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**LABORATORY EVALUATION OF UNDERWATER GROUTING OF CPP-603  
BASINS**

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**ABSTRACT**

A project is underway to deactivate a Fuel Storage Basin. The project specifies the requirements and identifies the tasks that will be performed for deactivation of the CPP-603 building at the Idaho Nuclear Technology and Engineering Center of the Idaho National Engineering and Environmental Laboratory. The Fuel Receiving and Storage Building (CPP-603) was originally used to receive and store spent nuclear fuel from various facilities. The area to undergo deactivation includes the three spent nuclear fuel storage basins and a transfer canal (1.5 million gallons of water storage). Deactivation operations at the task site include management of the hot storage boxes and generic fuel objects, removal of the fuel storage racks, basin sludge, water evaporation and basin grouting, and interior equipment, tanks, and associated components. This includes a study to develop a grout formulation and placement process for this deactivation project. Water will be allowed to passively evaporate to reduce the spread of contamination from the walls of the basin. The basins will be filled with grout, underwater, as the water evaporates to maintain the basin water at a safe level. The objective of the deactivation project is to eliminate potential exposure to hazardous and radioactive materials and eliminate potential safety hazards associated with the CPP-603 building.

**INTRODUCTION**

The CPP-603 Nuclear Fuel Storage Basins are located at the Idaho National Engineering and Environmental Laboratory (INEEL). This site is an 890-square mile reservation under the direction of the Department of Energy (DOE) and located in the desert of southeastern Idaho about 32 miles west of Idaho Falls, Idaho. The CPP-603 building was built in 1951 and contains three storage basins connected by a transfer canal, making it an E-shaped set of pools.

To comply with the 1995 settlement agreement with the state, the spent fuel was moved to more modern storage facilities. This was accomplished in April 2000.

The south basin #1 is the largest of three basins, which has dimension of approximately 42'x80', and the middle basin #2 and north basin #3 each has dimension of 38'x59'. All three basins have pool depth of 21 ft, and some of the basins have underwater rack supporting channels. Table 1 shows the major components and concentrations of CPP-603 basin water.

The present plans to deactivate, dismantle, and decommission this part of the facility started in 2001. In the year 2003, the basin water is to be evaporated naturally and replaced periodically with grout. The water level is maintained to prevent radioactivity on the walls of the basins from being exposed, flaking off, and becoming airborne. The evaporation and filling with grout will last approximately eight years in order to get the necessary funding for the grout, to allow for natural evaporation, and to maintain the basin in a radiologically safe environment.

### **Objective of Investigation**

The objective of this investigation was to perform laboratory studies to evaluate the underwater grouting process of CPP-603 basins. The specific objectives of the study were as follows:

1. Determine whether the selected grout mixture will cure/stabilize without significant thermal energy release and will cure when prepared using the concentrated CPP-603 basin water.
2. Determine the feasibility of underwater injection of the recommended grout mixture with the simulated concentrate.

### **EXPERIMENTAL**

The following section describes the material and methods used in this investigation.

#### **Materials**

##### CPP-603 Basin Water Concentrate

The basin water concentrate (at the volume reduction of 20 to 1) was used for this study as a worst-case final grout pour. To prepare the concentrate from a simulated non-radioactive concentrate solution with the following concentration was used:

- fluoride-3.2 PPM
- chloride-1147 PPM
- nitrate-3500 PPM
- sulfate-360 PPM
- sodium-1847 PPM
- calcium-320 PPM

This simulant was used to prepare the grout for underwater pouring. The use of a simulant eliminated contamination and minimized radioactive waste.

##### Grout Mixture

The grout mixture was prepared using either the CPP-603 basin water concentrate, the simulated solution, or water. The composition of the recommended grout formula for

evaluation is presented in Table 2. For each laboratory application, a 1200-gram batch with the chemical additives was used. The cured grout product has a volume of approximately 560 ml from a 1200-gram batch.

**Table 1. CPP-603 Basin Water Composition**

Component	Units	Minimum	Maximum
Chloride	PPM	35	53
Nitrate	PPM	149	194
Sulfate	PPM	18	
Sodium	PPM	69	96
Calcium	PPM	13	18
Co-57	pCi/L	91	200
Co-58	pCi/L	N/A	
Co-60	pCi/L	26	860
Cs-137	pCi/L	18000	44000
Eu-152	pCi/L	120	1500
Eu-154	pCi/L	69	840
Sb-125	pCi/L	25	140
Sr-90+Y-90	pCi/L	26000	45000
Tritium	pCi/L	N/A	
Uranium	PPB	7.1	
pH		7.7	8.3
TDS	PPM	376	
Density	G/ml	1.0	

CPP-603 basin water, an initial sample analysis of the three basins was performed. It was determined that the solution from the floor location of North Basin #3 would be used for the heat release test. The selected sample solution has the following concentration:

- fluoride-.16 PPM
- chloride-57.3 PPM
- nitrate-175 PPM,

and a radiation level of <1 mR. Eight liters of this CPP-603 basin water was placed in an open container and evaporated naturally in a laboratory fume hood. Approximately 410 ml of concentrate was collected at the end of evaporation (a 20 to 1 ratio).

To prepare the wet grout mixture, the simulant or concentrated basin water was slowly added to the pre-mixed dry concrete ingredients (cement, fly ash, sand, and gravel) and mixed in a Kitchen-Aid® mixer for 5 minutes. A water reducer and anti-washout admixture were then added separately, and allowed 3-4 minutes each of mixing before adding the other additive. The water reducer improves the flowability, and pumpability

**Table 2. Composition of Concrete Mixture**

Recommended Formula		1200 gram laboratory batch	
Materials	Amount	Materials	Amount/Equivalent
Cement (Type 2)	320 lbs	Portland Type I/II Cement	110 g/320 lbs
Pozzolan Class F (fly ash)	440 lbs	Type F fly ash	150 g/440 lbs
Fine Aggregate (sand)	1200 lbs	Sand	408 g/1200 lbs
Large Aggregate (gravel)	1200 lbs	Crushed Gravel, Size 1/8"	408 g/1200 lbs
Water	375 lbs	Concentrated solution	128 ml/375 lbs
Mid-range water reducer	144 ounces	Polyheed 997 (Master Builders)	4.5 ml/216 ounces
Anti-washout admixture	80 ounces	Rheomac UW 450 (Master Builders)	2 ml/88 ounces

of the wet mixture. The anti-washout admixture prevents segregation of grout solids and liquids during underwater placement. However, anti-washout admixture negates the flowability property of the mixture. Additional water reducer (1.5 mL/72 ounces equivalent) was used in the 1200-gram laboratory batch to compensate for the agglomeration caused by the anti-washout additive effect in the mixture (Table 2).

## **Experimental Method**

### Heat Release Test

Two laboratory grout mixtures were prepared using water (test 1) and actual basin water concentrate (tests 2 and 3). The 1200-gram test mixture was transferred into two sample test vessels with approximately 600 gram of material in each test vessel.

The sample vessels were placed in the Styrofoam thermal insulator (Figure 1) and a thermo-couple was inserted into the grout sample mixture and connected to a data acquisition system. Data collection for sample temperature was started immediately thereafter. In addition to monitoring the sample temperature, ambient temperatures were also recorded as a reference.

### Bench Scale Underwater Grout Layer Test Procedures

Four liters of the concentrated simulant were placed into a 4"x10"x20" Lexan® test box (1/4" thick Lexan®). A batch (4800 gram) of grout mixture using concentrated simulant was prepared. The grout mixture was transferred into a 2 1/2" diameter smooth, straight clear plastic injection pipe. The grout was then placed underwater by slowly removing the capping plate, and injecting the grout through the injection pipe. The lower end of the pipe should be kept at the lowest point in the test box or buried in the fresh grout to

maintain a seal. These steps were repeated for two more layers. The solution pH and grout hardening were monitored for all the pours.

## **DISCUSSION**

### Heat Release Test

The chemical reaction between unhydrated cement and water during curing releases heat. This results in a rise in the temperature of the fresh grout. The rate and amount of heat generated are important for placing grout underwater at CPP-603 basins. A significant rise in the grout temperature may be undesirable for two reasons: (1) it could result in non-uniform cooling of the grout and create undesirable stresses, (2) the additional heat may cause volatility of some of the radioactive species in CPP-603 basin water. Calorimetry is a preferred and widely used tool in the study of the heat of hydration. Radiological concerns directed that the heat of reaction be monitored by the sample temperature changes (temperatures measured were obtained under non-adiabatic conditions). The initial temperature of the grout is very important in determining the heat produced by a particular grout. The hydration reaction is dependant on initial temperature, i.e. the colder the grout, the less rapid and intense the hydration reaction.

A total of three tests were performed with duplicate samples for each test. Deionized (DI) water was used to prepare the concrete mixes for the 1<sup>st</sup> test, and the actual, radioactive concentrated CPP-603 basin water (20:1) was used for the 2<sup>nd</sup> and the 3<sup>rd</sup> tests. Sample-to-sample variability in the temperature measurements is insignificant. Samples show an overall response delay versus ambient temperature, due to the Styrofoam thermal insulator. During the first 12 hours of testing, the rate of sample temperature increase was slower than the change of ambient measurements for all three tests (6 samples). Generally, the heat of hydration of the cement material, most of the heat release and the highest rate of heat release occur within the first 24 hours (Figure 2). Thus, it may be concluded that the total heat release and its impact from the grout tested should be minimal, especially, in the presence of a large quantity of CPP-603 basin water.

### Bench Scale Underwater Grout Layer Tests

The focus of this report is to explain the process to place the grout underwater and to evaluate a grout formulation that would flow and harden underwater. In concrete industry, methods for placing grout underwater include the following: injection pipe (tremie), bottom-dump buckets, preplaced aggregate, bagwork, and the diving bell. An injection pipe was used for this study. To secure flowable grout and minimize washout of the cement and fines during underwater placement, Polyheed 997 waster reducer and Rheomac UW450 (anti-washout) admixture was added to the grout mixture. In the concrete/grout industry, the slump test is the most universally used test. This test measures the consistency of concrete/grout. ASTM C 143 is the procedure used to do the slump test. The slump of concrete/grout is a good measure of the consistency and flow characteristics of a concrete/grout mixture. For underwater applications, it is desirable to have highly flowable grout that can resist water dilution, segregation, and spread readily into place. This equates to a mid range slump. A very high slump grout gives maximum

water dilution. A very low slump grout results in little or no flow characteristics. For underwater grout, the slump flow is influenced (in order of influence) by the anti-washout admixture concentration and the binder content, by the water-cementitious material ratio, and by water reducer concentration (ref. 1, 2). The slump flow corresponds to the mean base diameter when the grout is at the end of the slump test. The washout mass loss (ref. 3) is affected by (in order of importance) the anti-washout admixture concentration, binder content, water-cementitious material ratio, and the water reducer concentration.

The concentrated simulant was used to prepare the grout mixture (to simulate a worst case condition). A total of three grout layers (Figure 3) were periodically injected below 6" of solution into the box. Each layer of grout had an average thickness of 3 1/4", and the grout layer was allowed to cure for approximately 2 weeks before the next layer was placed on top of it. While injecting the grout underwater, the lower end of the injection pipe was lifted slightly and moved slowly along the bottom of the test box to allow the grout to flow out of the injection pipe.

The solution was cloudy during and after the grout injection, due to washout, and diffusion/mixing of some materials from the wet grout mixture. Depending on the extent of washout, the solution remained cloudy for several days before the suspended particulate settled and the solution turned clear. The simulant had pH of 7.95. After injecting the grout underwater, the solution pH after the first concrete layer was approximately 12.1; and increased to between 12.2 and 12.4 when the second and the third grout layers were injected. During the first week after the injection of the second concrete layer, a crack was developed on the test box (Figure 11) and solution leaked out at approximately 160 ml/day. DI water was added to the system every 3-4 days to make up for the lost amount. The cause of the resulting cracks on the test box is unknown. After each injection of grout, a small amount of materials floated on the solution surface. From the first and the third injections, the solution remained cloudy for 3-4 days and approximately 1/8" of light colored sediment material settled on top of the grout layer. This flocculent like material was soft, cream colored, and easy to move around. The solution from the second concrete layer remained cloudy for 2-3 days before it cleared. This grout layer had about a 1/16" thick layer of sediment (see figure 4). For each of the three-grout layers, the poured mixtures were mostly hardened within 48 hours.

Two items affected the amount of material washout into the solution. They are: the grout formulation and the procedure of injecting the grout. For underwater applications, the concrete industry recommends that the slump of the grout should not be less than 7 inches and it is important that the grout flow without segregation. However, in order to allow the material to spread and cover a larger area, a grout with high slump and self-leveling property is most desirable. The grout mixture that was used has adequate flowability, but the exact slump is unknown (slump equipment was not available). Greater flow ability and greater slump generally translate to a higher washout.

Please note that some voids are present at the left corner of all three concrete layers. These voids are probably due to failure of the mixture to settle well at the corner and/or the grout viscosity. Vibration is the most widely used method for compacting grout to

minimize voids or air entrainment. However, it is difficult to use the vibrator underwater and in a radioactive environment.

Two weeks after placing the 3<sup>rd</sup> concrete layer, the test box was disassembled and the grout layers were removed (Figure 4). All of the grout layers were easily separated, due to very weak cold joints. Results from the compression test are relatively consistent except one sample from the 1<sup>st</sup> layer; this sample probably had some defect (Table 3). The test results showed that the first layer of concrete is the strongest of the three layers; and the third layer is the weakest, due to the short curing time.

**Table 3. Results of Compression Test**

Concrete Layer	Cure Time (wk)	Compressive Strength (psi)
#1	6	5500, 3130, 5570
#2	4	4020, 3783, 3370
#3	2	2380, 2620, 2740

### **Operation Considerations**

In addition to the results from this laboratory study, some additional considerations were identified. A significant amount of planning is needed before implementing this process. Some of the variables/factors that could affect placing grout underwater at CPP-603 basins are discussed below:

1. **Grout Pumpability**  
The flowability/pumpability of the grout application will depend on the grout formula, the equipment, the distance to pump, and whether to maintain a seal during pumping. It is noted that when handling a large amount of the grout that lacks flowability (low slump), the backpressure could be very significant, especially if an underwater injection seal is needed to minimize washout.
2. **Extent of Grout Washout**  
While injecting grout underwater, it is very important to maintain a seal and to force grout to flow into position by pressure, to minimize washout of cement or fines. Otherwise, the amount of fine particulate flow into liquid solution could be very significant, which forms a thick layer of sediment.  
Delete extra line
3. **Voids in Grout**  
The grout mixtures were compacted well at most locations. However, further adjustment on the grout formula may be needed to minimize voids, and improve its compacting property. Some mechanical settling may be necessary.
4. **Curing of Grout**

In laboratory bench scale test, the grout mixture was cured/hardened in relative short duration. For low slump grout, it is very possible that a longer time will be required to cure the grout when a large pile of the material is placed underwater.

5. **Cold Joint between Grout Layers**  
A cold joint usually results when freshly mixed grout is placed on hardened grout. For underwater application, a sediment layer was sandwiched between the grout layers. The amount of sediment material depends on the extent of washout; however, it is not clear whether the presence of sediment will have any impact on the performance criteria.
6. **Number of Injection and Pumper Units**  
For a self-leveling grout, a batch of grout material would cover a large area. This would tend to minimize the number of injection points and pumper units needed. But, if a low slump grout is used, more grout transferring/injecting equipment and/or relocation of the injection apparatus will be needed.
7. **Dimensional Stability of Grout**  
During the curing process, the grout should not expand or contract excessively. If the grout expands too much, the pool liner/wall could be cracked and containment lost. Contraction should not be a problem because the next pour/lift should fill in the voids.

Some other operation issues also need to be addressed. An example of this is how to deliver the grout product by vendor to the application site at CPP-603, since the properties of the grout mixture may change over time. On site preparation may involve some quality control issues because the addition of the admixtures increases the complexity of the operation. Advance planning would minimize some of the potential problems.

## **RECOMMENDATIONS**

These tests have demonstrated that a high flowability, anti-washout grout may be prepared using simulated CPP-603 concentrated water. The grout had a high strength but contained some void areas and “cold joints”. The heat of curing for this grout mixture was insignificant.

One important topic that needs to be addressed is the definition of the performance criteria for the CPP-603 basins grouting project. Some issues are important, and some may be neglected. Some examples of performance criteria are: the slump and flow properties of the grout, extent of washout, cold joints, voids, floated particulates, etc. In addition, the procedures used to inject the grout underwater at CPP-603 basins will need to be identified. This would provide additional guidance for development of a workable grout formula.

Based on the results and the knowledge gained from this study, future studies are recommended for the grout formulation and demonstration tests. These studies may include flow tests, slump tests, tests to determine the pumping characteristics for the grout, and other tests related to the proposed grout placement equipment or systems. There is a limited amount of experimental data on the chemical admixture, and possible interaction between the cement, mineral admixtures and other admixtures. It is very difficult to predict the specific behavior of any particular mixture without testing. When more than one admixture is added, the impact on the grout behavior cannot be accurately predicted. If the grout's self-leveling property needs to be evaluated to minimize the number of injection points, a scale up underwater demonstration test is recommended. If possible, the materials used for further testing needs to be the same as the materials used for use in the CPP-603 basins decommissioning application.

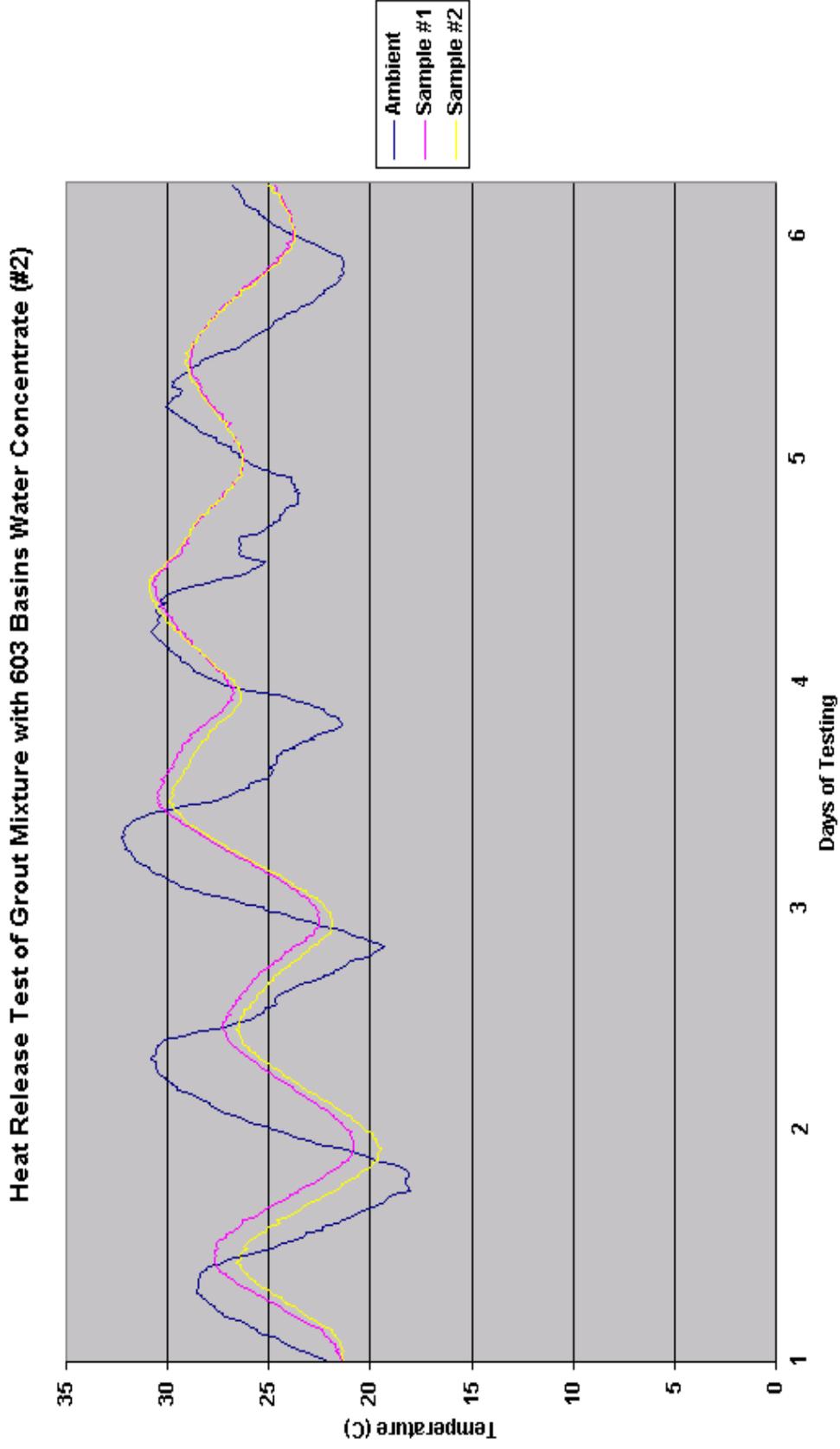
## REFERENCES

1. K. H. Khayat, M. Sonebi, A. Yahia, and C.B. Skaggs, "Statistical Models to Predict Flowability, Washout Resistance and Strength of Underwater Concrete." Proceedings, RILEM Conference on Production Methods and Workability of Concrete, Glasgow, 1996. pp 463-481.
2. K. H. Khayat, and M. Sonebi, "Effect of Mixture Composition on Washout Resistance of Highly Flowable Underwater Concrete." ACI Material Journal, July- August 2001, pp 289-295.
3. CRD C61-89A. "Test Method for Determining the Resistance of Freshly Mixed Concrete to Washing Out in Water." U.S Army Experiment Station Handbook for Concrete. Vicksburg, Miss. 1989. 3 pp.

**Figure 1** Temperature Test for Grout.



Figure 2.



**Figure 3** Three layers of grout poured under CPP-603 simulant.



**Figure 4 Separated Grout Layers.**

