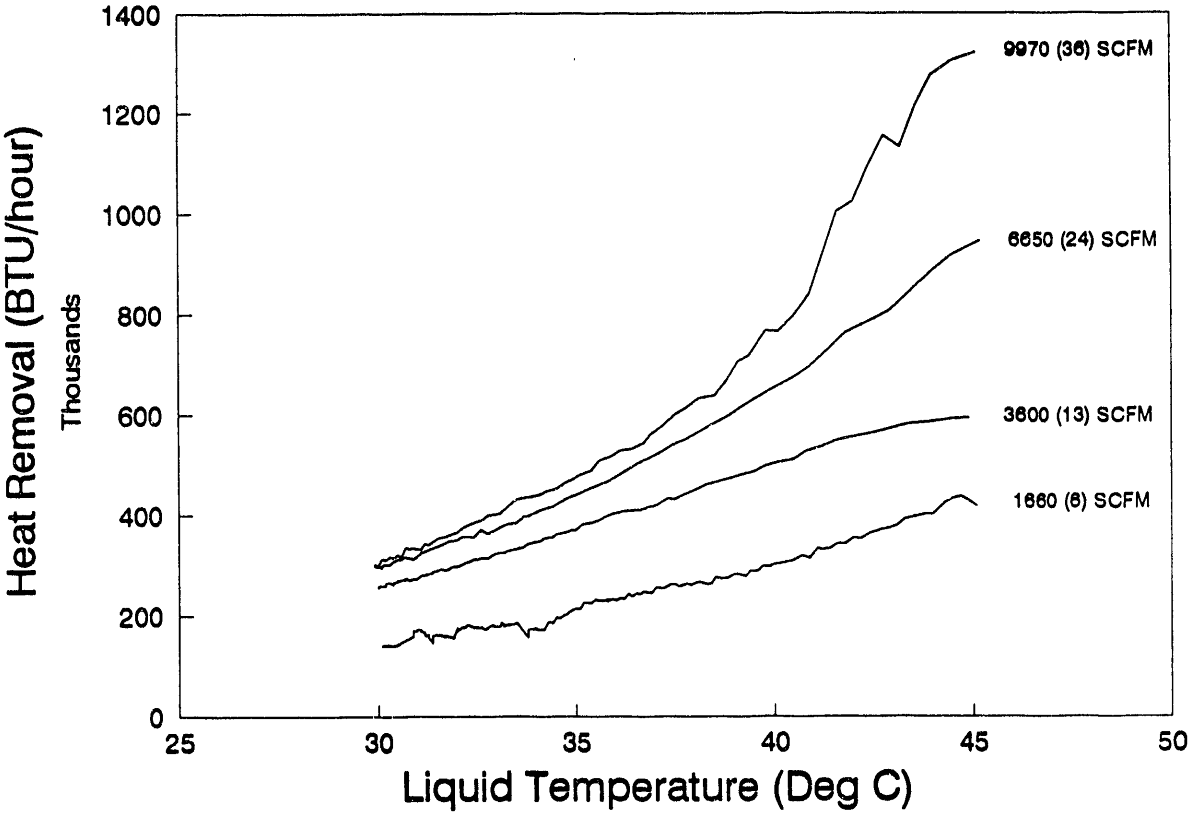


FIGURE 6.7. Heat Removal from the Surface of Water

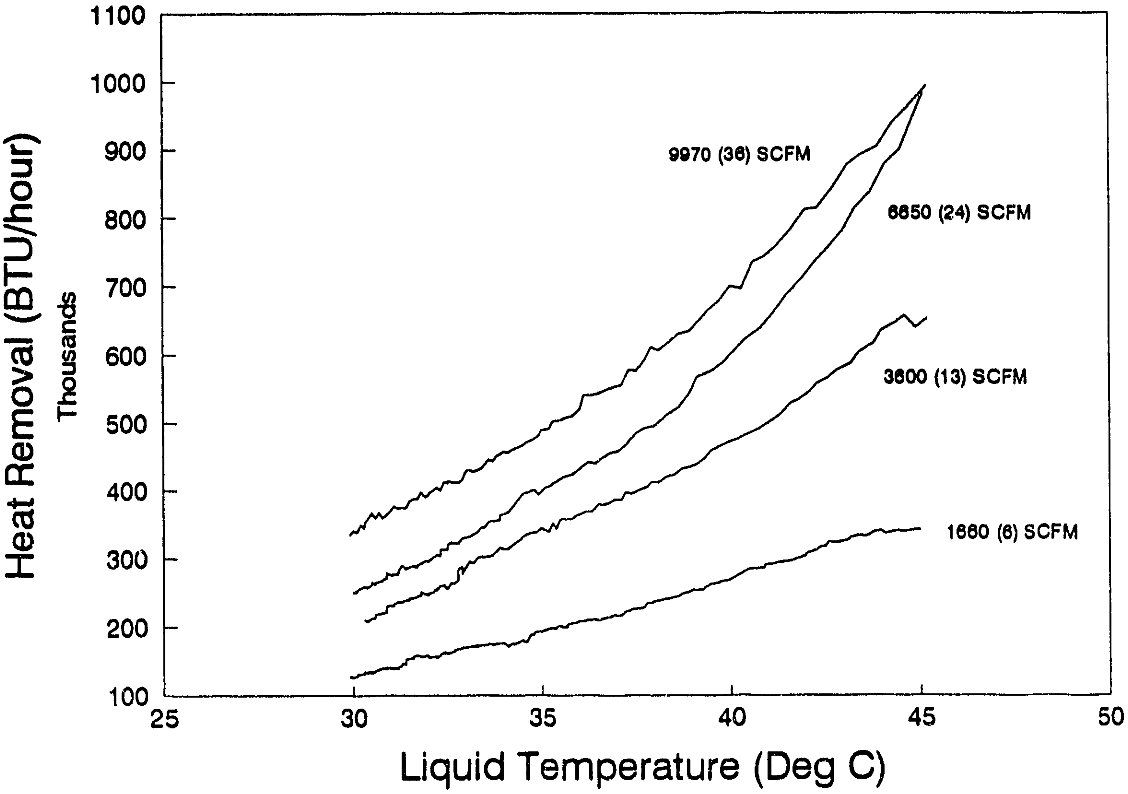


FIGURE 6.8. Heat Removal from the Surface of a $NaNO_3$ Solution

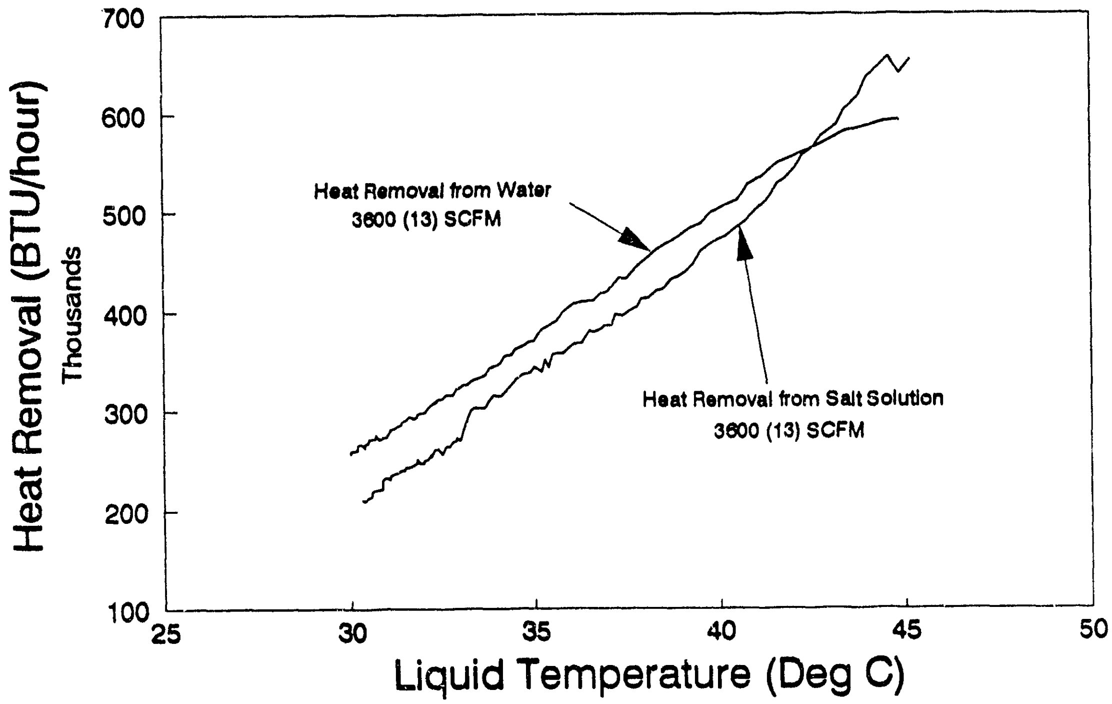


FIGURE 6.9. Comparison of Heat Removal from Water and $NaNO_3$ Solution with a 3600 (13) scfm Airflow

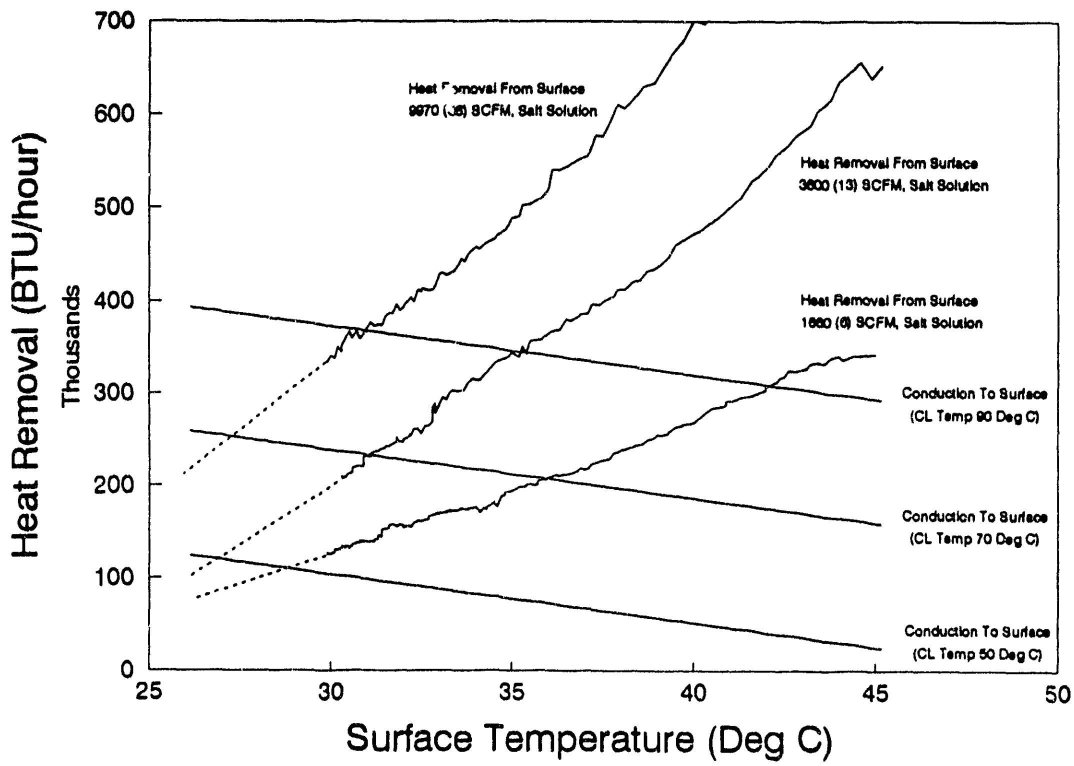


FIGURE 6.10. Rate Limiting Step in Grout Heat Removal

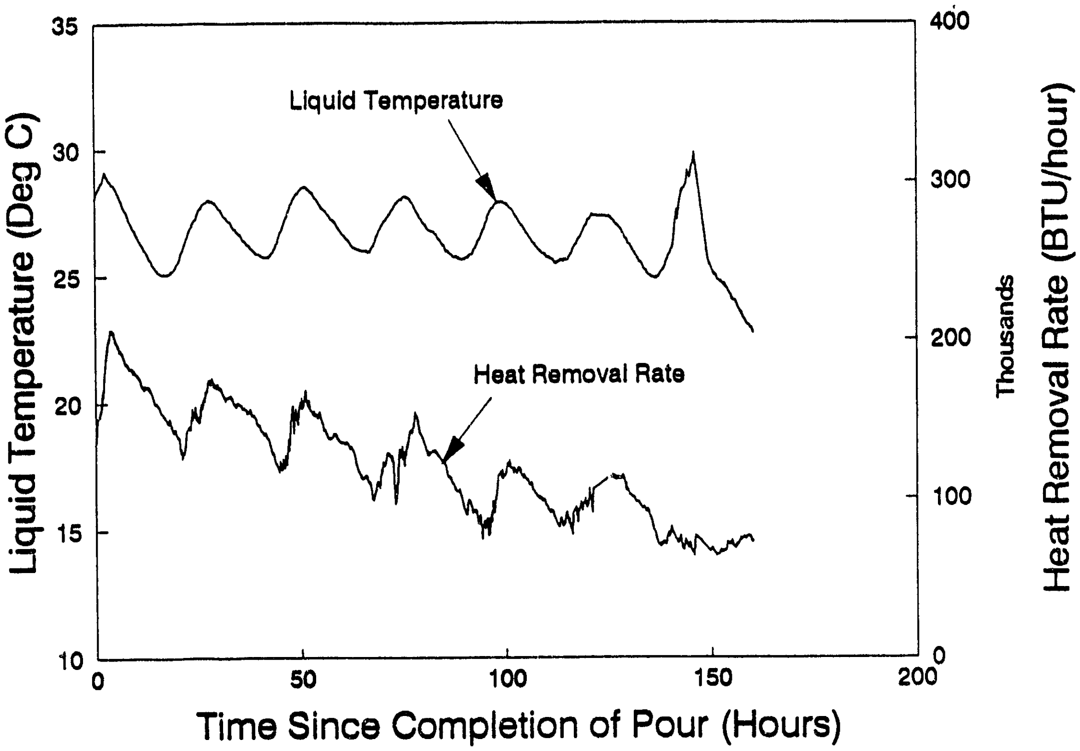


FIGURE 6.11. Heat Removal Rates and Liquid Temperatures for Lift 2

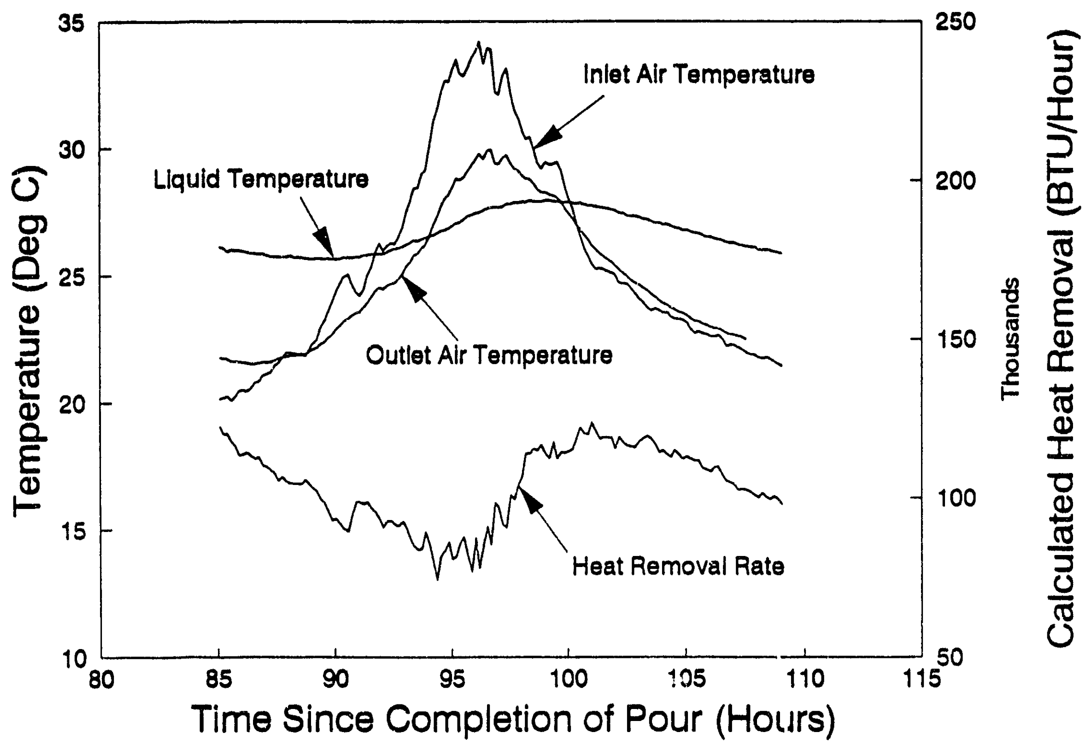


FIGURE 6.12. 24-Hour Temperature Cycle for Lift 2

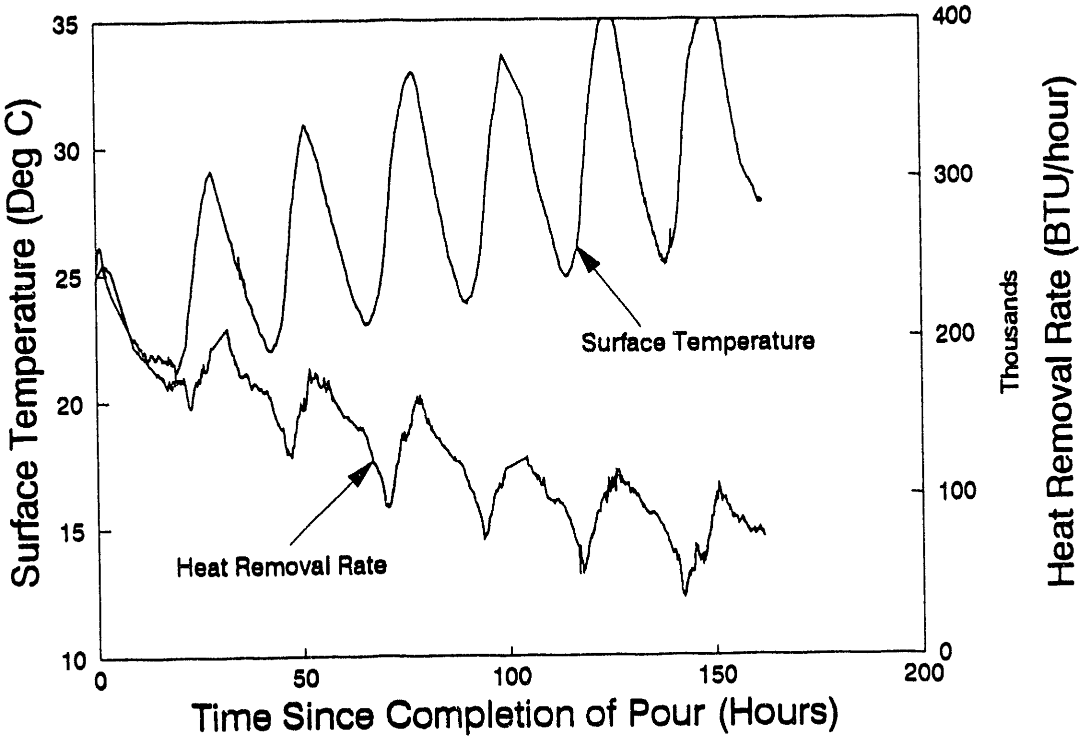


FIGURE 6.13. Heat Removal Rates and Surface Temperatures for Lift 3

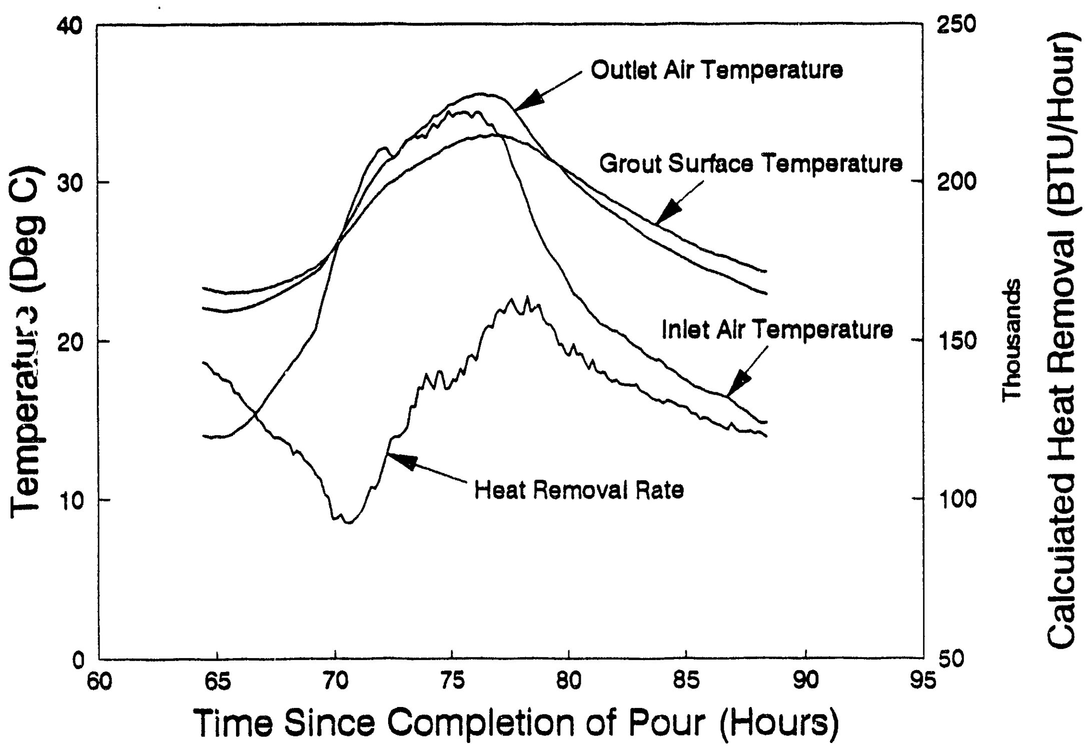


FIGURE 6.14. 24-Hour Temperature Cycle for Lift 3

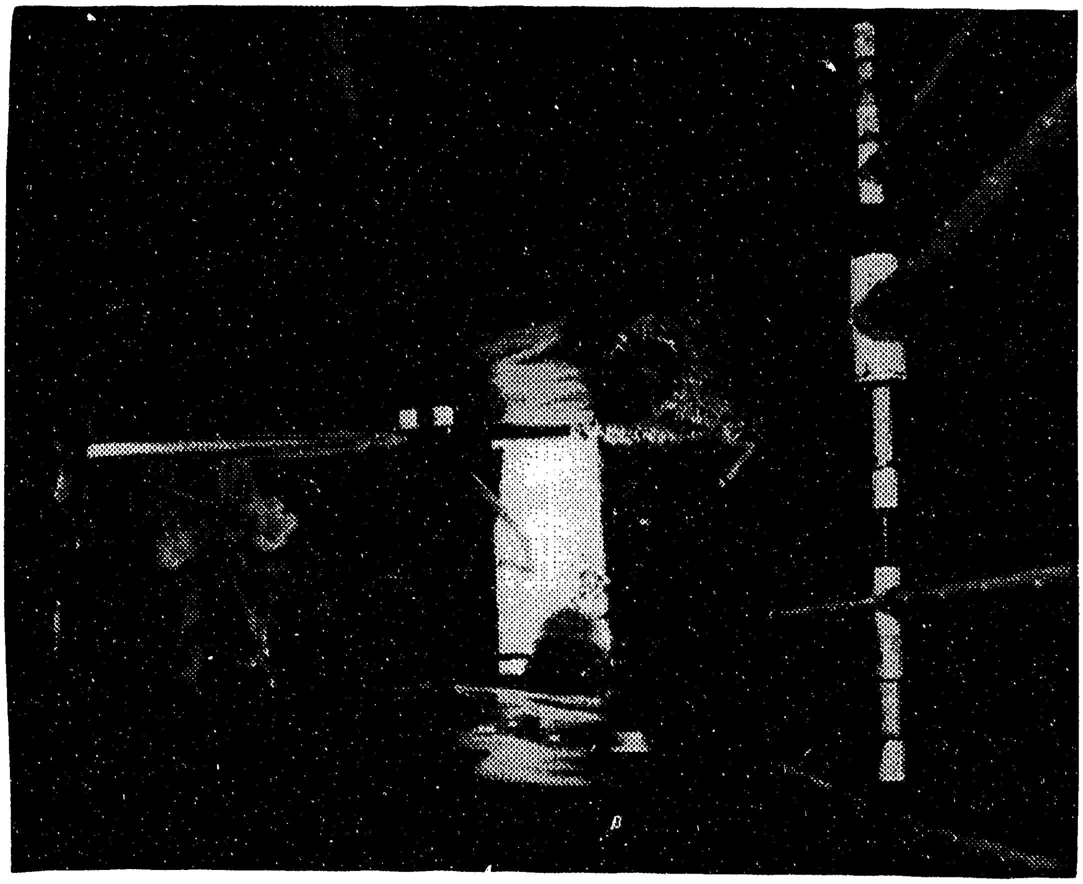


FIGURE 6.15. Surface of Second Lift 1 Week After Pouring

FIGURE 6.16. Surface of Third Lift 1 Week After Pouring

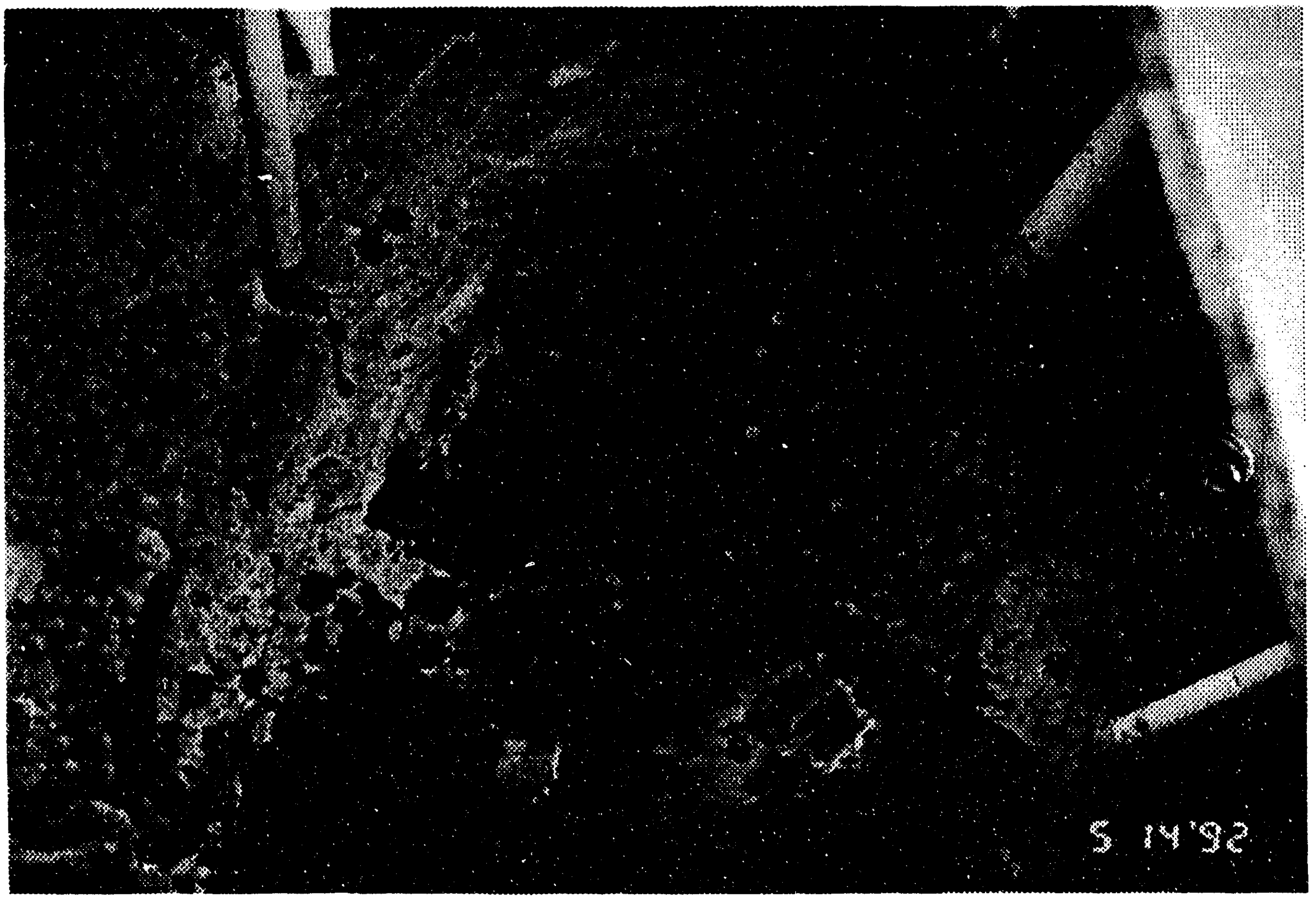
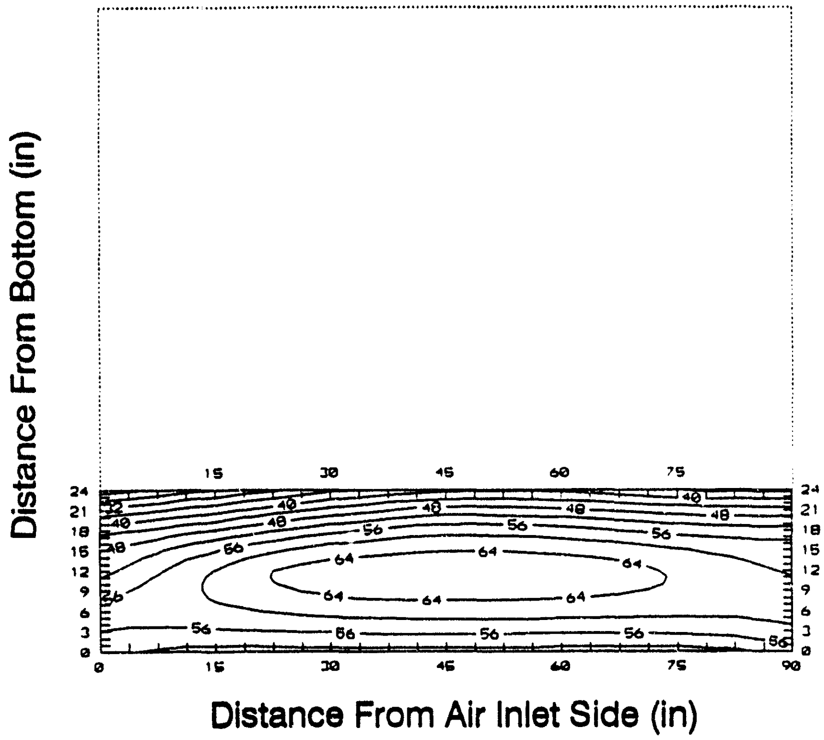
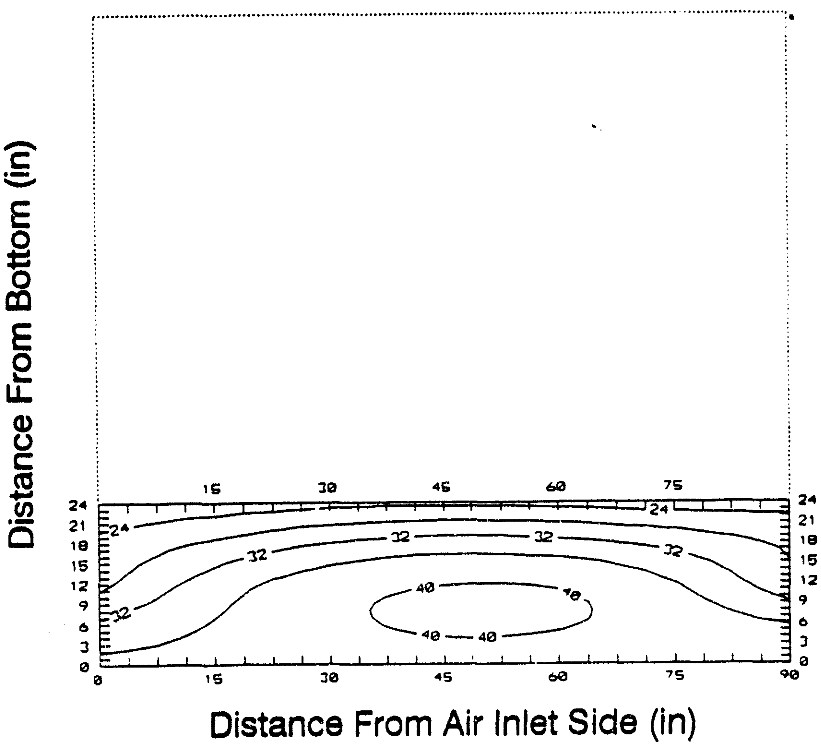


FIGURE 6.17. Surface of Fourth Lift 1 Week After Pouring



<u>FIGURE 6.18</u>. Peak Temperature Profile for Lift 1
(Centerline Temperatures at t = 33 hours)



<u>FIGURE 6.19</u>. Temperature Profile for Lift 1 After 1-Week
(Centerline Temperatures at t = 164 hours)

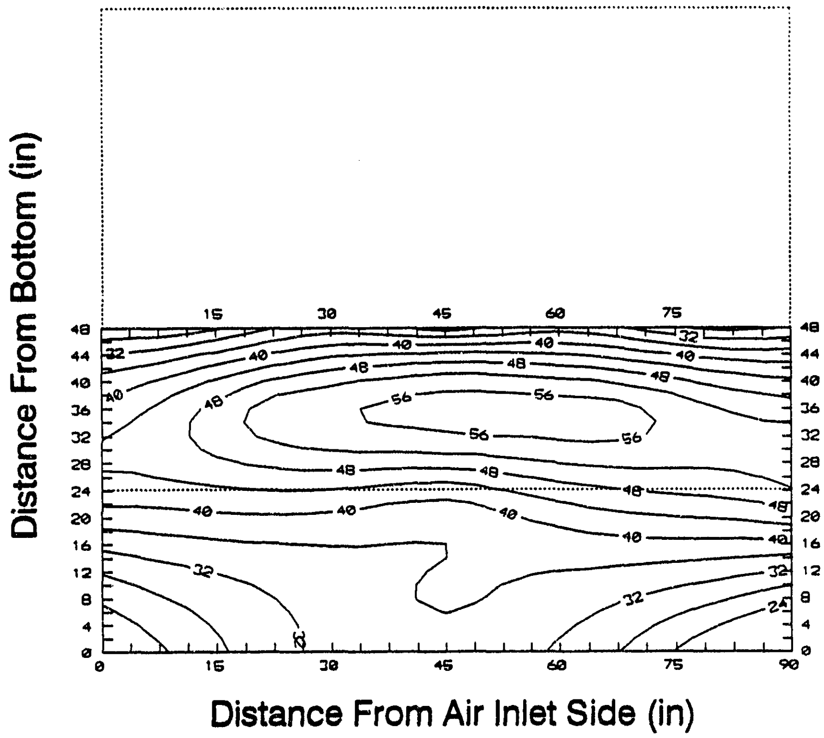


FIGURE 6.20. Peak Temperature Profile for Lift 2 (Centerline Temperatures at t = 195 hours)

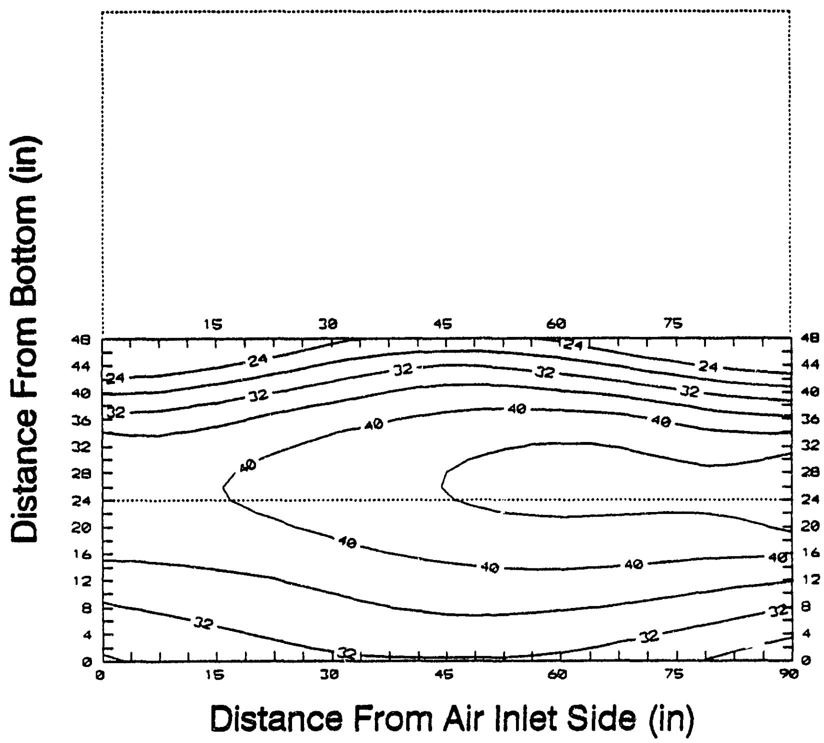


FIGURE 6.21. Temperature Profile After Cooling Lift 2 for 1 Week (Centerline Temperatures at t = 332 hours)

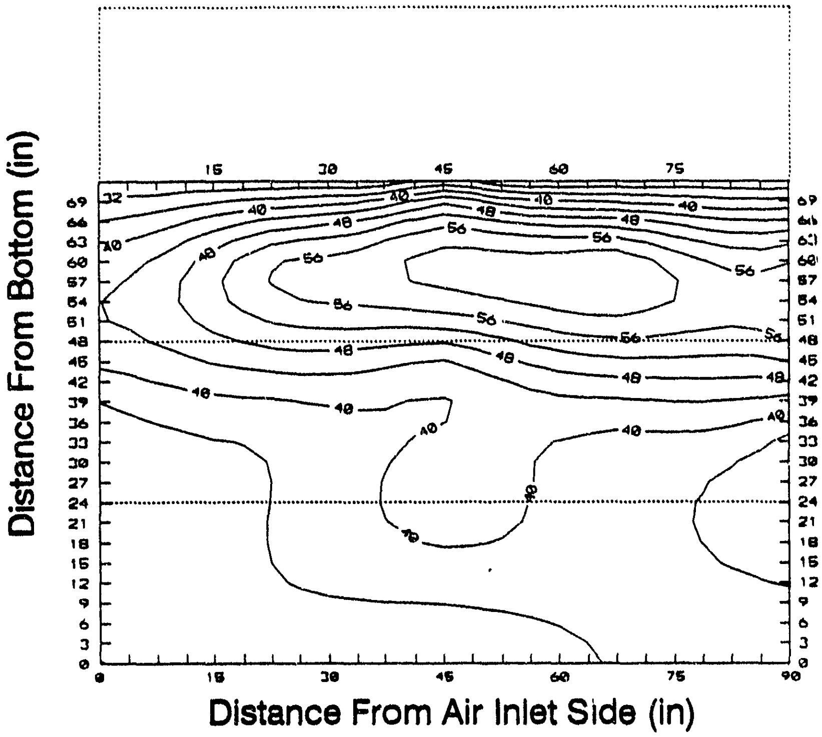


FIGURE 6.22. Peak Temperature Profile for Lift 3 (Centerline Temperatures at t = 365 hours)

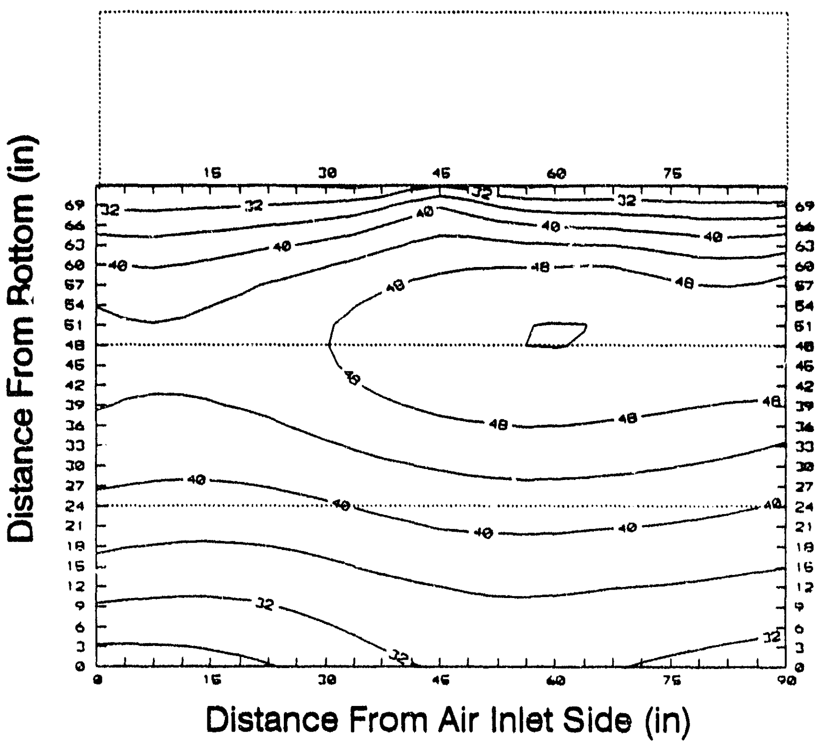


FIGURE 6.23. Temperature Profile After Cooling Lift 3 for 1 Week (Centerline Temperatures at t = 500 hours)

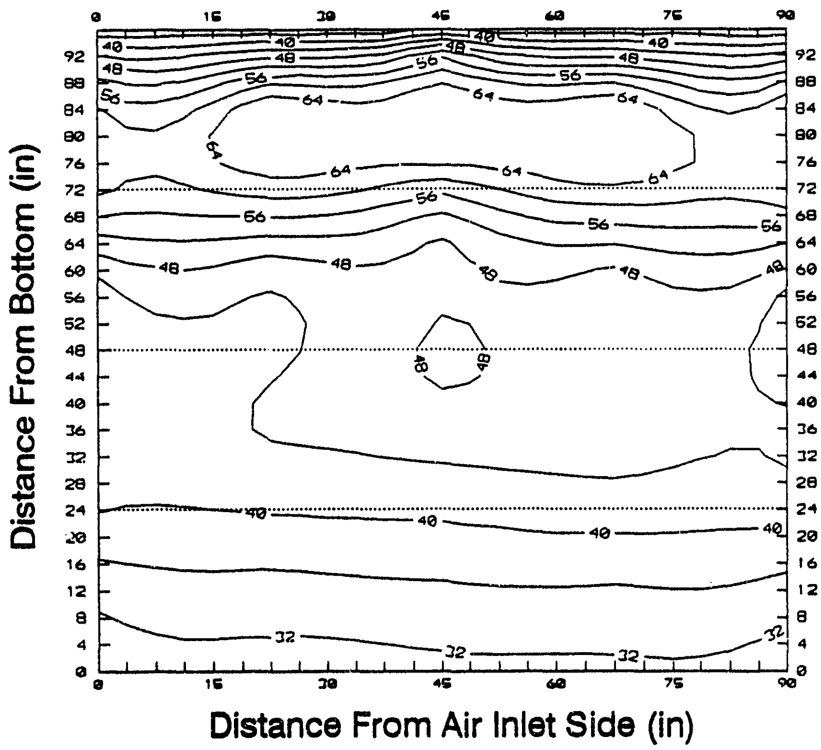


FIGURE 6.24. Peak Temperature Profile for Lift 4 (Centerline Temperatures at t = 535 hours)

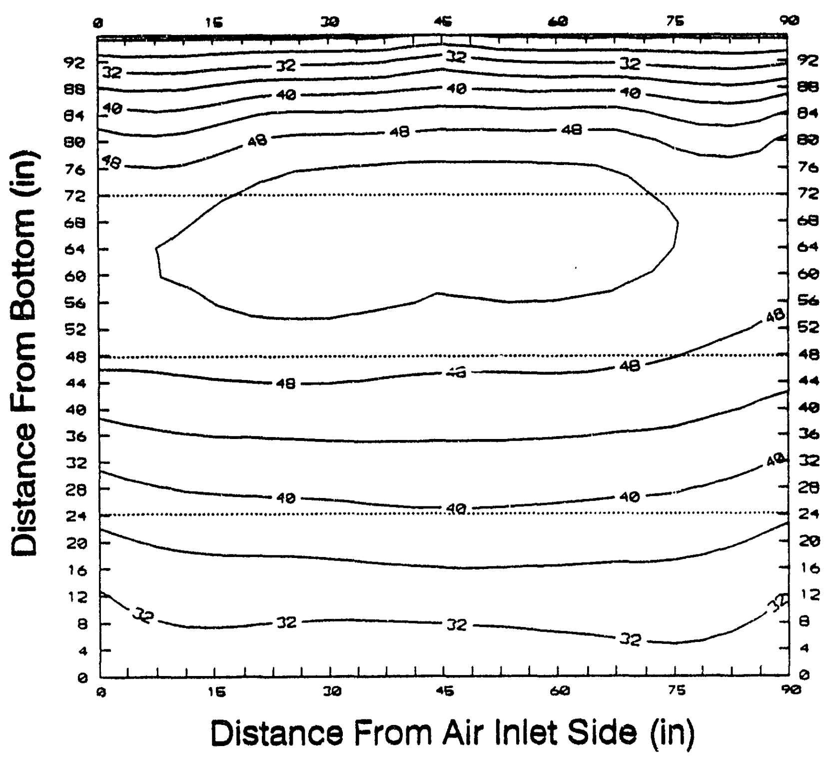


FIGURE 6.25. Temperature Profile After Cooling Lift 4 for 1 Week (Centerline Temperatures at t = 668 hours)

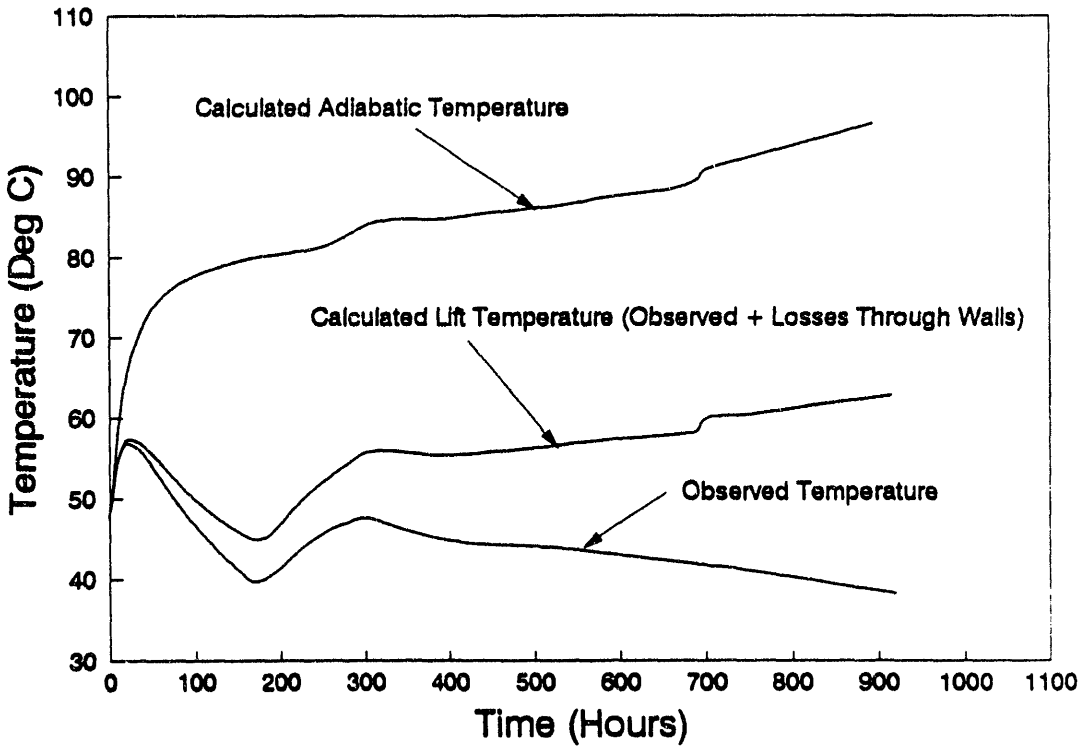


FIGURE 6.26. Estimated Long-Term Temperature Profiles for Lift 2

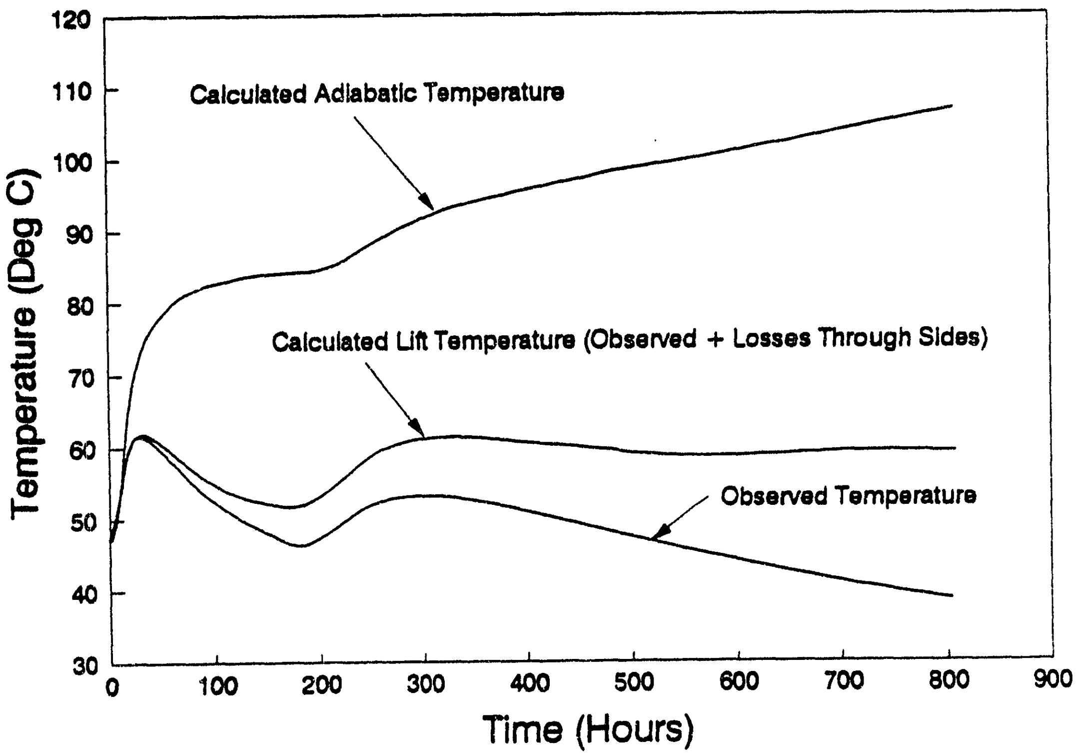


FIGURE 6.27. Estimated Long-Term Temperature Profiles for Lift 3

7.0 POST RUN EQUIPMENT OBSERVATIONS

Three weeks after completion of the fourth lift, the grout equipment was disassembled and inspected for grout buildup and mixer wear.

7.1 GROUT BUILDUP

The grout buildup in the equipment was the result of three aborted runs on the first day that grout was processed, the main grout pour 2 days later, and three smaller lift pours spaced at 1-week intervals. The pump restart tests were also conducted prior to inspection. At the conclusion of each of these runs, 25 gal of water was used to rinse the mixer, surge tank, grout pump, and grout pipe. After this water was pumped to the mold or dumpster, an additional 15 gal of water was circulated through the mixer, surge tank, and grout pump for 10 minutes. This water was then pumped to the dumpster. No attempt was made to remove the water that remained in the grout pipe.

The grout buildup in the mixer occurs in several different locations starting at the dry-blend inlet and continuing throughout the mixer. Figure 7.1 shows a large amount of grout buildup in the rectangular portion of the dry-blend inlet. Grout buildup was also noted in the dry-blend inlet line from the shaker screen (see Figure 7.2). While operating, these areas are not exposed to moisture, and grout should not form. However, rinses of the dry-blend inlet might introduce water that can form grout in subsequent runs. In addition, during the waste feed interruption, which caused an unscheduled shutdown during the main production run, wetted material had backed up into the dry-blend feed tube. This may have been when the grout in the rectangular area of the dry-blend inlet formed. The buildup in the dry-blend inlet line was the only area that had to be cleaned between runs to prevent grout production problems.

Figure 7.3 shows a thin layer of grout buildup on the lid of the mixer. This layer shows the tolerance between the mixer paddles and the mixer lid. Grout buildup in this location is probably not an operational concern because large pieces that fall off during production will be reduced to a small enough size to pass through the pump. However, this area may be difficult to decontaminate.

Figures 7.4 and 7.5 show the interior of the mixer. The paddles under the waste inlet are very clean, while the other paddles and the dry-blend feed screws have patches of grout. This implies that direct impingement of water will clean off the uncured grout, while other portions, which are simply wetted with water, will not be cleaned. Again, these patches of grout pose no problems during production but may be difficult to decontaminate.

Buildup in the grout discharge funnel is shown in Figure 7.6. In areas where grout flow is continuous, there is little buildup. Areas that are exposed to only splashed materials have a significant buildup. The discharge is designed differently in the production facility, and the pattern of grout buildup will probably be different.

Figures 7.7 and 7.8 show that no large areas of grout buildup occur in the surge tank but that a thin layer of material coats most of the interior surface. The clean areas on the surge tank lid again show that direct impingement by the water spray will remove the unsolidified grout. The spray nozzle used in the surge tank clogged easily and generally did a poor job of rinsing the surge tank. An improved spray nozzle design could reduce the buildup in the surge tank. The grout retained in the surge tank should not present an operational problem but may be difficult to completely decontaminate.

Figure 7.9 shows the grout buildup at the pump inlet. The design of the pump inlet has a dead spot that traps grout at the completion of a run. Water rinses removed enough grout to prevent operational difficulties, but this area will require decontamination.

Figure 7.10 shows the small amount of grout buildup at the pump outlet. This buildup is where stagnant grout would tend to settle and probably occurred after allowing grout to sit in this area during the restart tests. Therefore, grout buildup in this area during normal operations would probably not be a concern.

The initial 25 gal of rinse water used to flush the systems amounted to approximately 7.5 pipe volumes. Seven and one half pipe volumes of rinse water in the production system would equate to approximately 600 gal of water. At the end of the 25-gal rinse, the water at the pipe discharge was fairly clean and was probably adequate. However, since the grout pipe had to be used for four separate runs, the 15 gal of recirculated rinse water was also pumped through the grout pipe 15 minutes later.

A different procedure was used for the final flush of the grout pipe. Instead of immediately flushing the pipe with 25 gal of water, the 25 gal of rinse water was first recirculated through the grout equipment for 10 minutes before the pipe rinse was conducted. Thus, the total rinse water used for the final rinse was 7.5 pipe volumes of water.

Previous pilot-scale runs had encountered problems when attempting to reuse the grout pipeline. However, the proposed rinse at that time was only 2 to 3 pipe volumes of water. No problems were encountered when the grout pipe was reused four times for these tests, so the 40-gal flush (11.2 pipe volumes) was adequate. This rinse was also adequate to prevent buildup in the three-way valves used to divert the grout flow.

The last rinse used 7.5 pipe volumes of water. Three weeks after the completion of the last rinse, the interior of the grout pipe was examined at several points along its length. No buildup of grout was observed.

7.2 MIXER WEAR

Significant wear on the first set of mixer paddles and the dry-blend feed screws was observed in previous pilot-scale pours (Fow et al. 1987). For the current pilot-scale tests, dry-blend feed screws were replaced with stellite feed screws and the first four pairs of mixer paddles were replaced with stellite-tipped paddles. Since the production mixer has stellite components, the wear seen in this pilot-scale test should be more representative of the wear that might be expected in the production equipment. Figure 7.5 shows that there is little wear on either the first paddle or the feed screws. This indicates that stellite components reduce wear concerns.

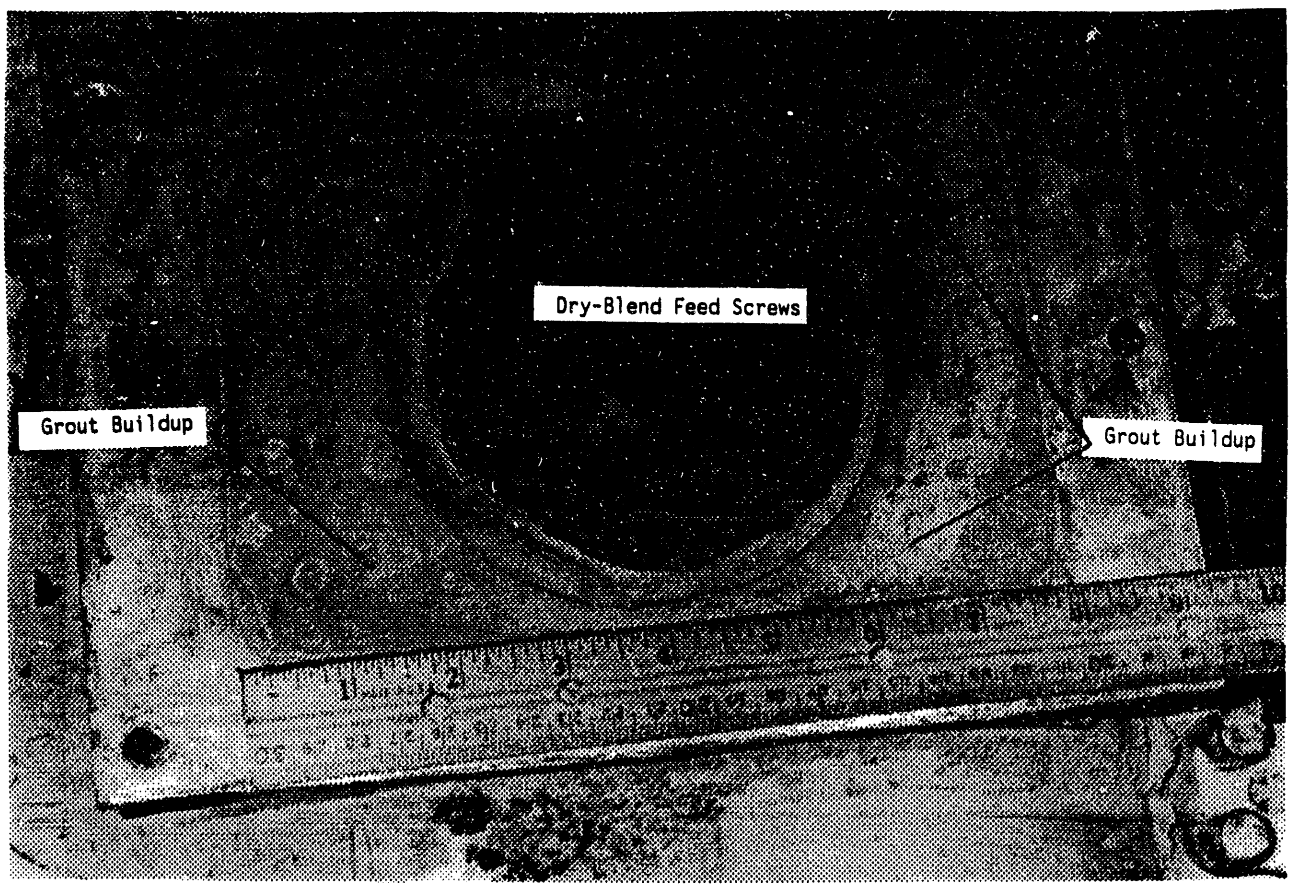


FIGURE 7.1. Grout Buildup at Mixer Inlet

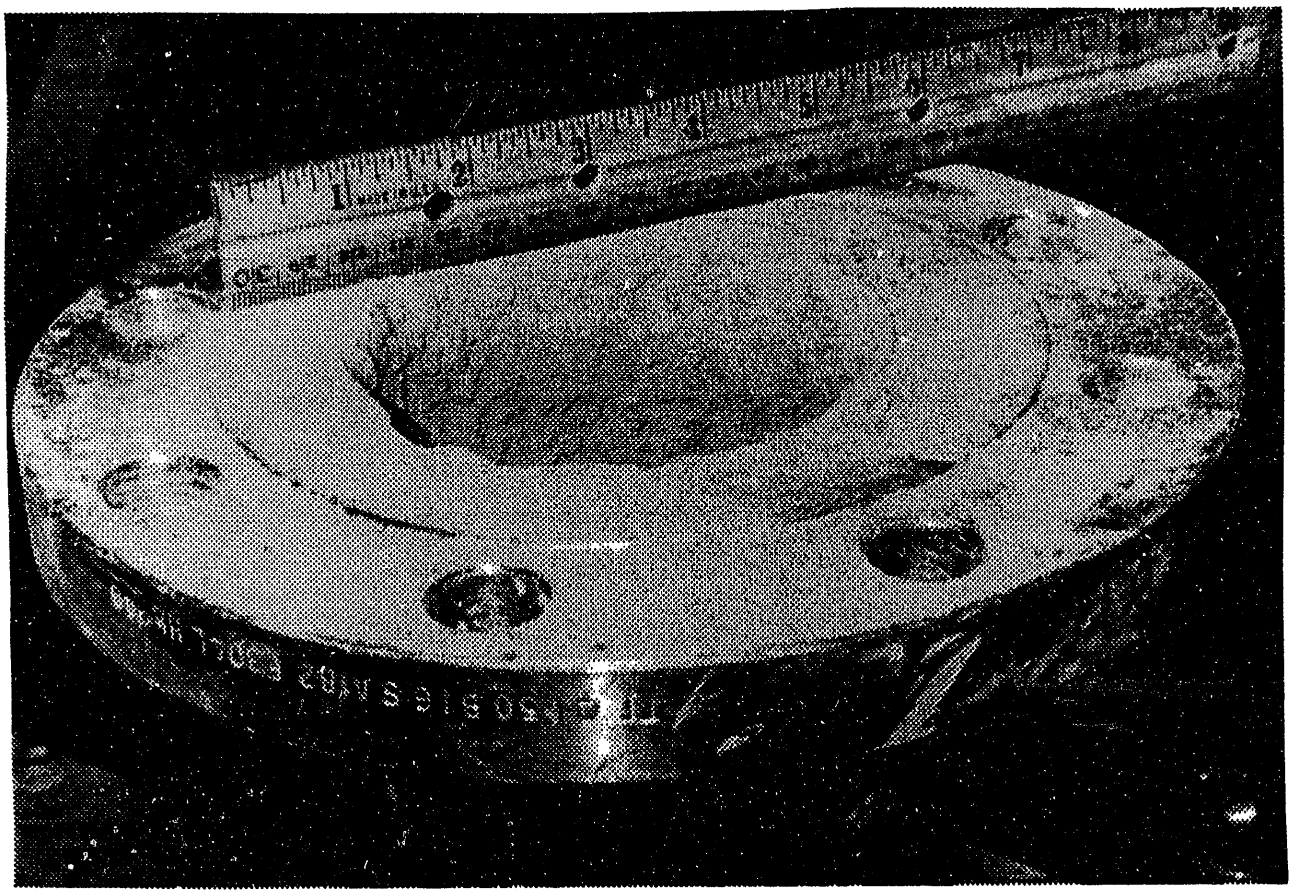


FIGURE 7.2. Grout Buildup in Dry-Blend Inlet Line

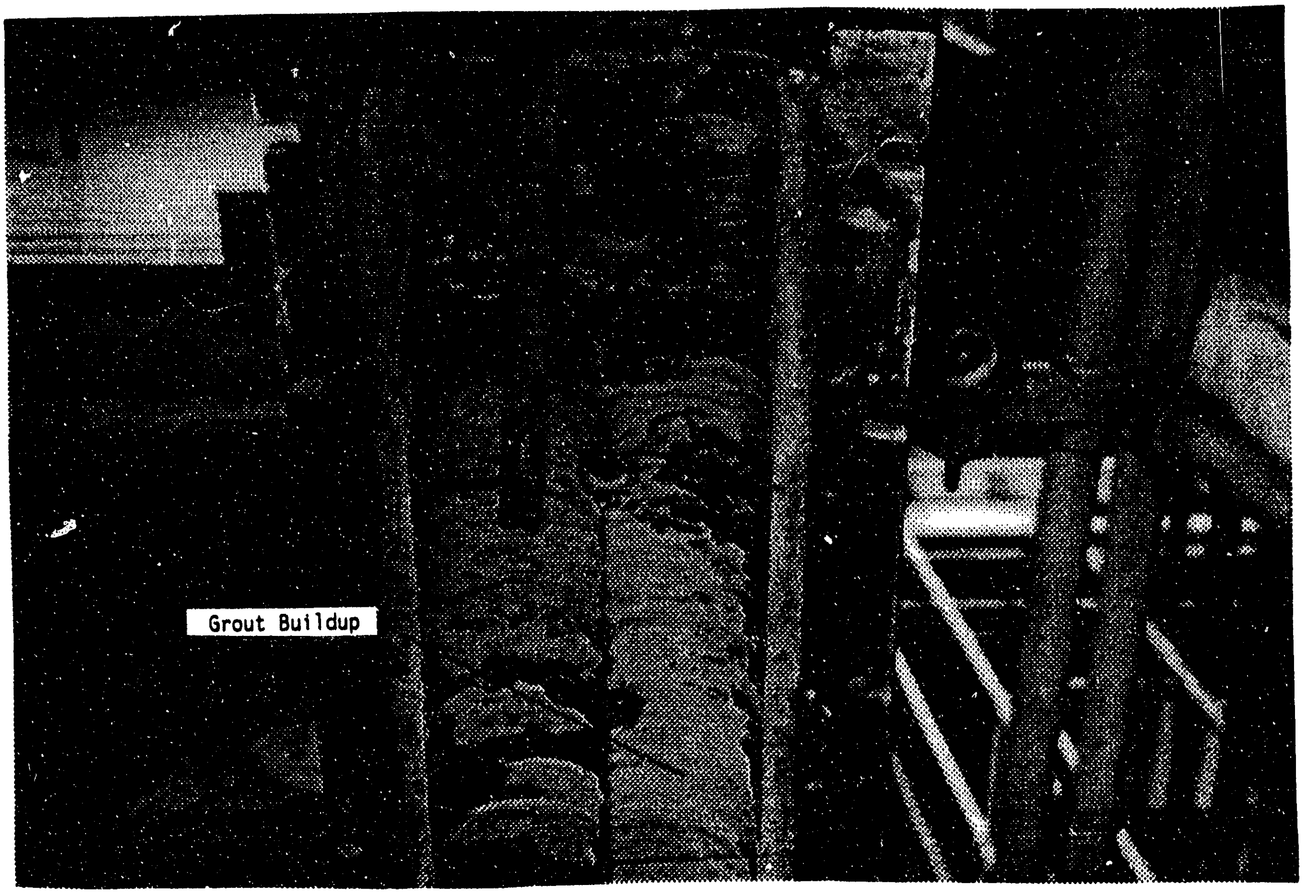


FIGURE 7.3. Grout Buildup on Mixer Lid

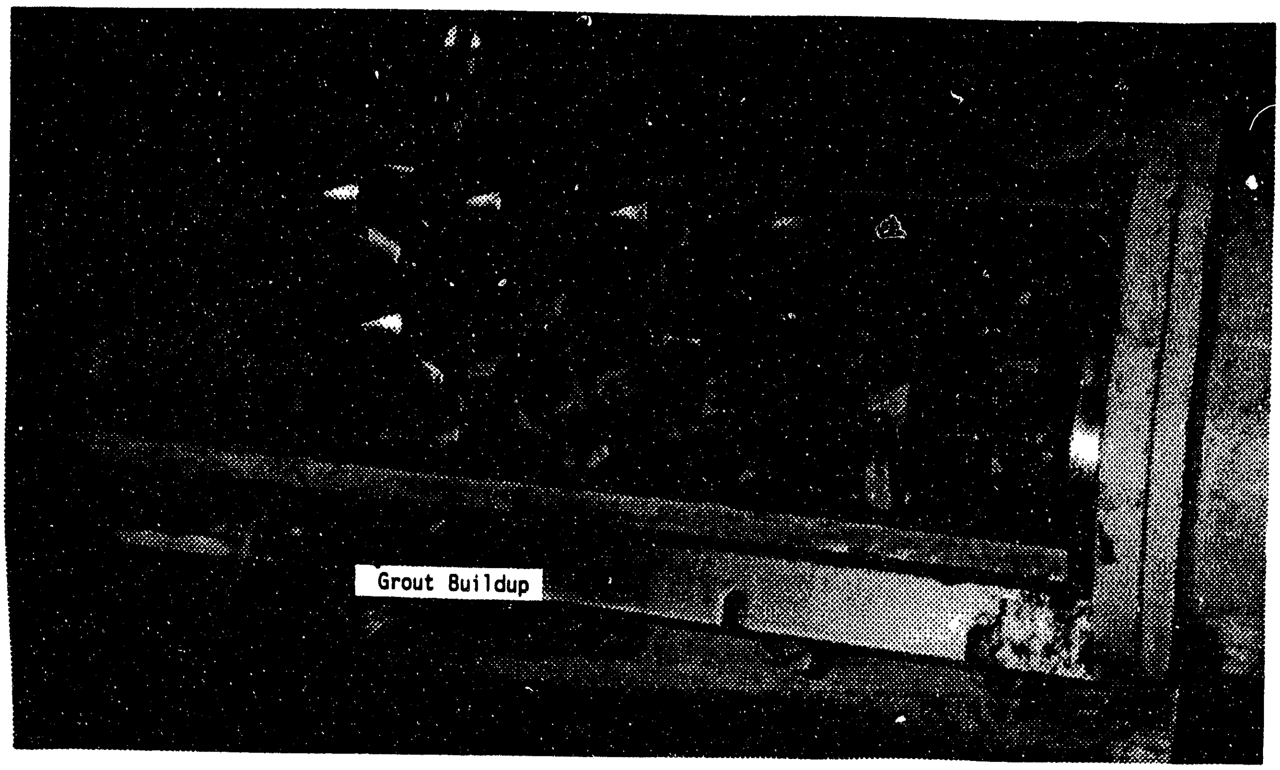


FIGURE 7.4. Grout Buildup on Mixer Paddles

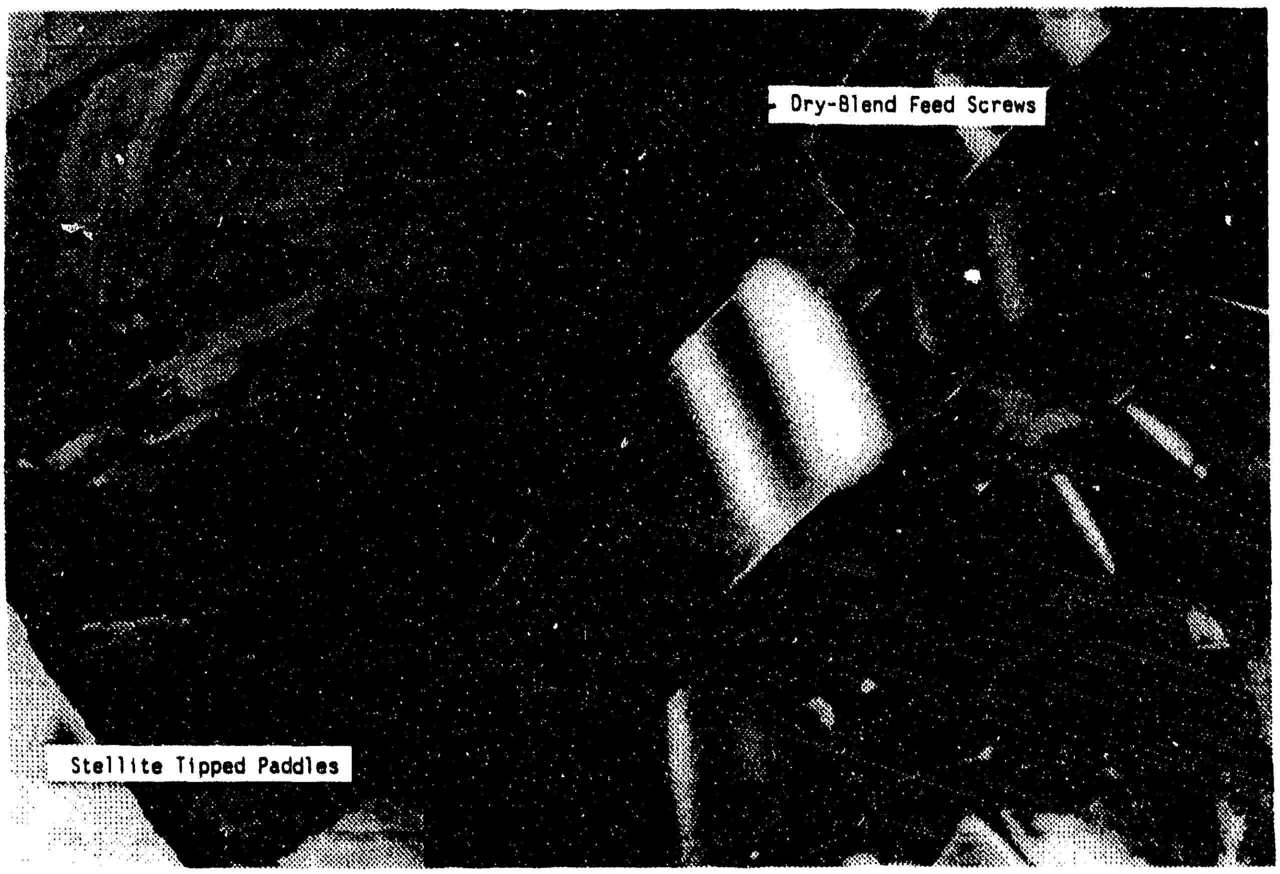


FIGURE 7.5. Stellite Tipped Mixer Paddles Under Waste Feed Inlet

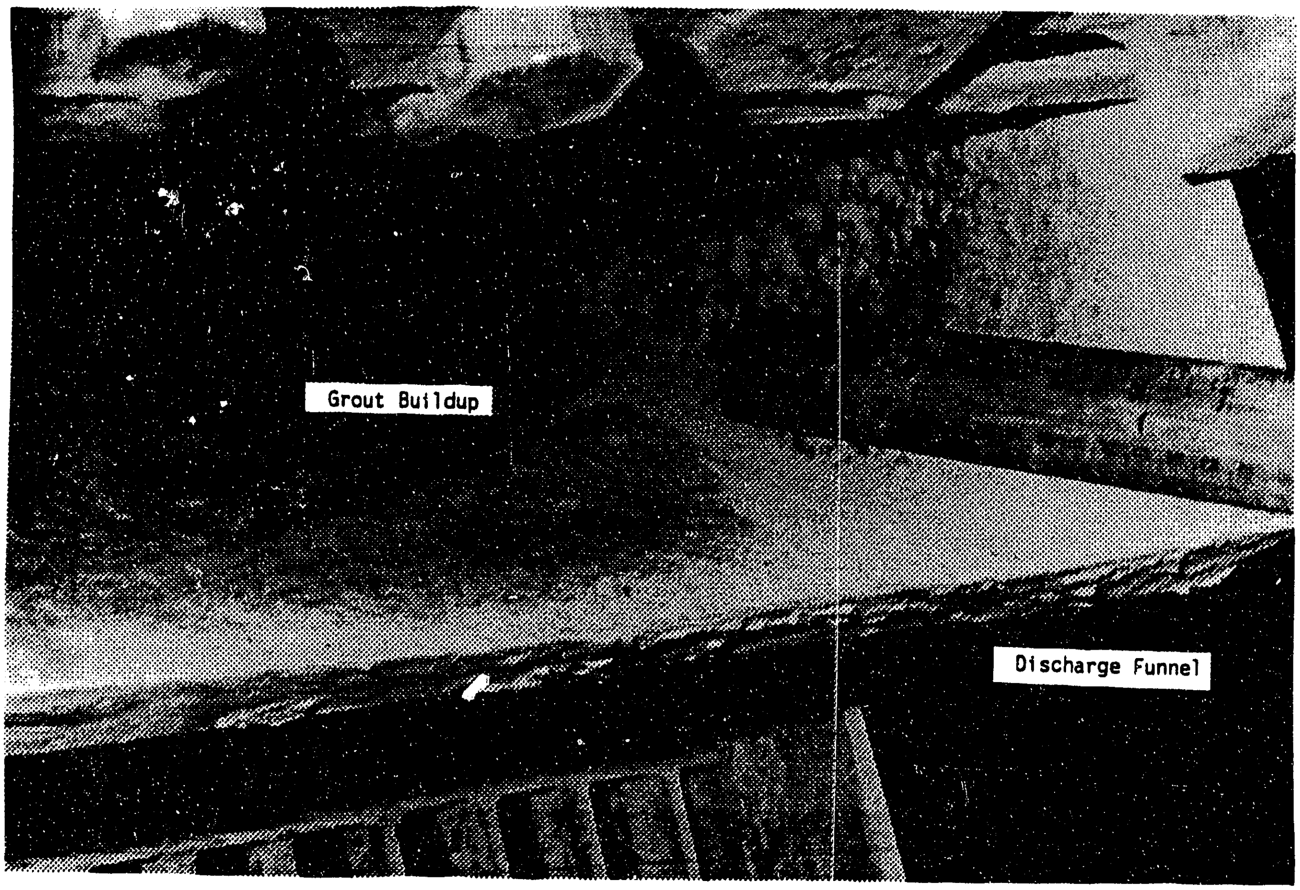


FIGURE 7.6. Grout Buildup at Mixer Discharge

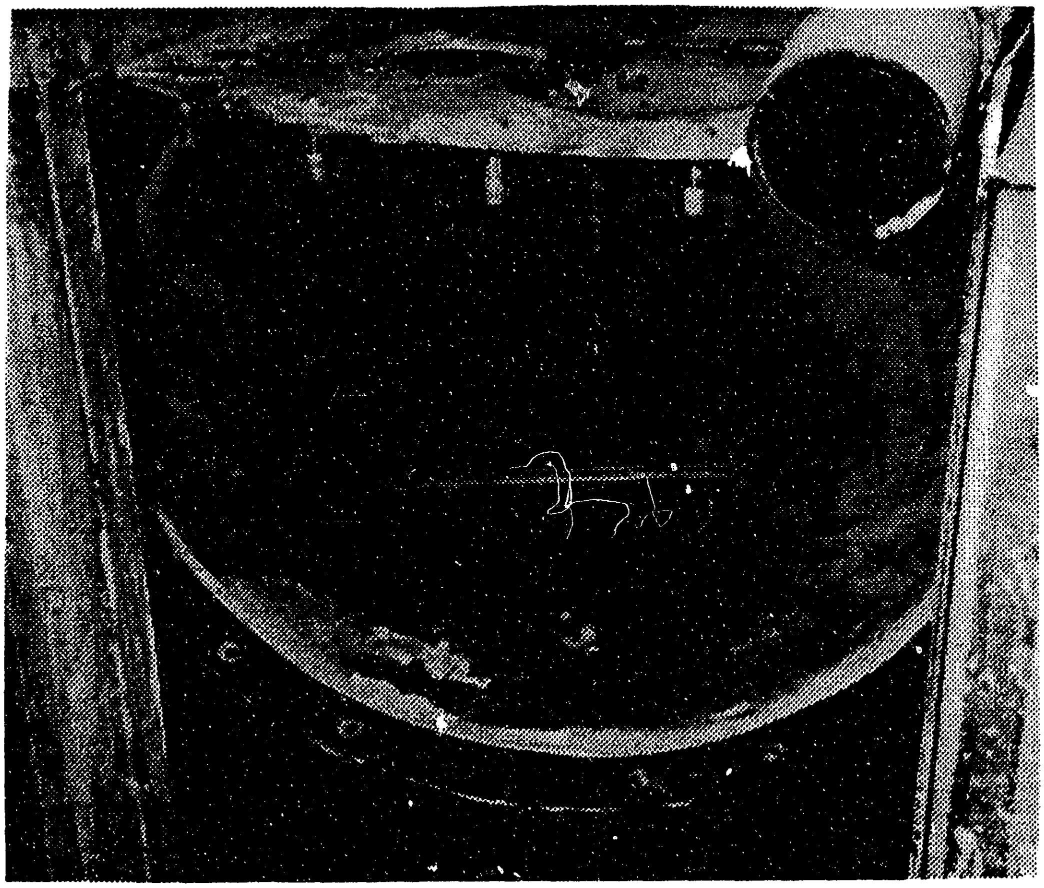


FIGURE 7.7. Grout Buildup in Surge Tank

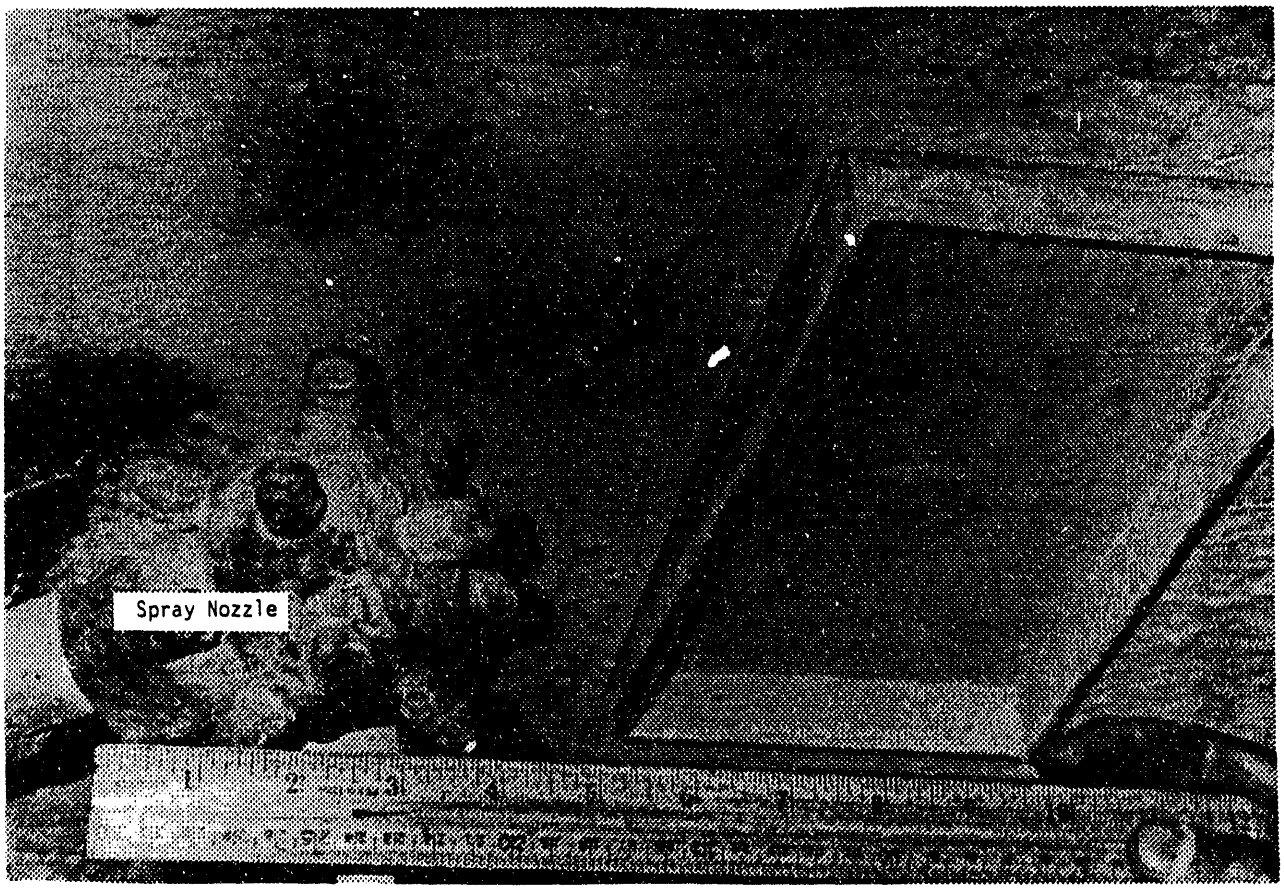


FIGURE 7.8. Grout Buildup on Surge Tank Lid

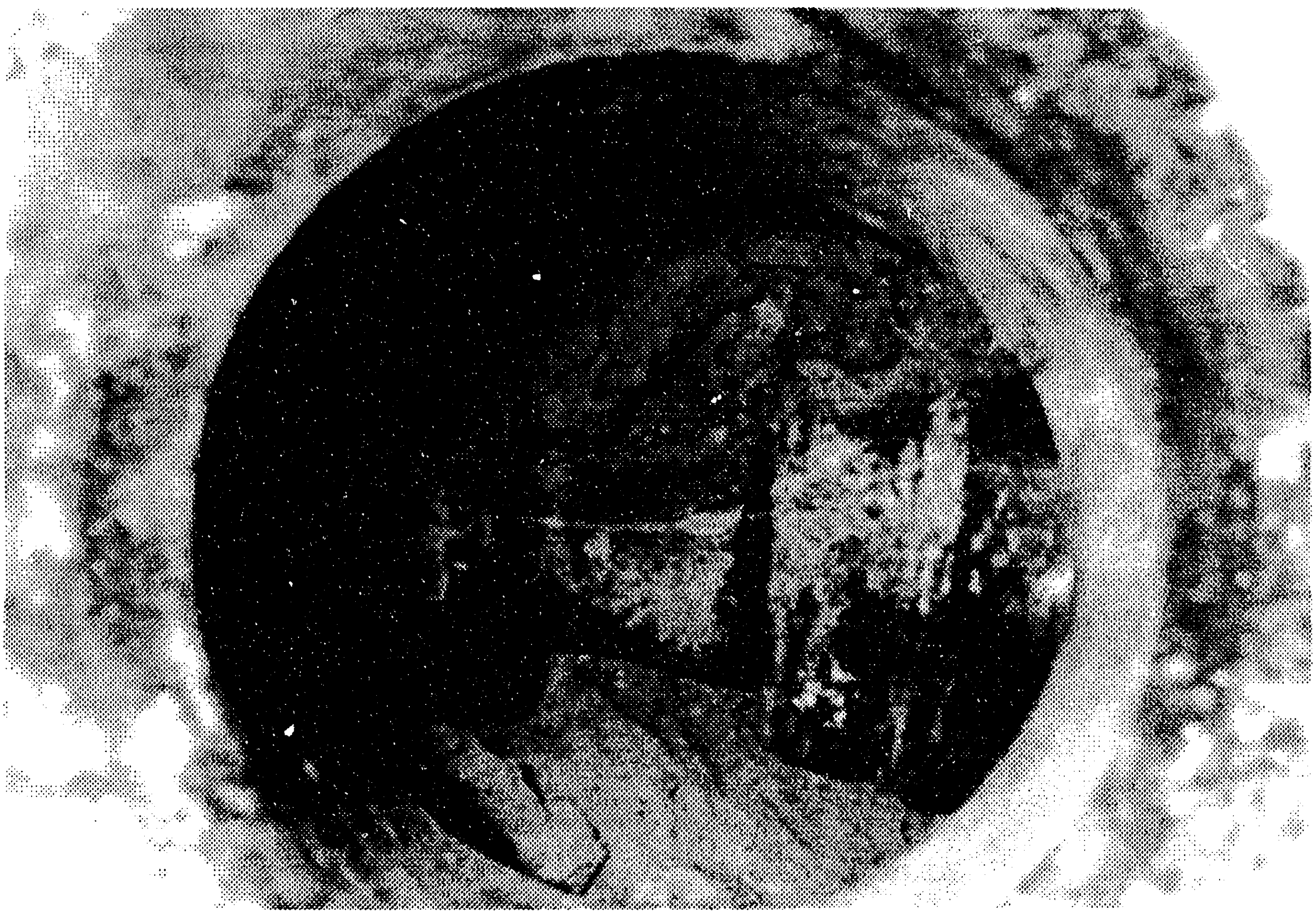


FIGURE 7.9. Grout Buildup at Pump Inlet

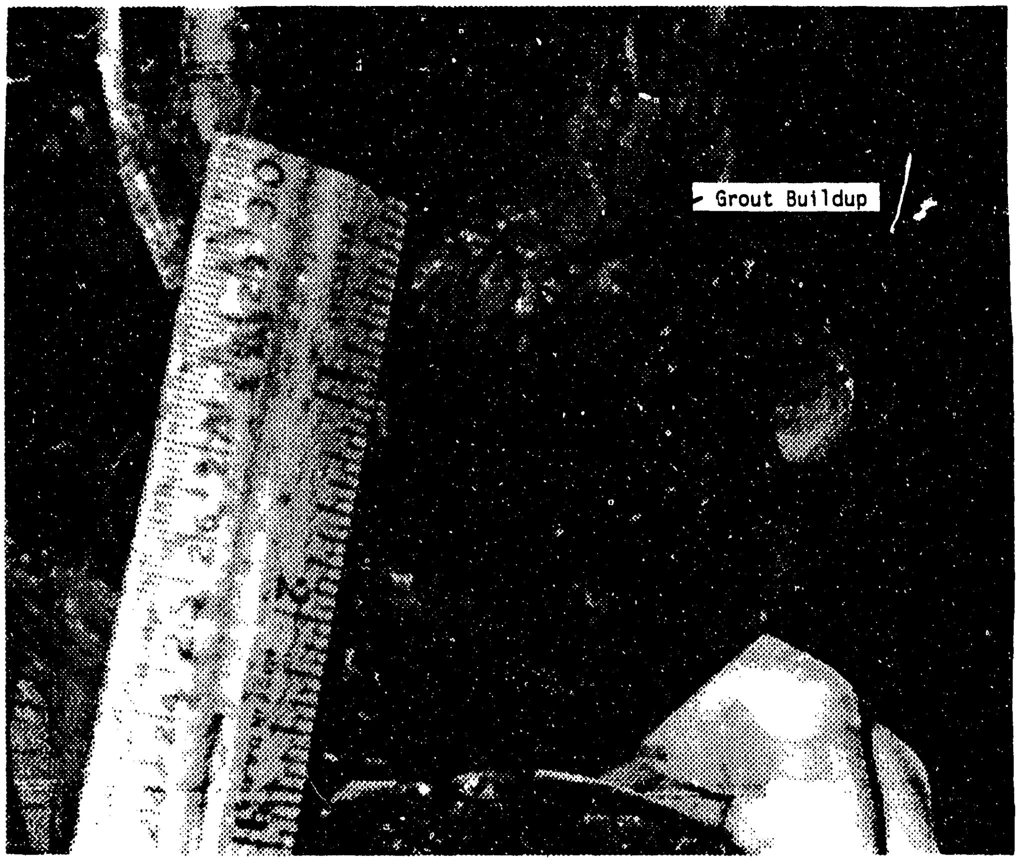


FIGURE 7.10. Grout Buildup at Pump Discharge

8.0 CONCLUSIONS/RECOMMENDATIONS

The following conclusions and recommendations are based on the results of the pilot-scale tests.

8.1 CONCLUSIONS

The pilot-scale testing satisfied the original objectives. The conclusions drawn from the testing are listed below:

- The grout produced with a dry-blend formulation consisting of 14 wt% attapulgite clay, 20 wt% cement, and 66 wt% class F fly ash showed significant shear thickening and had calculated critical flow rates at the pipe discharge that were above the criterion value of 60 gpm. Slight modification of the dry-blend formulation to 11 wt% attapulgite, 20.7 wt% cement, and 68.3 wt% class F fly ash reduced the critical flow rate to below 40 gpm. Other than the critical flow rate concerns, both formulations tested were readily processed by the pilot-scale equipment.

- The DMF handled the dry ingredients of the proposed production formulation and mixed dry-blend product within the desired tolerances.

- The restart pressure tests showed that process interruptions as long as 20 minutes did not pose a problem for the pilot-scale equipment. These tests indicated that interruptions of 30 minutes or greater should not be allowed without flushing the system.

- No significant wear was seen on the stellite feed screws and stellite-tipped paddles installed in the grout mixer.

- A 7.5 pipe-volume flush of the pilot-scale grout pipe at 10 gpm was sufficient to prevent buildup.

- Grout buildup in the equipment was similar to that seen in other pilot-scale runs. Buildup in the area of the dry-blend mixer inlet was a concern and may have interfered with grout production if it had not been cleaned between runs. Buildup in other areas did not interfere with grout production but might present decontamination problems.

- The dimensional changes of the grout over the first 7 weeks of curing were small (0.06% shrinkage).

- The thermal conductivity of this grout formulation was 0.81 W/m°K.

- Neither the original 14 wt% attapulgite clay formulation nor the modified 11 wt% attapulgite clay formulation had free liquids when poured at 40°C.

- The calculated adiabatic temperature rise of the grout poured in the gradient mold was 57°C.

- Comparing calculated heat conduction rates through grout to the experimentally-determined airflow heat removal rates from water/salt solutions showed that conduction of heat through the grout controls the heat removal rate when using the airflow rates planned for the production vault. As a result, increased airflows (e.g. larger blowers) would not significantly increase the heat removal rates.

- An airflow of 13 scfm in the lift mold (which simulated a 3600 scfm airflow in the production vault) kept the maximum short-term grout temperatures below 70°C for all four of the 2-ft lifts poured and maintained average grout surface temperatures below 30°C.

- The net temperature reduction obtained by cooling the surface of a 2 ft lift for 1 week was approximately 30°C.

- Heat removal rates throughout the week between pours were not significantly different for lifts with and without free-standing liquid.

- The lift mold thermal profiles after 1 week of cooling showed a general tendency, as lifts were added, for the peak temperatures to be higher and located farther below the surface with each subsequent lift.

8.2 RECOMMENDATIONS

The following are recommendations based on the results of the pilot-scale tests.

- A grout formulation of 11 wt% attapulgite clay, 20.7 wt% cement, and 68.3 wt% class F fly ash mixed at 8.4 lb/gal should be used for verification tests. This slightly modified formulation eliminates the critical flow rate concerns that were discovered while processing the 14 wt% attapulgite formulation. However, since this formulation has the same relative amounts of cement and fly ash as the original formulation, information obtained from grout produced with the first formulation should still be representative of that expected for the adjusted formulation.

- The grout surface should not be flooded with 1 to 2 in. of cooling water at the completion of the run. Flooding the surface affects the short-term curing of the grout at the surface of the lift and is not necessary for effective cooling. There is probably sufficient water in the grout pour solution to supply all the evaporation water, but tests that investigate the water-vapor pressure over the grout as the pore water evaporates would help determine if additional evaporation water is necessary. If additional evaporation water is necessary, it should be added after the grout has cured for several days.

- It is difficult to suggest a pour schedule from the information obtained from the lift mold tests, but several useful observations were made that helped direct the modeling efforts. 1) When using the planned airflow rates, the heat removal from the grout is mainly controlled by the rate at which heat will conduct through the grout to the surface. As long as water is available, convective/evaporative cooling can be expected to keep the grout surface temperature below 30°C. 2) The lower grout temperatures, which result due to surface cooling, may also reduce the hydration reaction rates. This is an important factor in determining the pour schedule since hydration heat released while the grout is close to the surface is relatively easy to remove, but heat released from grout at significant depths can only be removed through much slower heat-release mechanisms. In order to model these effects, the heat release as a function of temperature and extent of reaction must be available. Calorimetry work should be conducted on the proposed formulation to generate the required data. 3) The total hydration heat determined in this experiment was 5100 Btu/Cubic Foot. This number is important in estimating the long-term temperature profiles in the grout and the final pour schedule. Calorimetry work should be performed to confirm this number.

- Future pilot-scale tests should use initial dry-blend active hopper settings of 90% for the high level and 70% for the low level to avoid dry-blend flooding problems.

- The shear-thickening effects of attapulgite should be studied in greater detail if this material is part of future formulations.

9.0 REFERENCES

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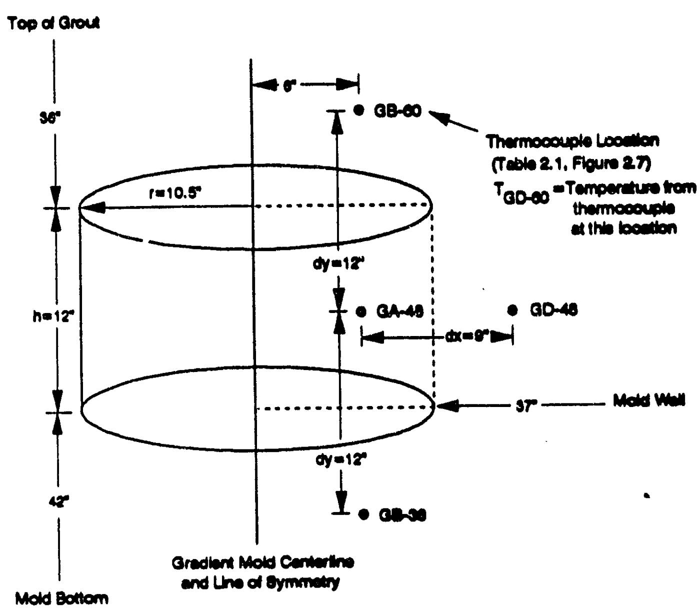
APPENDIX

ADIABATIC TEMPERATURE RISE CALCULATION FOR GRADIENT MOLD

APPENDIX

CALCULATION OF ADIABATIC TEMPERATURE RISE FOR GRADIENT MOLD

Heat Balance Geometry



Assumptions:

(1) Approximate thermal gradients as linear

(2) Temperature profile is symmetric with respect to vessel centerline

(3) Grout Heat Capacity - 0.527 cal/g°C

(4) Grout Thermal Conductivity - 0.81 W/m°K

(5) Grout Density - 1.55 g/cm^3

Heat balance for cylinder shown above:

TOTAL HEAT LOSS = HEAT LOSS THROUGH SIDES + HEAT LOSS THROUGH TOP + HEAT LOSS THROUGH BOTTOM

$$HEAT\ LOSS = Q = \left(\frac{dT}{dx}\right) A K_{GROUT} \Delta t$$

Where dT/dx = thermal gradient

A = heat loss area corresponding to thermal gradient

Δt = heat loss time period

K_{GROUT} = grout thermal conductivity

$$Q_{TOTAL} = Q_{SIDES} + Q_{TOP} + Q_{BOTTOM} \tag{1}$$

$$Q_{SIDES} = \left(\frac{T_{GA-48} - T_{GD-48}}{dx} \right) (2\pi rh) K_{GROUT} \Delta t$$

$$Q_{SIDES} = 0.4326 \left(T_{GA-48} - T_{GD-48} \right) \Delta t \quad cal \tag{2}$$

$$Q_{BOTTOM} = \left(\frac{T_{GA-48} - T_{GB-36}}{dy} \right) (\pi r^2) K_{GROUT} \Delta t$$

$$Q_{BOTTOM} = 0.1419 \left(T_{GA-48} - T_{GB-36} \right) \Delta t \quad cal \tag{3}$$

$$Q_{TOP} = 0.1419 \left(T_{GA-48} - T_{GB-60} \right) \Delta t \quad cal \tag{4}$$

Combining Equations 1-4 gives:

$$Q_{TOTAL} = \left(0.4316 \left(T_{GA-48} - T_{GD-48} \right) + 0.1419 \left(2T_{GA-48} - T_{GB-36} - T_{GB-60} \right) \right) \Delta t \quad cal$$

where Δt is in seconds

THEN: $$T_{ADIABATIC} = T_{GROUT} + \frac{Q_{TOTAL}}{THERMAL\ MASS\ OF\ GROUT}$$

$$T_{ADIABATIC} = T_{GA-48} + \frac{Q_{TOTAL}}{\rho_{GROUT} Cp_{GROUT}\left(\pi r^2 h\right)}$$

Where ρ_{GROUT} = Grout density

Cp_{GROUT} = Grout heat capacity

The adiabatic temperature at time = t is given by:

$$T_{ADIABATIC_t} = \left(T_{GA-48_t} + \frac{Q_{TOTAL_t}}{55{,}636}\right) {}^\circ C$$

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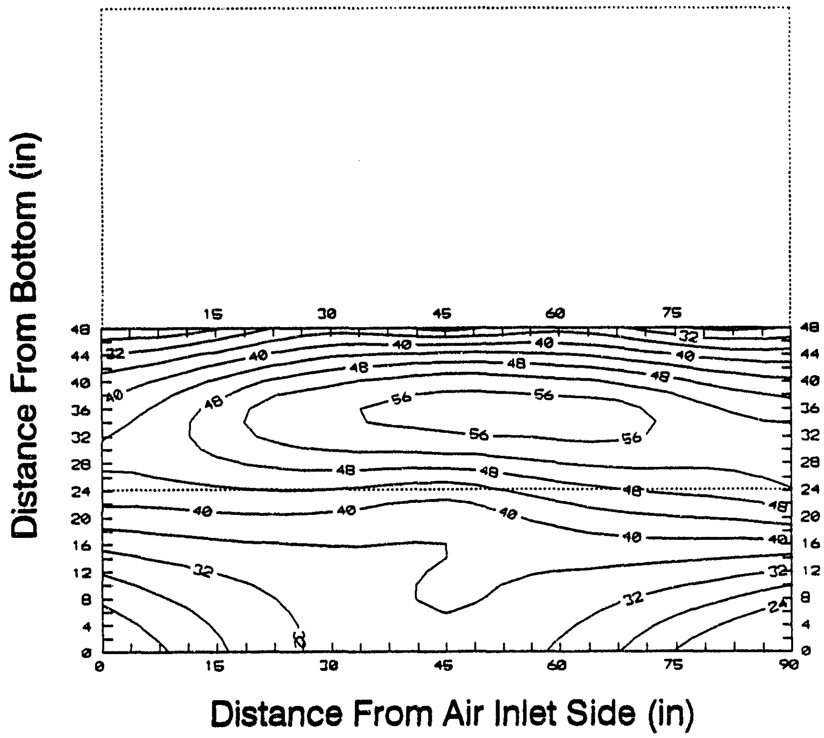
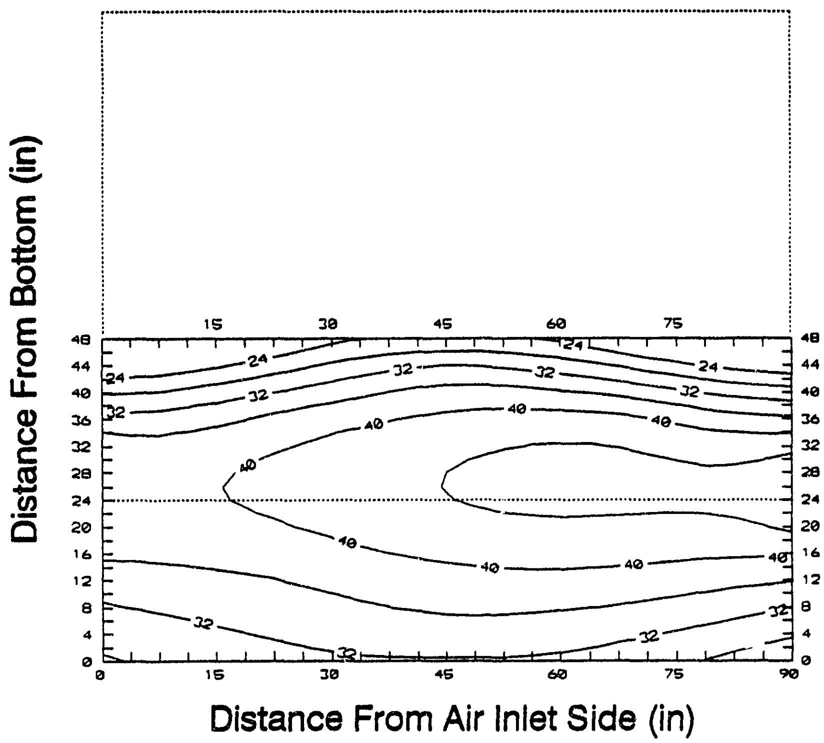


FIGURE 6.20. Peak Temperature Profile for Lift 2
(Centerline Temperatures at t = 195 hours)



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(Centerline Temperatures at t = 332 hours)

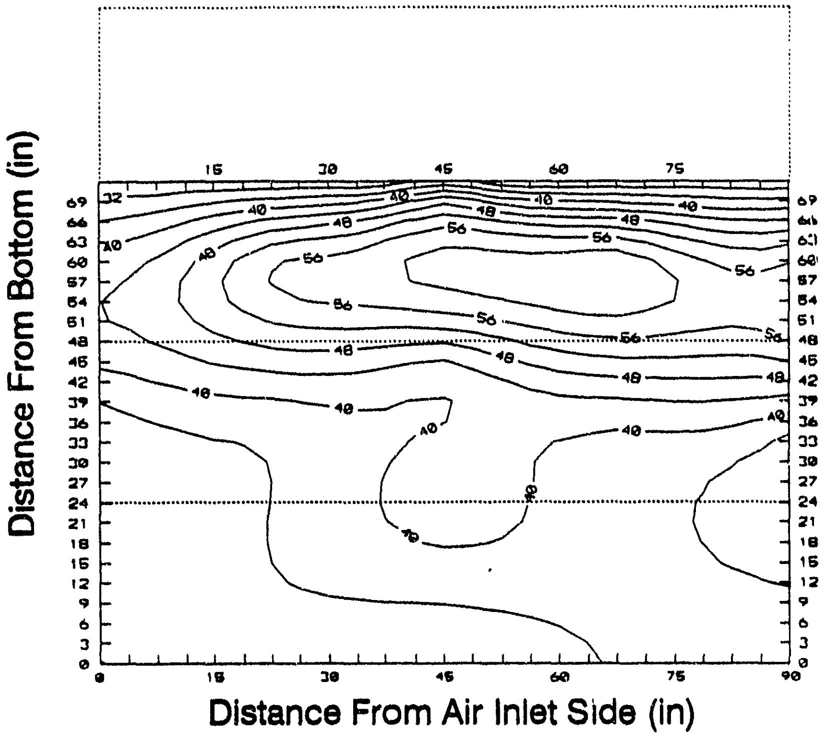
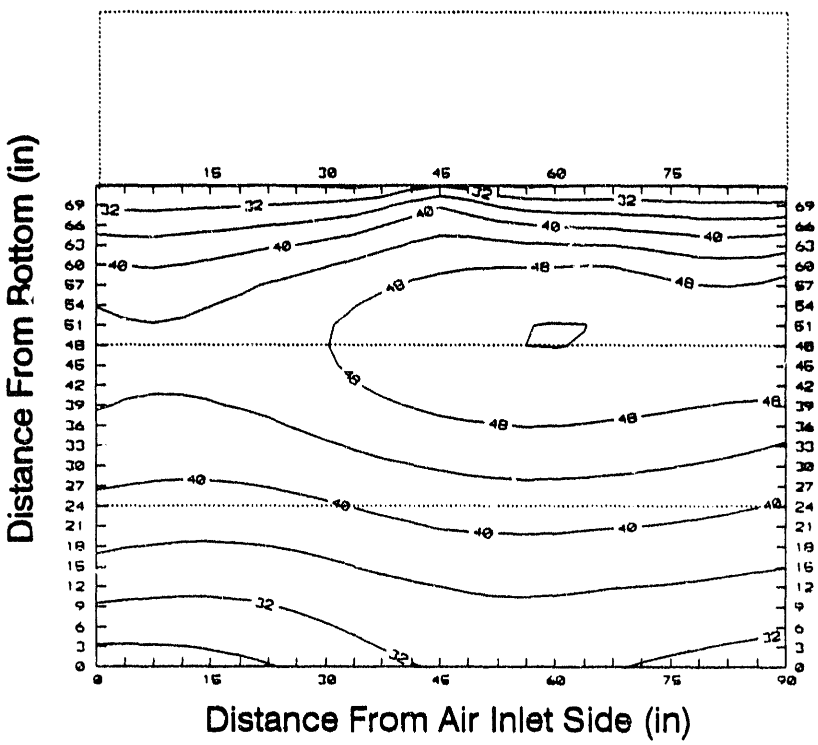


FIGURE 6.22. Peak Temperature Profile for Lift 3 (Centerline Temperatures at t = 365 hours)



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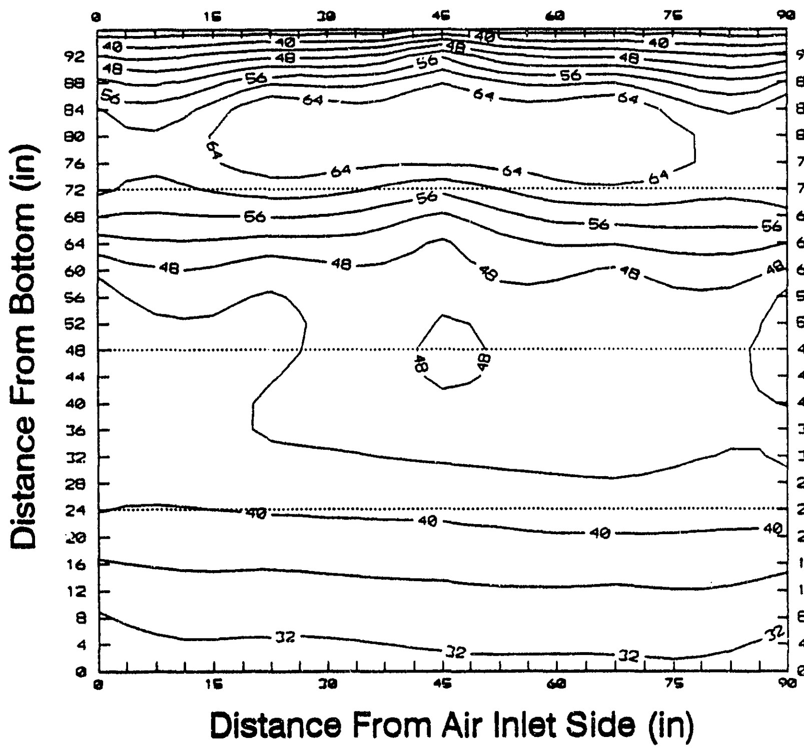

FIGURE 6.24. Peak Temperature Profile for Lift 4
(Centerline Temperatures at t = 535 hours)

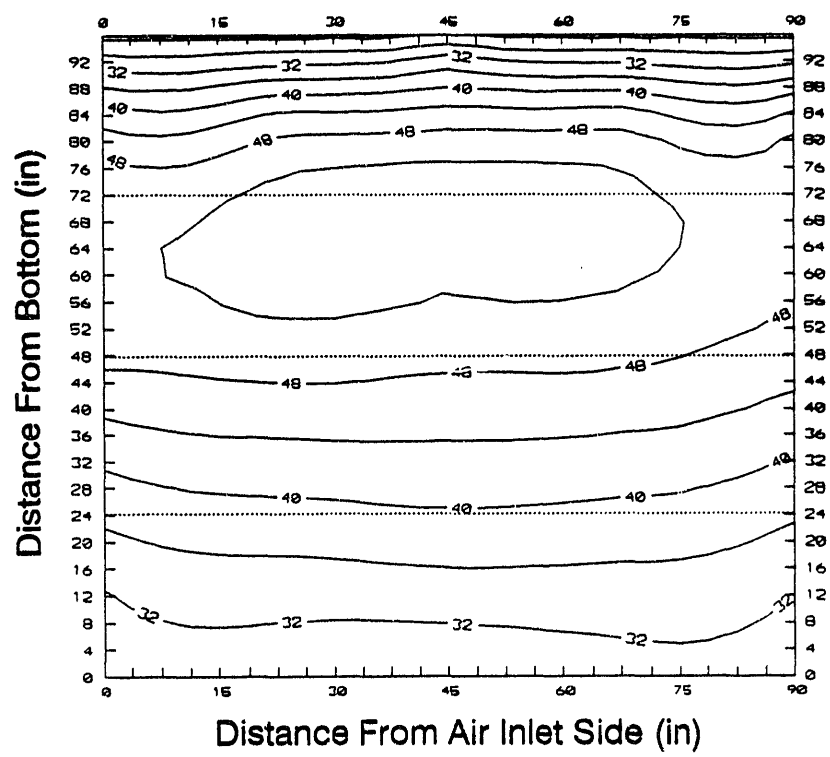

FIGURE 6.25. Temperature Profile After Cooling Lift 4 for 1 Week
(Centerline Temperatures at t = 668 hours)

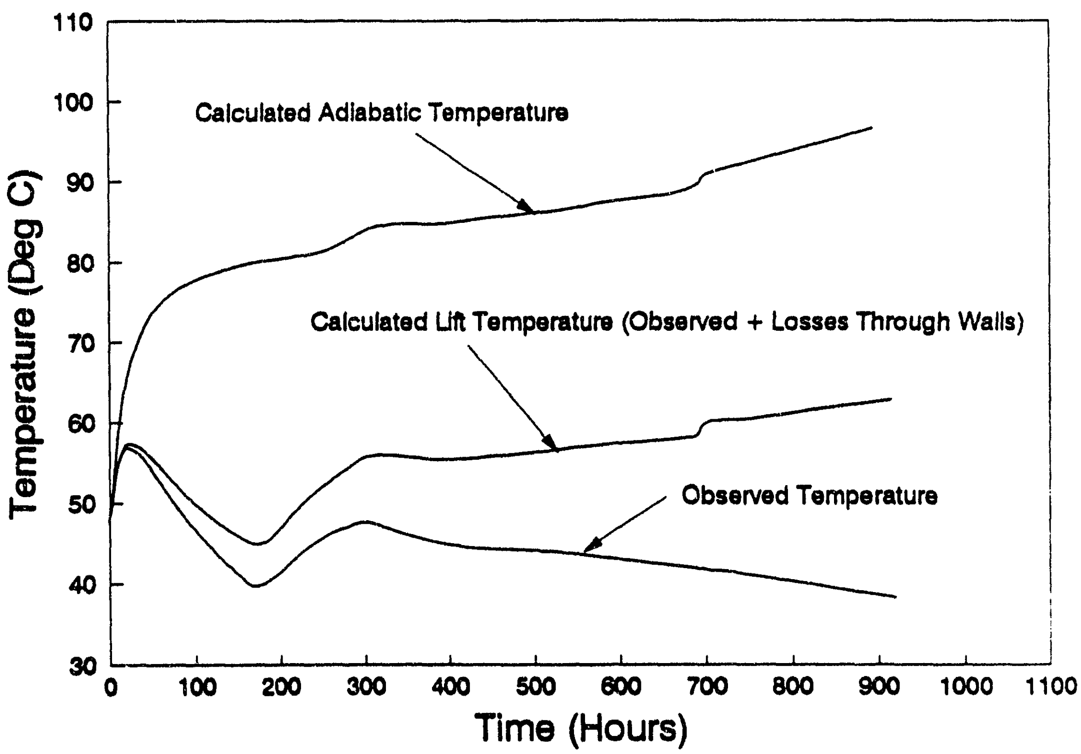

FIGURE 6.26. Estimated Long-Term Temperature Profiles for Lift 2

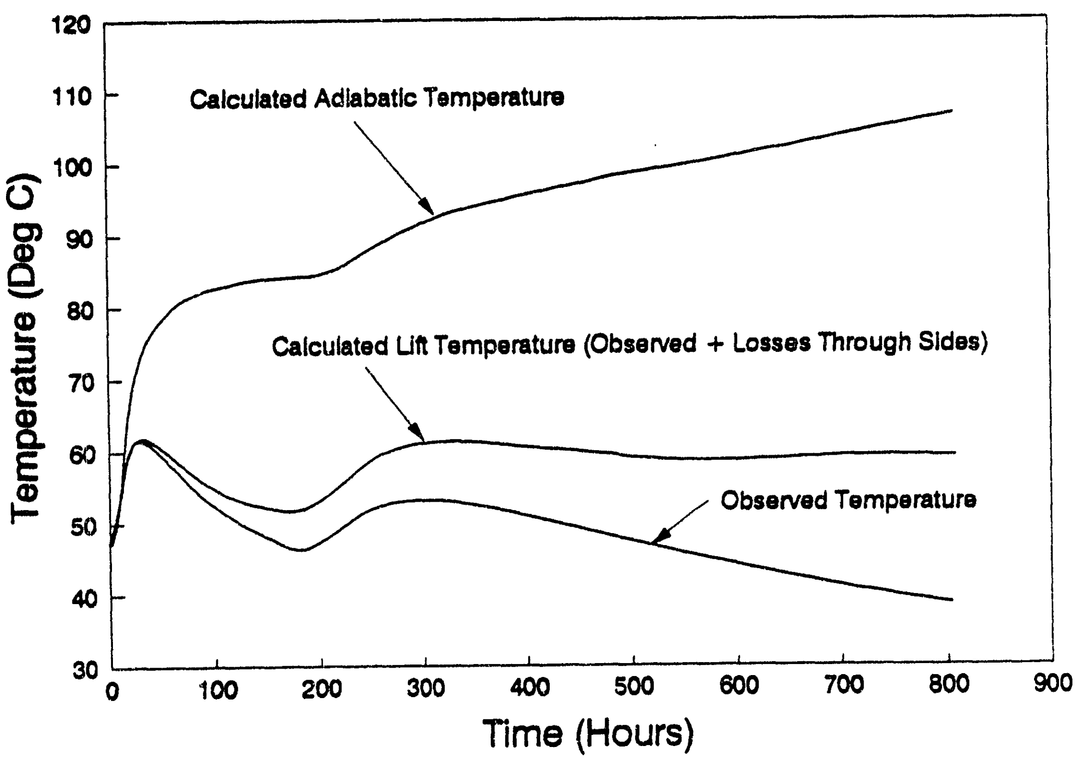

FIGURE 6.27. Estimated Long-Term Temperature Profiles for Lift 3

7.0 POST RUN EQUIPMENT OBSERVATIONS

Three weeks after completion of the fourth lift, the grout equipment was disassembled and inspected for grout buildup and mixer wear.

7.1 GROUT BUILDUP

The grout buildup in the equipment was the result of three aborted runs on the first day that grout was processed, the main grout pour 2 days later, and three smaller lift pours spaced at 1-week intervals. The pump restart tests were also conducted prior to inspection. At the conclusion of each of these runs, 25 gal of water was used to rinse the mixer, surge tank, grout pump, and grout pipe. After this water was pumped to the mold or dumpster, an additional 15 gal of water was circulated through the mixer, surge tank, and grout pump for 10 minutes. This water was then pumped to the dumpster. No attempt was made to remove the water that remained in the grout pipe.

The grout buildup in the mixer occurs in several different locations starting at the dry-blend inlet and continuing throughout the mixer. Figure 7.1 shows a large amount of grout buildup in the rectangular portion of the dry-blend inlet. Grout buildup was also noted in the dry-blend inlet line from the shaker screen (see Figure 7.2). While operating, these areas are not exposed to moisture, and grout should not form. However, rinses of the dry-blend inlet might introduce water that can form grout in subsequent runs. In addition, during the waste feed interruption, which caused an unscheduled shutdown during the main production run, wetted material had backed up into the dry-blend feed tube. This may have been when the grout in the rectangular area of the dry-blend inlet formed. The buildup in the dry-blend inlet line was the only area that had to be cleaned between runs to prevent grout production problems.

Figure 7.3 shows a thin layer of grout buildup on the lid of the mixer. This layer shows the tolerance between the mixer paddles and the mixer lid. Grout buildup in this location is probably not an operational concern because large pieces that fall off during production will be reduced to a small enough size to pass through the pump. However, this area may be difficult to decontaminate.

Figures 7.4 and 7.5 show the interior of the mixer. The paddles under the waste inlet are very clean, while the other paddles and the dry-blend feed screws have patches of grout. This implies that direct impingement of water will clean off the uncured grout, while other portions, which are simply wetted with water, will not be cleaned. Again, these patches of grout pose no problems during production but may be difficult to decontaminate.

Buildup in the grout discharge funnel is shown in Figure 7.6. In areas where grout flow is continuous, there is little buildup. Areas that are exposed to only splashed materials have a significant buildup. The discharge is designed differently in the production facility, and the pattern of grout buildup will probably be different.

Figures 7.7 and 7.8 show that no large areas of grout buildup occur in the surge tank but that a thin layer of material coats most of the interior surface. The clean areas on the surge tank lid again show that direct impingement by the water spray will remove the unsolidified grout. The spray nozzle used in the surge tank clogged easily and generally did a poor job of rinsing the surge tank. An improved spray nozzle design could reduce the buildup in the surge tank. The grout retained in the surge tank should not present an operational problem but may be difficult to completely decontaminate.

Figure 7.9 shows the grout buildup at the pump inlet. The design of the pump inlet has a dead spot that traps grout at the completion of a run. Water rinses removed enough grout to prevent operational difficulties, but this area will require decontamination.

Figure 7.10 shows the small amount of grout buildup at the pump outlet. This buildup is where stagnant grout would tend to settle and probably occurred after allowing grout to sit in this area during the restart tests. Therefore, grout buildup in this area during normal operations would probably not be a concern.

The initial 25 gal of rinse water used to flush the systems amounted to approximately 7.5 pipe volumes. Seven and one half pipe volumes of rinse water in the production system would equate to approximately 600 gal of water. At the end of the 25-gal rinse, the water at the pipe discharge was fairly clean and was probably adequate. However, since the grout pipe had to be used for four separate runs, the 15 gal of recirculated rinse water was also pumped through the grout pipe 15 minutes later.

A different procedure was used for the final flush of the grout pipe. Instead of immediately flushing the pipe with 25 gal of water, the 25 gal of rinse water was first recirculated through the grout equipment for 10 minutes before the pipe rinse was conducted. Thus, the total rinse water used for the final rinse was 7.5 pipe volumes of water.

Previous pilot-scale runs had encountered problems when attempting to reuse the grout pipeline. However, the proposed rinse at that time was only 2 to 3 pipe volumes of water. No problems were encountered when the grout pipe was reused four times for these tests, so the 40-gal flush (11.2 pipe volumes) was adequate. This rinse was also adequate to prevent buildup in the three-way valves used to divert the grout flow.

The last rinse used 7.5 pipe volumes of water. Three weeks after the completion of the last rinse, the interior of the grout pipe was examined at several points along its length. No buildup of grout was observed.

7.2 MIXER WEAR

Significant wear on the first set of mixer paddles and the dry-blend feed screws was observed in previous pilot-scale pours (Fow et al. 1987). For the current pilot-scale tests, dry-blend feed screws were replaced with stellite feed screws and the first four pairs of mixer paddles were replaced with stellite-tipped paddles. Since the production mixer has stellite components, the wear seen in this pilot-scale test should be more representative of the wear that might be expected in the production equipment. Figure 7.5 shows that there is little wear on either the first paddle or the feed screws. This indicates that stellite components reduce wear concerns.

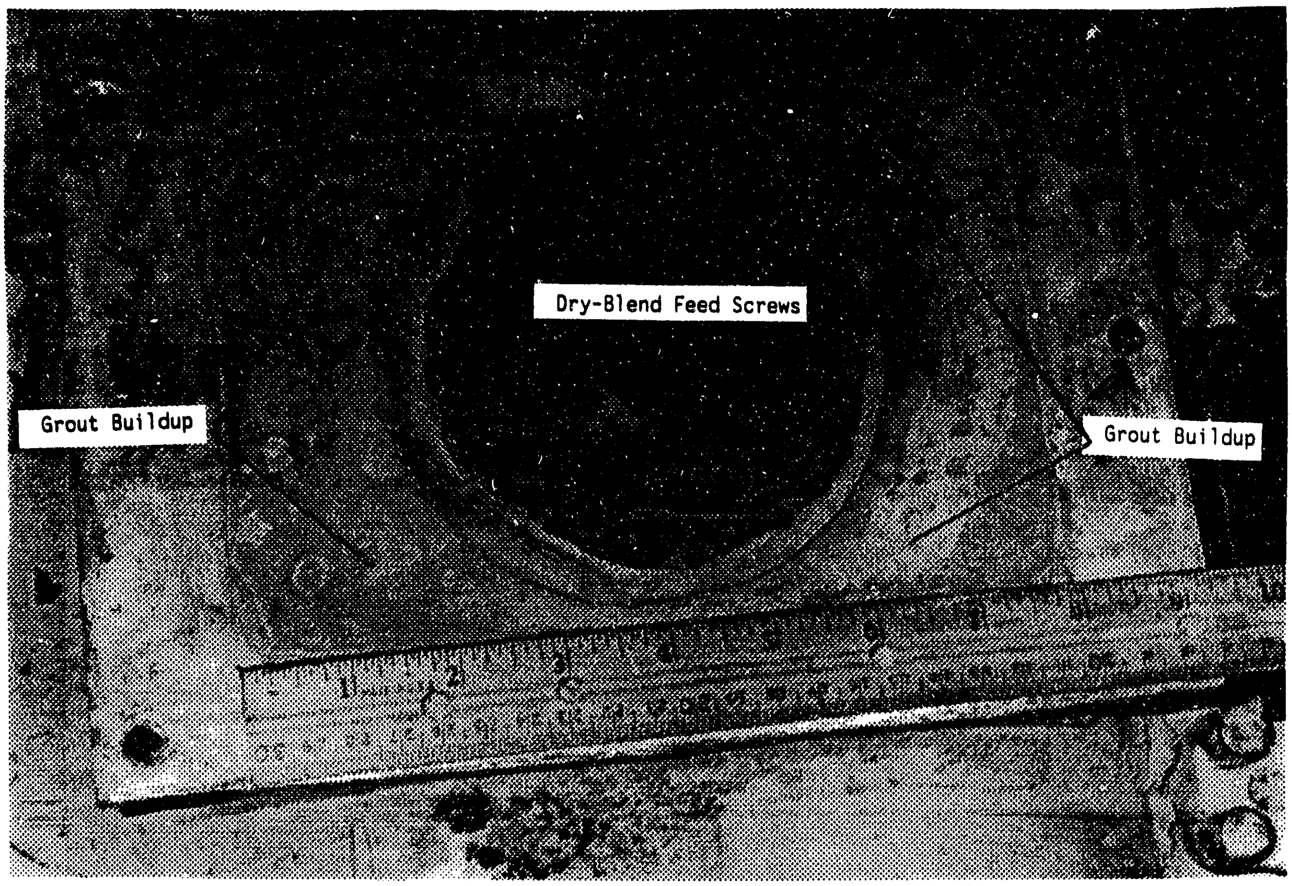

FIGURE 7.1. Grout Buildup at Mixer Inlet

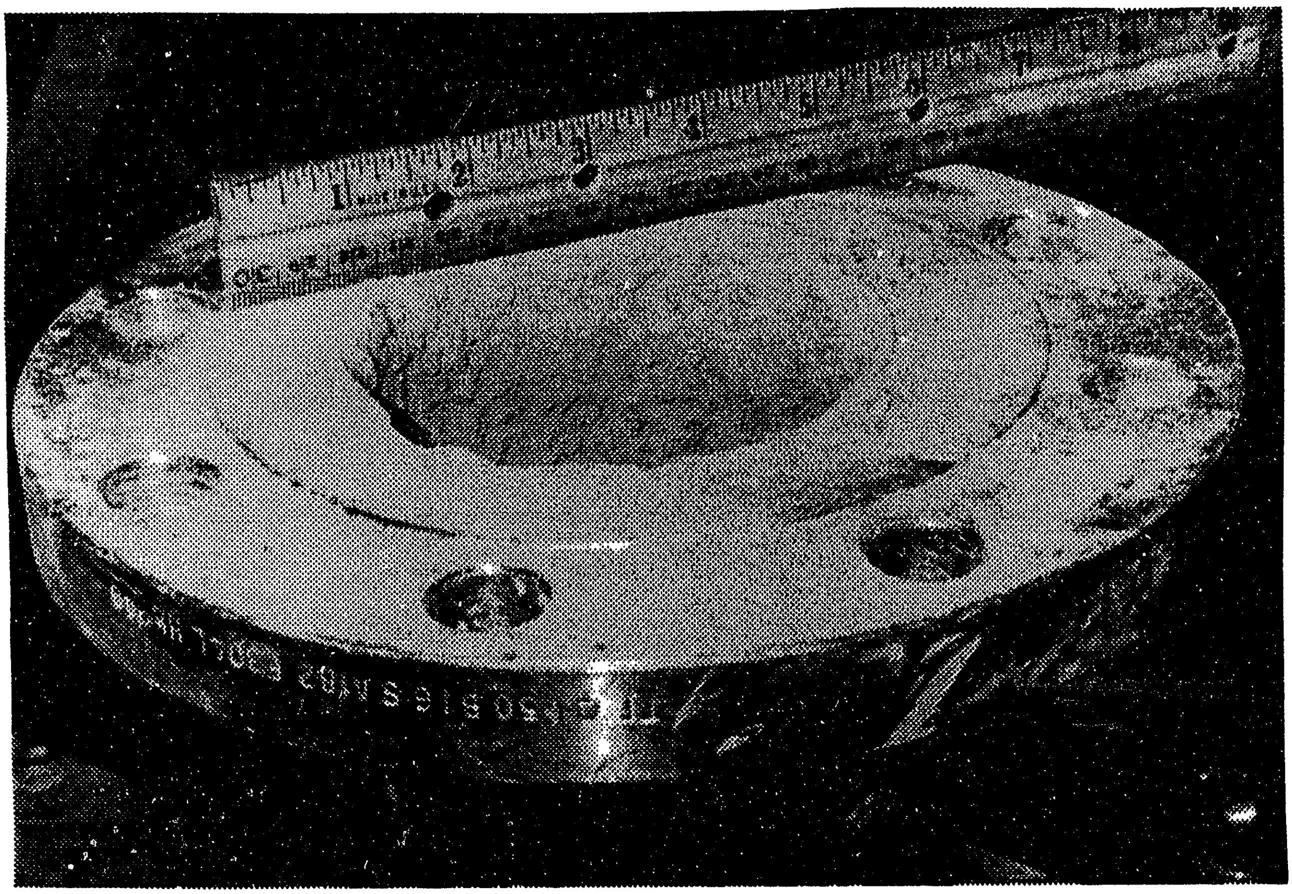

FIGURE 7.2. Grout Buildup in Dry-Blend Inlet Line

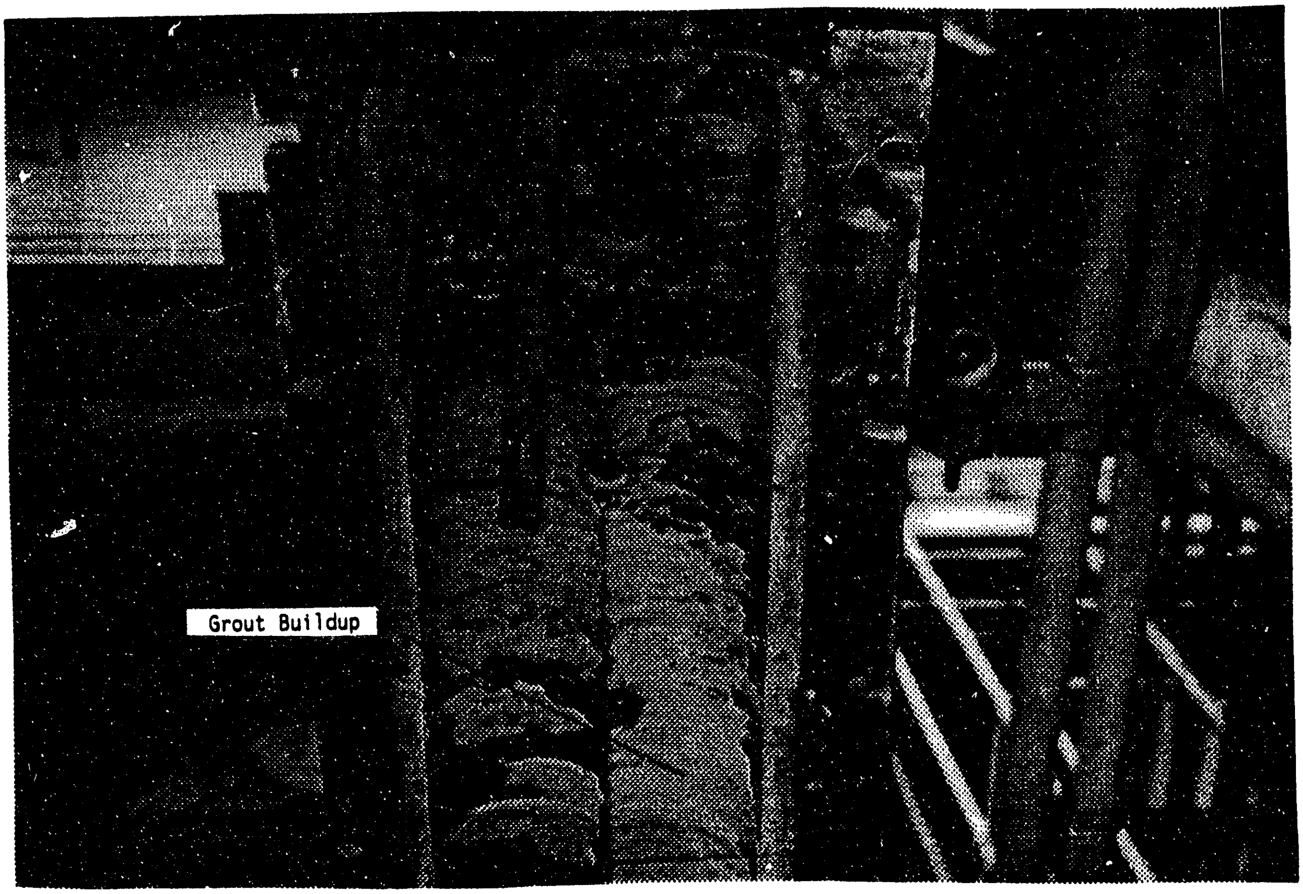

FIGURE 7.3. Grout Buildup on Mixer Lid

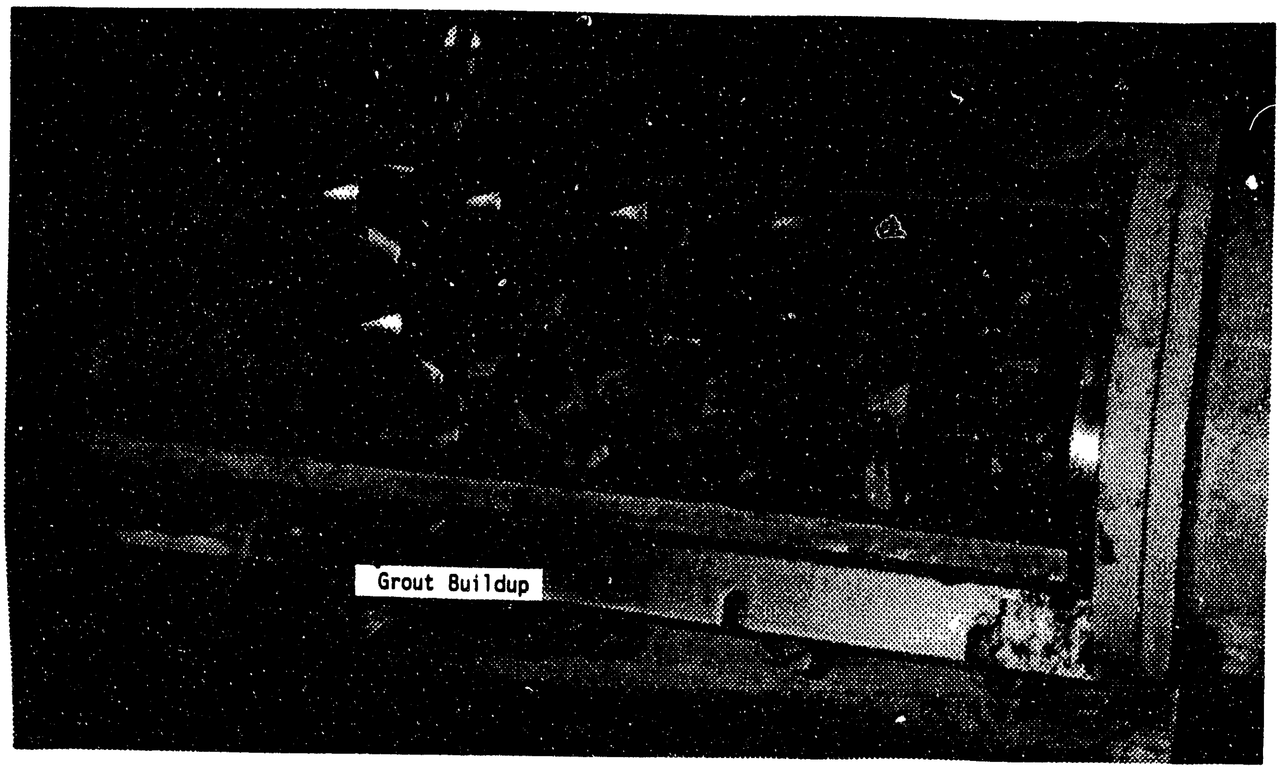

FIGURE 7.4. Grout Buildup on Mixer Paddles

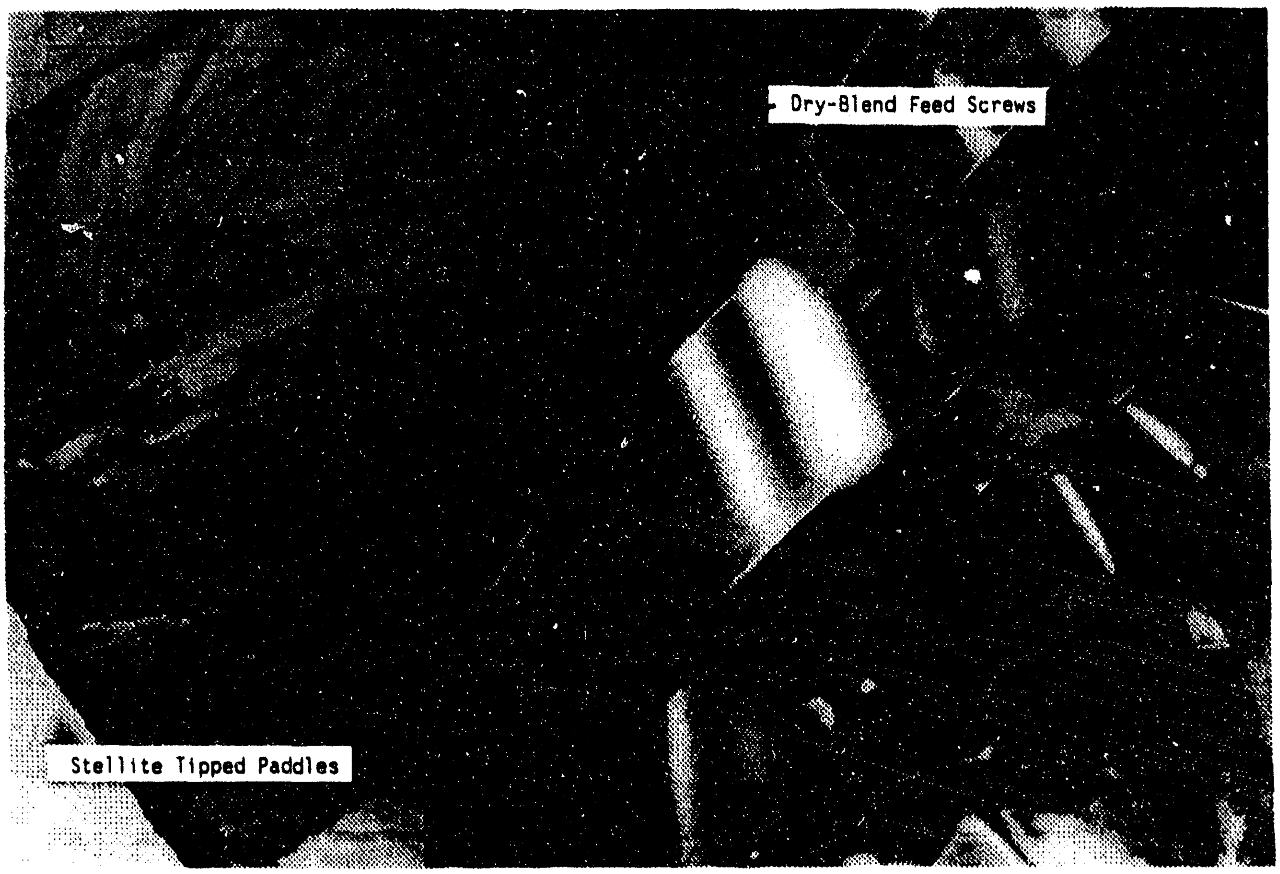

FIGURE 7.5. Stellite Tipped Mixer Paddles Under Waste Feed Inlet

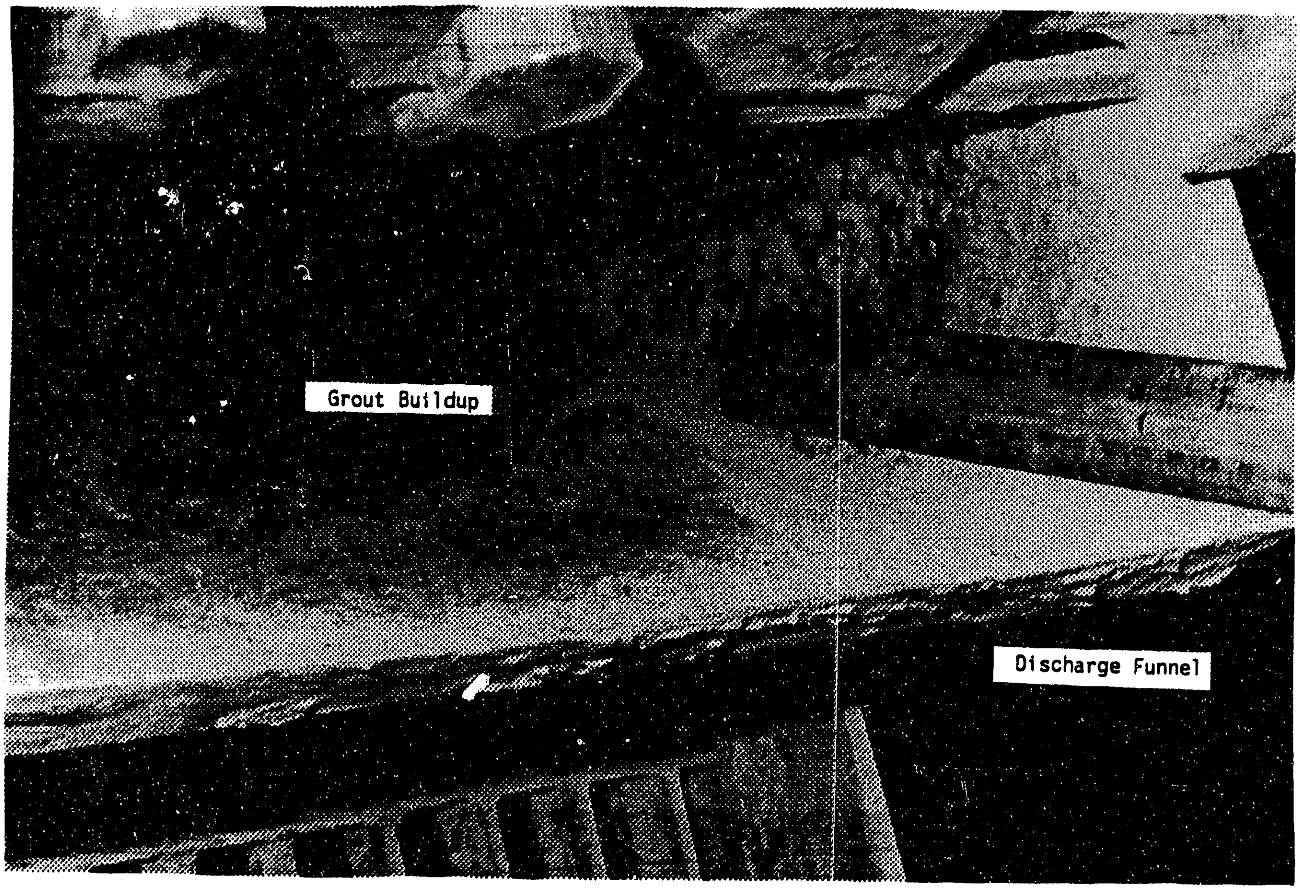

FIGURE 7.6. Grout Buildup at Mixer Discharge

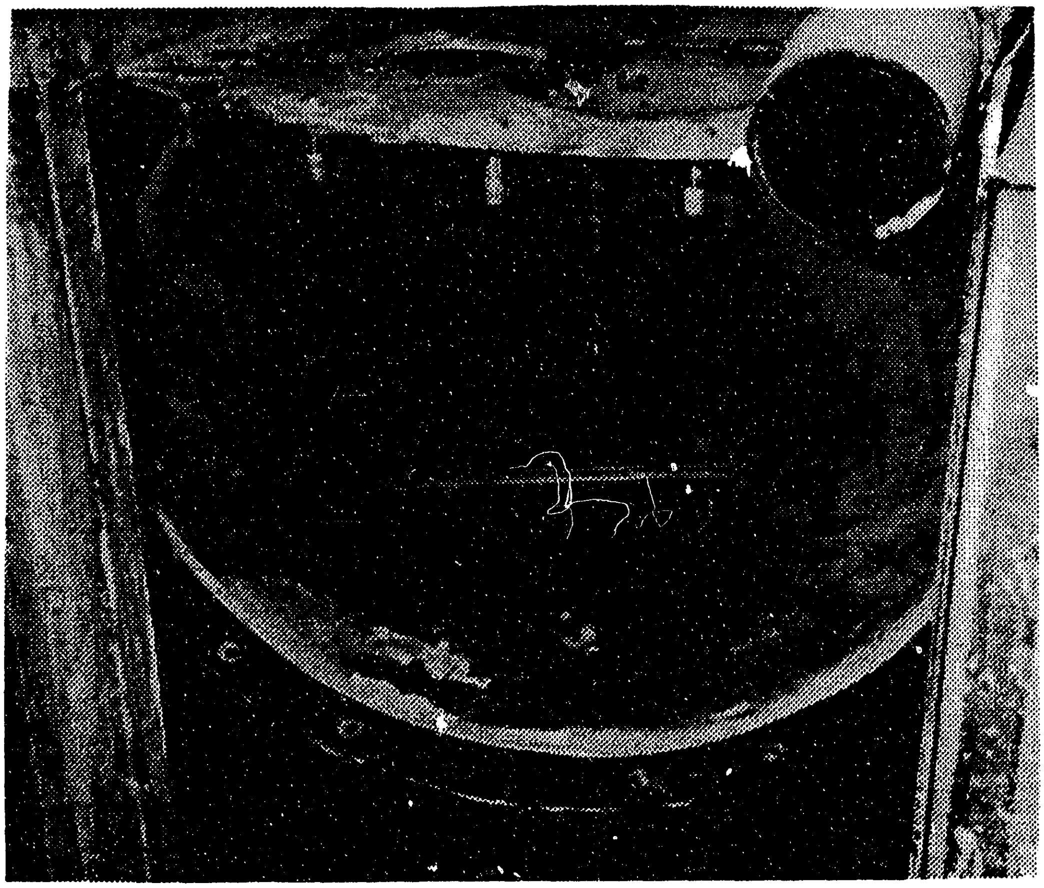

FIGURE 7.7. Grout Buildup in Surge Tank

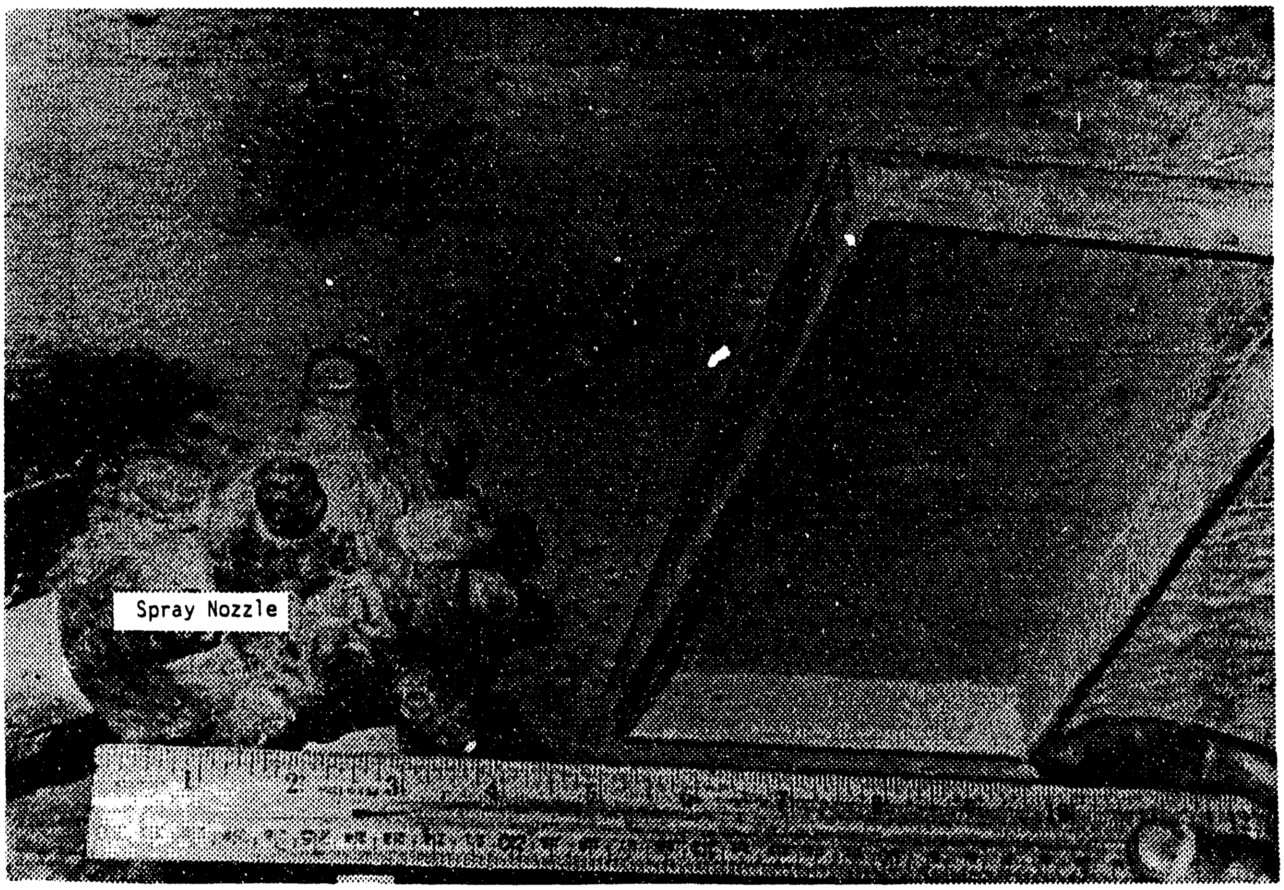

FIGURE 7.8. Grout Buildup on Surge Tank Lid

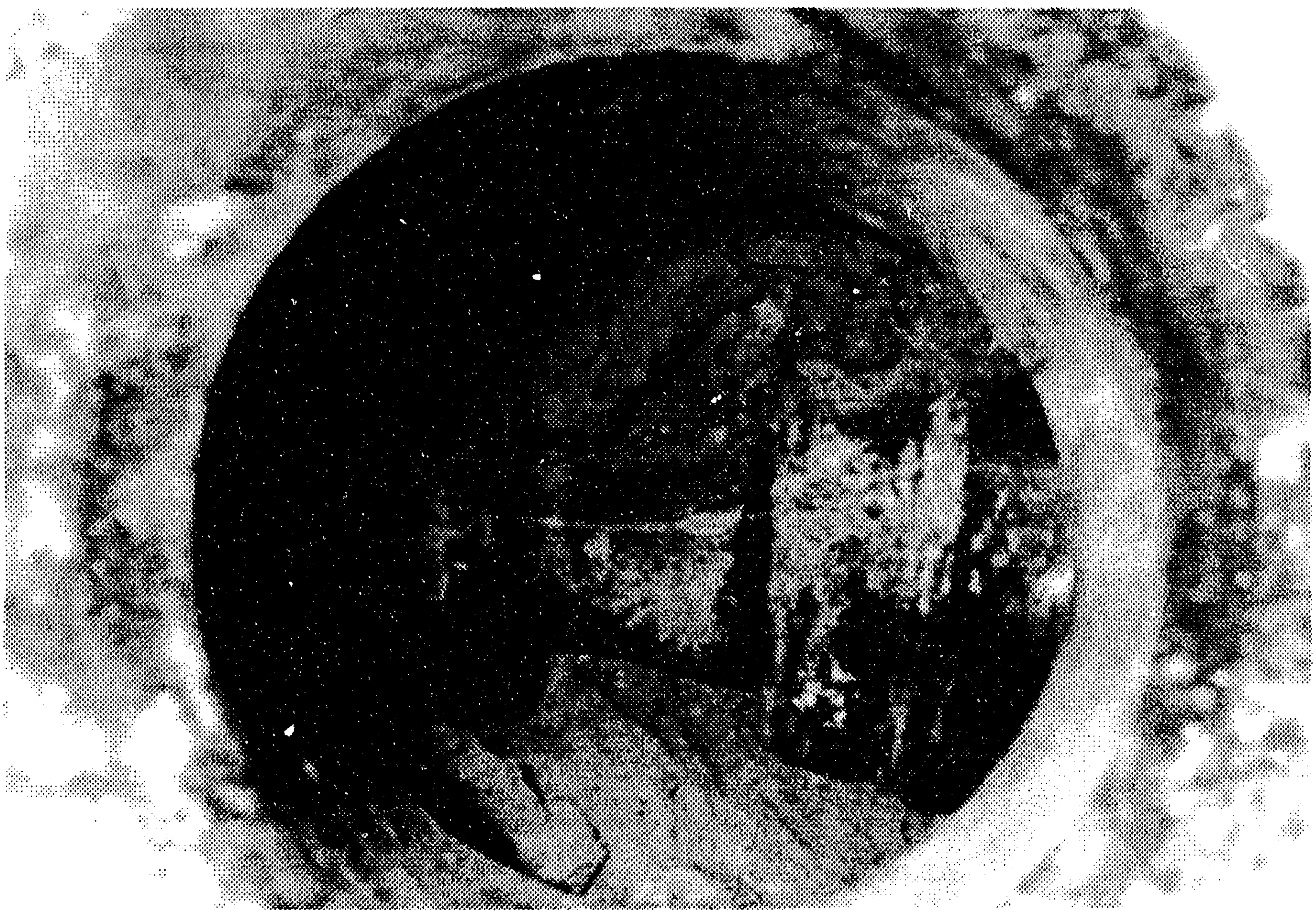

FIGURE 7.9. Grout Buildup at Pump Inlet

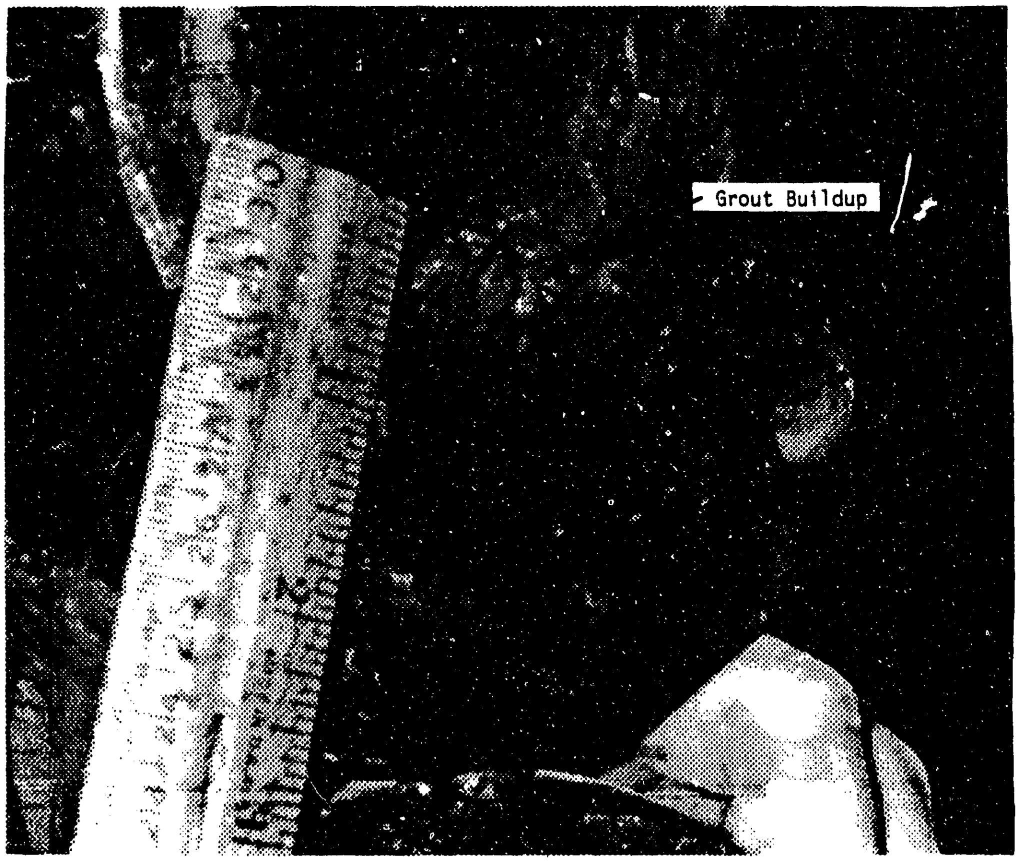

FIGURE 7.10. Grout Buildup at Pump Discharge

8.0 CONCLUSIONS/RECOMMENDATIONS

The following conclusions and recommendations are based on the results of the pilot-scale tests.

8.1 CONCLUSIONS

The pilot-scale testing satisfied the original objectives. The conclusions drawn from the testing are listed below:

- The grout produced with a dry-blend formulation consisting of 14 wt% attapulgite clay, 20 wt% cement, and 66 wt% class F fly ash showed significant shear thickening and had calculated critical flow rates at the pipe discharge that were above the criterion value of 60 gpm. Slight modification of the dry-blend formulation to 11 wt% attapulgite, 20.7 wt% cement, and 68.3 wt% class F fly ash reduced the critical flow rate to below 40 gpm. Other than the critical flow rate concerns, both formulations tested were readily processed by the pilot-scale equipment.

- The DMF handled the dry ingredients of the proposed production formulation and mixed dry-blend product within the desired tolerances.

- The restart pressure tests showed that process interruptions as long as 20 minutes did not pose a problem for the pilot-scale equipment. These tests indicated that interruptions of 30 minutes or greater should not be allowed without flushing the system.

- No significant wear was seen on the stellite feed screws and stellite-tipped paddles installed in the grout mixer.

- A 7.5 pipe-volume flush of the pilot-scale grout pipe at 10 gpm was sufficient to prevent buildup.

- Grout buildup in the equipment was similar to that seen in other pilot-scale runs. Buildup in the area of the dry-blend mixer inlet was a concern and may have interfered with grout production if it had not been cleaned between runs. Buildup in other areas did not interfere with grout production but might present decontamination problems.

- The dimensional changes of the grout over the first 7 weeks of curing were small (0.06% shrinkage).

- The thermal conductivity of this grout formulation was 0.81 W/m°K.

- Neither the original 14 wt% attapulgite clay formulation nor the modified 11 wt% attapulgite clay formulation had free liquids when poured at 40°C.

- The calculated adiabatic temperature rise of the grout poured in the gradient mold was 57°C.

- Comparing calculated heat conduction rates through grout to the experimentally-determined airflow heat removal rates from water/salt solutions showed that conduction of heat through the grout controls the heat removal rate when using the airflow rates planned for the production vault. As a result, increased airflows (e.g. larger blowers) would not significantly increase the heat removal rates.

- An airflow of 13 scfm in the lift mold (which simulated a 3600 scfm airflow in the production vault) kept the maximum short-term grout temperatures below 70°C for all four of the 2-ft lifts poured and maintained average grout surface temperatures below 30°C.

- The net temperature reduction obtained by cooling the surface of a 2 ft lift for 1 week was approximately 30°C.

- Heat removal rates throughout the week between pours were not significantly different for lifts with and without free-standing liquid.

- The lift mold thermal profiles after 1 week of cooling showed a general tendency, as lifts were added, for the peak temperatures to be higher and located farther below the surface with each subsequent lift.

8.2 RECOMMENDATIONS

The following are recommendations based on the results of the pilot-scale tests.

- A grout formulation of 11 wt% attapulgite clay, 20.7 wt% cement, and 68.3 wt% class F fly ash mixed at 8.4 lb/gal should be used for verification tests. This slightly modified formulation eliminates the critical flow rate concerns that were discovered while processing the 14 wt% attapulgite formulation. However, since this formulation has the same relative amounts of cement and fly ash as the original formulation, information obtained from grout produced with the first formulation should still be representative of that expected for the adjusted formulation.

- The grout surface should not be flooded with 1 to 2 in. of cooling water at the completion of the run. Flooding the surface affects the short-term curing of the grout at the surface of the lift and is not necessary for effective cooling. There is probably sufficient water in the grout pour solution to supply all the evaporation water, but tests that investigate the water-vapor pressure over the grout as the pore water evaporates would help determine if additional evaporation water is necessary. If additional evaporation water is necessary, it should be added after the grout has cured for several days.

- It is difficult to suggest a pour schedule from the information obtained from the lift mold tests, but several useful observations were made that helped direct the modeling efforts. 1) When using the planned airflow rates, the heat removal from the grout is mainly controlled by the rate at which heat will conduct through the grout to the surface. As long as water is available, convective/evaporative cooling can be expected to keep the grout surface temperature below 30°C. 2) The lower grout temperatures, which result due to surface cooling, may also reduce the hydration reaction rates. This is an important factor in determining the pour schedule since hydration heat released while the grout is close to the surface is relatively easy to remove, but heat released from grout at significant depths can only be removed through much slower heat-release mechanisms. In order to model these effects, the heat release as a function of temperature and extent of reaction must be available. Calorimetry work should be conducted on the proposed formulation to generate the required data. 3) The total hydration heat determined in this experiment was 5100 Btu/Cubic Foot. This number is important in estimating the long-term temperature profiles in the grout and the final pour schedule. Calorimetry work should be performed to confirm this number.

- Future pilot-scale tests should use initial dry-blend active hopper settings of 90% for the high level and 70% for the low level to avoid dry-blend flooding problems.

- The shear-thickening effects of attapulgite should be studied in greater detail if this material is part of future formulations.

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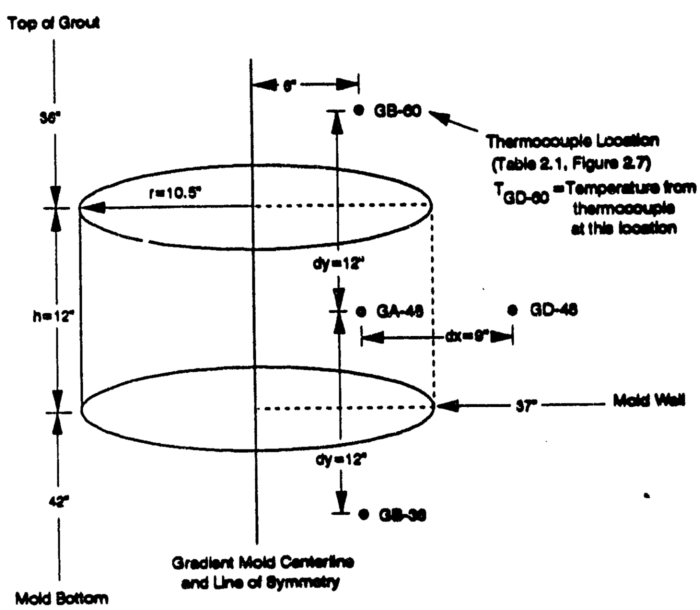
APPENDIX

ADIABATIC TEMPERATURE RISE CALCULATION FOR GRADIENT MOLD

APPENDIX

CALCULATION OF ADIABATIC TEMPERATURE RISE FOR GRADIENT MOLD

Heat Balance Geometry



Assumptions:

(1) Approximate thermal gradients as linear

(2) Temperature profile is symmetric with respect to vessel centerline

(3) Grout Heat Capacity - 0.527 cal/g°C

(4) Grout Thermal Conductivity - 0.81 W/m°K

(5) Grout Density - 1.55 g/cm^3

Heat balance for cylinder shown above:

TOTAL HEAT LOSS = HEAT LOSS THROUGH SIDES + HEAT LOSS THROUGH TOP + HEAT LOSS THROUGH BOTTOM

$$HEAT\ LOSS = Q = \left(\frac{dT}{dx}\right) A K_{GROUT} \Delta t$$

Where dT/dx = thermal gradient

A = heat loss area corresponding to thermal gradient

Δt = heat loss time period

K_{GROUT} = grout thermal conductivity

$$Q_{TOTAL} = Q_{SIDES} + Q_{TOP} + Q_{BOTTOM} \quad (1)$$

$$Q_{SIDES} = \left(\frac{T_{GA-48} - T_{GD-48}}{dx} \right) (2\pi r h) K_{GROUT} \Delta t$$

$$Q_{SIDES} = 0.4326 \left(T_{GA-48} - T_{GD-48} \right) \Delta t \quad cal \quad (2)$$

$$Q_{BOTTOM} = \left(\frac{T_{GA-48} - T_{GB-36}}{dy} \right) (\pi r^2) K_{GROUT} \Delta t$$

$$Q_{BOTTOM} = 0.1419 \left(T_{GA-48} - T_{GB-36} \right) \Delta t \quad cal \quad (3)$$

$$Q_{TOP} = 0.1419 \left(T_{GA-48} - T_{GB-60} \right) \Delta t \quad cal \quad (4)$$

Combining Equations 1-4 gives:

$$Q_{TOTAL} = \left(0.4316 \left(T_{GA-48} - T_{GD-48} \right) + 0.1419 \left(2T_{GA-48} - T_{GB-36} - T_{GB-60} \right) \right) \Delta t \quad cal$$

where Δt is in seconds

$$\textit{THEN:}\quad T_{ADIABATIC} = T_{GROUT} + \frac{Q_{TOTAL}}{\textit{THERMAL MASS OF GROUT}}$$

$$T_{ADIABATIC} = T_{GA\text{-}48} + \frac{Q_{TOTAL}}{\rho_{GROUT} Cp_{GROUT} \left(\pi r^2 h\right)}$$

Where ρ_{GROUT} = Grout density

Cp_{GROUT} = Grout heat capacity

The adiabatic temperature at time = t is given by:

$$T_{ADIABATIC_t} = \left(T_{GA\text{-}48_t} + \frac{Q_{TOTAL_t}}{55{,}636}\right) {}^\circ C$$

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