INEEL/EXT-03-01095

Out-of-Drum Grout Mixer Testing with Simulated Liquid Effluents Originating from Sodium-Bearing Waste at the Idaho Nuclear Technology and Engineering Center

B. A. Scholes
A. K. Herbst
S. V. Raman
S. H. Hinckley

September 2003

Idaho National Engineering and Environmental Laboratory
Bechtel BWXT Idaho, LLC

INEEL/EXT-03-01095

Out-of Drum Grout Mixer Testing with Simulated Liquid Effluents Originating from Sodium-Bearing Waste at the Idaho Nuclear Technology and Engineering Center

B. A. Scholes
A. K. Herbst
S. V. Raman
S. H. Hinckley

September 2003

Idaho National Engineering and Environmental Laboratory
Idaho Falls, Idaho 83415

Prepared for the
U.S. Department of Energy
Assistant Secretary for Environmental Management
Under DOE Idaho Operations Office
Contract DE-AC07-99ID13727

ABSTRACT

The Idaho National Engineering and Environmental Laboratory (INEEL) is considering several optional processes for disposal of liquid sodium-bearing waste. During fiscal year 2003, alternatives were evaluated for grout formulation development and associated mixing for the Sodium-Bearing Waste cesium ion exchange process. The neutralization agents calcium or sodium hydroxide and the solidification agents Portland cement, with or without blast furnace slag were evaluated. A desired uniform formulation was pursued to develop a grout waste form without any bleed liquid and solidify within a reasonable period of about twenty-eight days. This testing evaluates the out-of-drum alternative of mixing the effluent with solidification agents prior to being poured into drums versus the in-drum alternative of mixing them all together after being poured into the drums. Experimental results indicate that sodium-bearing waste can be immobilized in grout using the Autocon continuous mixer within the range of 66 to 72 weight percent. Furthermore, a loading of 30 weight percent NWCF scrubber simulant also produced an acceptable grout waste form.

EXECUTIVE SUMMARY

The Idaho National Engineering and Environmental Laboratory, specifically the Idaho Nuclear Technology and Engineering Center (INTEC) High-Level Waste Program, is to prepare the liquid sodium-bearing waste for eventual disposal. Several alternative treatment processes and disposal paths have been explored for these wastes. This report discusses research conducted on one of these process options, which is grouting of sodium-bearing waste following cesium removal.

The out-of-drum grout mixer testing has demonstrated that the Autocon continuous mixer is capable of producing an acceptable grout waste form from the WM-180, WM-189, and NWCF scrub simulants. The best grout formulations were made by combining the solids (calcium hydroxide and Portland cement and/or blast furnace slag) then mixing the combined solids with the liquid SBW to both neutralize and solidify the acidic simulant. The Autocon mixer was sized to allow processing of grout batch sizes in the range of one to fifty-five gallons.

CONTENTS

FIGURES

TABLES

APPENDICES

OUT-OF-DRUM GROUT MIXER TESTING WITH SIMULATED LIQUID EFFLUENTS ORIGINATING FROM SODIUM-BEARING WASTE AT THE IDAHO NUCLEAR TECHNOLOGY AND ENGINEERING CENTER

1. INTRODUCTION AND BACKGROUND

1.1 Previous History and Prior Data

The grouting of sodium-bearing waste (SBW) as part of the cesium ion exchange (CsIX) process was proposed several years ago as a treatment for SBW.[1, 2] It was determined that SBW could be grouted with two general formulations. The first formulation provides a 40 wt% loading of SBW to the total mass of the waste form. This uses liquid sodium hydroxide to neutralize the waste acidity with subsequent cementation with Portland cement and blast furnace slag. The second formulation provides a 70 wt% loading and uses solid calcium hydroxide to neutralize the acid and then mixing with Portland cement and blast furnace slag. The latter formulation was recommended for disposal at WIPP for the CsIX SBW. In general, the waste form must be solid with less than 1% free liquid and the radiation level must be less than 200 millirem (mR) for contact handled waste. Recent regulation changes to allow the RCRA listed code for hydrofluoric acid (U-134), now specify that no free liquids are allowed for U-134 listed wastes.[3]

The blast furnace slag is added to the waste form to help meet toxic metal leach resistance requirements. Since, there are no toxic metal leach requirements for Waste Isolation Pilot Plant (WIPP), the slag may be eliminated, thereby simplifying the grout formulation. The deletion of slag from the baseline formulation was evaluated in the pilot-scale tests and described in this report.

Over the years, the estimated composition of the SBW has varied due to projected tank farm operations and waste evaporation. Current sampling has established the composition of tanks WM-180 and WM-189.[4, 5] The recommended waste formulations to meet the requirements of an out-of-drum mixer will be tested against these new SBW compositions. The projected simulant compositions are shown in Table 1.

If the calcination alternative were to be pursued, the SBW would be calcined in the existing New Waste Calcination Facility (NWCF) with additional, upgraded off-gas treatment equipment.[6, 7] This is a heated fluidized bed process where the liquid SBW is solidified. The process requires an off-gas scrubber system. In the past, the scrubber solution was recycled to the tank farm and the calciner; however, with the closure of the tank farm, the scrubber solution needs to be disposed of directly as remote handled transuranic (TRU) WIPP waste. This testing will also try to determine if the scrubber solution can be grouted using the Autocon continuous mixer.

Preliminary testing with a continuous grout mixer was done in July 2000 at a test facility on two grout formulations.[8] This demonstration revealed that a continuous mixer was capable of blending the liquid acidic simulant and dry powder cement additives to produce a homogenous grout mix. A continuous mixer could handle a wide range of grout formulations and fluid properties. There are mixers available that can handle any of the projected feed rates. Possible advantages of using a continuous mixer to process grout are: 1) less drums that need to be shipped, 2) shorter processing times, and 3) small multiples of a continuous formulation verses a lot of individual batches.

1.2 Purpose and Scope

One candidate technology for sodium-bearing waste (SBW) treatment is the removal of cesium via ion exchange (CsIX) followed by solidification of the liquid for disposal at the Waste Isolation Pilot Plant (WIPP). Removal of the cesium eliminates the majority of the gamma radiation in the waste; thus, the liquid can be treated as contact handled transuranic (TRU) waste. An alternative technology that also needs a solidification process is the scrubber solution from the calcination process that can no longer be recycled back to the tank farm. This report covers the use of a pilot-plant continuous grout mixer as one of the solidification options for SBW and the NWCF scrubber solutions. The main purpose of this pilot scale testing is to determine the optimum waste loading for use with a continuous grout mixer.

This experimental work was conducted in accordance with "Liquid Low Level Waste Stabilization / Solidification Experimental Testing," Independent Hazard Review Package IHR# INTEC-00-17, Modifications 2 and 3.

1.3 Test Objectives and Evaluation Criteria

The objective of the SBW solidification tests is to provide experimental data to assist in the overall process design and equipment selection efforts. Specific grout data is needed regarding wet grout viscosity, cure time, waste form density and volume, and the recommended formulation for optimum waste loading that meets the waste acceptance criteria. In the preliminary testing described in this report, the wet grout viscosity for various waste loadings of WM-180 and WM-189 SBW simulants were obtained from acceptable grouts defined as those that cured within one week.

2. THEORY AND APPROACH

Grout is a mixture of Portland cement, other powdered additives, waste, and water that hardens with hydration of the cement to form a solid. Portland cement is a generic name for hydraulic cement and is composed mainly of high-lime calcium silicates with lesser amounts of high-lime aluminates and ferrites, which are ground together with a small amount of gypsum to a fine powder. It may contain fine-grained sand and does not include large aggregate material. During hardening, the calcium compounds chemically combine with water to produce the hard, finished product.

In general, the liquid to be grouted must be chemically basic (caustic) in order for the cement to hydrate properly; thus, the need to neutralize the acidity in the SBW prior to grouting. Grouting was completed in either a single step or a two-step process. In the two-step process, neutralization was first done through the mixer as step one and the neutralized solution was then the feed for step two. For the single step process, all the powders (cement, slag, and calcium hydroxide) were combined and then simultaneously mixed with the simulant. For the cesium ion exchange experimental test runs, it may be necessary to partially neutralize the acid (about pH ~ 1-2) to avoid resin degradation. Liquid sodium hydroxide may be used prior to ion exchange since calcium hydroxide would produce undissolved solids. A disadvantage is the slight dilution resulting from use of liquid sodium hydroxide.

Several optimum formulations from the laboratory tests on the SBW simulants of WM-180 and WM-189 and the NWCF scrubber simulant were tested in the continuous grout mixer. The baseline grout formulation for CsIX-TRU grout to be tested without any slag is 70 wt% SBW, 14 wt% calcium hydroxide, and 16 wt% Portland cement. The baseline grout formulation for NWCF-TRU grout to be tested is 35 wt% NWCF scrub, 18 wt% sodium hydroxide, 1 wt% calcium hydroxide, 41 wt% furnace slag, and 5 wt% Portland cement. These formulations were iterated as needed to develop optimum fluid properties for the continuous mixer.

3. EXPERIMENTAL

3.1 Facility and Equipment Description

The general compositions of SBW represented in storage tanks WM-180 and WM-189, not including the radioisotopes, are presented in Table 1. Appendix A contains copies of batch sheets that contain the non-hazardous composition of a WM-180 and WM-189 SBW simulant used in this testing. The non-hazardous SBW simulant was made up, as needed for either the 5-gallon or 55-gallon test runs, using the makeup tanks located in CPP-620 High bay. After making up the simulant it was analyzed by INTEC-Analytical Laboratory Department (ALD) to ensure that it adequately represented the targeted SBW simulant composition. The acidic liquid SBW simulant was transferred using a drum pump into carboys or drums and stored for use in the CPP-637 Low Bay where the grout mixer is located.

Additives to be used for these tests were:

- Commercial grade Portland cement Type I/II.
- Blast furnace slag, NewCem from Blue Circle Cement.
- 50% (by volume) sodium hydroxide liquid reagent from Fisher Scientific.
- Calcium hydroxide solid reagent from Fisher Scientific.

This experimental work was conducted in the CPP-637 Low Bay enclosed ventilated module. The continuous grout mixer setup includes a 6-inch diameter turbo-type mixer made by Autocon with a volumetric dry solids feeder containing a 1.5-inch diameter metering auger and liquid peristaltic pump driven by a 1/3hp AC motor. Each of the AC electrical motors for the mixer, liquid pump, and solids feeder has the speed controlled by a 240 VAC 3-phase variable voltage frequency drive. This continuous mixer setup was used to either neutralize the SBW simulant with calcium hydroxide and then mix this with the cement in a two-step process, or combine a mixture of cement, furnace slag, and calcium hydroxide with the SBW simulant in a single step to produce a grout formulation without any free liquid. The solid powders were weighed out and measured in the northwest HEPA-filtered hood located in CPP-637 Low Bay. A Turbula mixer/shaker is also located in the module for mixing any of the dry solids. For each test run, enough SBW liquid simulant and dry solids mixture were prepared in advance to produce a 5-gallon batch of grout. An Ergo-matic HR-800 drum lifter or portable lift table was used to lift or move the 5-gallon containers into place under the grout mixer for loading. After each test run, the mixer was rinsed out with water that was collected and reused as rinse water in the next test. The excess rinse water was grouted for disposal.

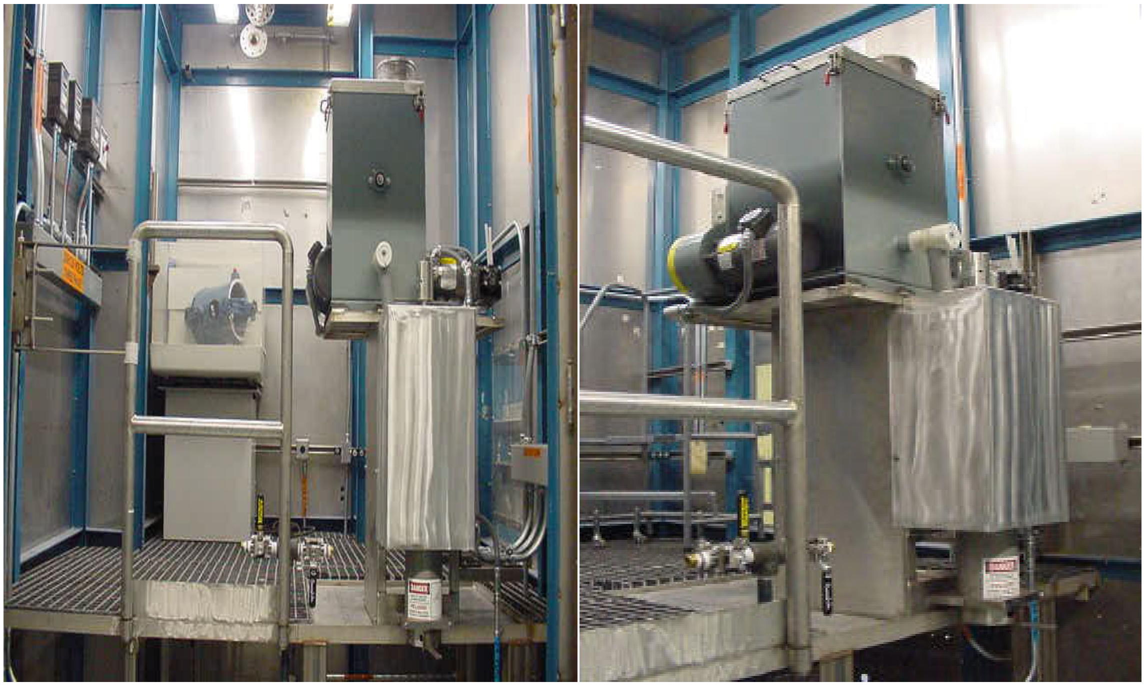
Viscosity measurements were made using a Brookfield Model DV-E viscometer. This viscometer is designed for thick solutions such as cements and pastes. Viscosity standards were used to check the instrument accuracy.

Curing set time was noted by Vicat testing.[7] This instrument utilizes a 1 millimeter diameter needle mounted below a 300 gram weight. The needle is placed at the top surface of the grout sample and allowed to drop and penetrate the grout. The depth of penetration indicates the amount of curing. If penetration is greater than 25 millimeters, there is no set. A penetration of 25 millimeters or less indicates initial set has started. No penetration shows final set has been achieved.

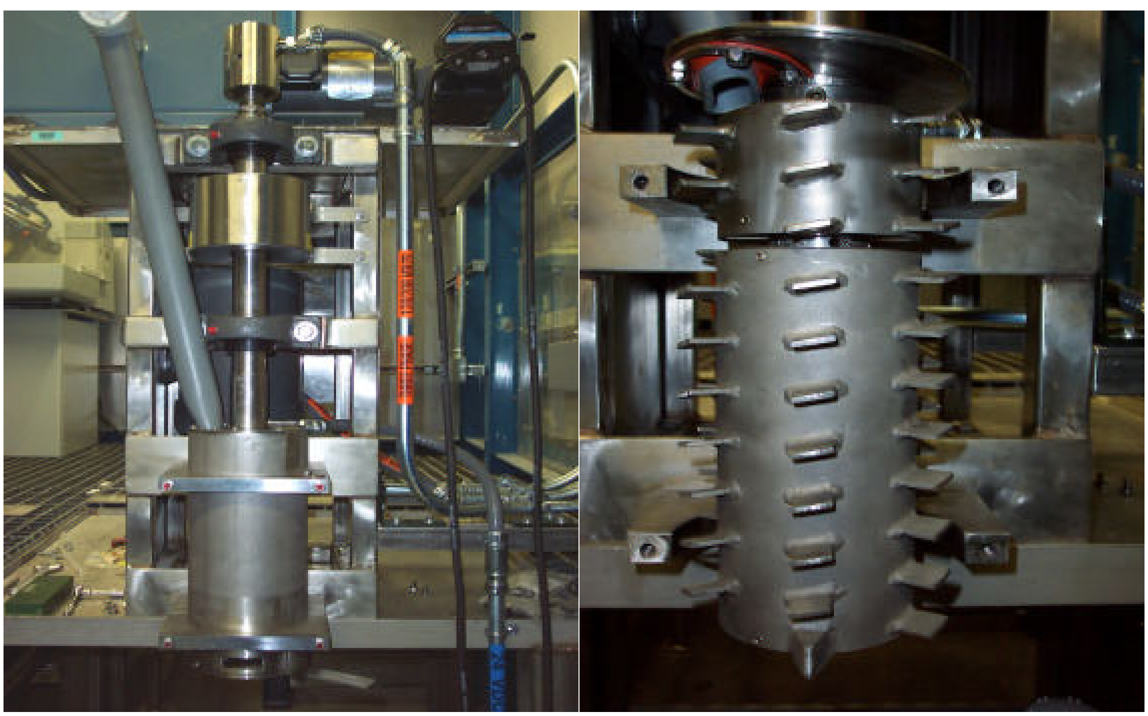
Grout samples were allowed to cure for about 1 week. If any free liquid was noted in or on the sample following this cure time, the sample was deemed unacceptable and no further tests were done on such samples.

All the equipment components of this continuous grout mixer were assembled and operated according to the manufacturer's instructions. Digital images and measurements were taken of the Autocon continuous mixer assembly and mixing chamber for corrosion determination as necessary. These images are presented in Figures 1 and 2.

Initial scoping tests were performed to show proof of concept and determine approximate operational parameters. Further testing was conducted to optimize and more clearly define the ranges of operating parameters. These images during testing are shown in Figures 3.



Figures 1a and 1b. Out-Of-Drum Mixer Assembly



Figures 2a and 2b. Autocon Continuous Mixing Chamber

Figures 3a and 3b. Grout Mixer Testing – 5 Gallon Test


Figures 4a and 4b. Grout Mixer Testing – 55-Gallon Test

Table 1. Projected Simulant Concentrations

Element	WM-180 SBW Simulant (M)	WM-189 SBW Simulant (M)	NWCF Scrubber Simulant (M)
Acids (H+)	1.01E+0	2.86E+0	2.33E+0
Aluminum (Al)	6.63E-1	7.11E-1	1.56E+0
Arsenic (As) *	4.99E-4	0	9.03E-5
Barium (Ba) *	5.58E-5	5.62E-5	2.42E-5
Boron (B)	1.23E-2	2.12E-2	5.42E-3
Cadmium (Cd) *	7.54E-4	3.91E-3	9.68E-4
Calcium (Ca)	4.72E-2	7.30E-2	5.04E-2
Cesium (Cs) *	7.73E-6	2.68E-5	3.87E-6
Chloride (Cl)	3.00E-2	2.06E-2	3.81E-2
Chromium (Cr) *	3.35E-3	5.64E-3	1.73E-3
Copper (Cu)	6.97E-4	9.54E-4	1.56E-4
Fluoride (F)	4.74E-2	1.38E-2	8.88E-2
Gadolinium (Gd) *	1.77E-4	1.35E-4	3.42E-5
Iron (Fe)	2.17E-2	2.68E-2	1.22E-2
Lead (Pb) *	1.31E-3	1.16E-3	3.48E-4
Lithium (Li) *	3.39E-4	3.84E-4	8.11E-5
Magnesium (Mg)	1.20E-2	2.21E-2	4.32E-3
Manganese (Mn)	1.41E-2	1.95E-2	4.28E-3
Mercury (Hg) *	2.02E-3	6.50E-3	2.10E-1
Molybdenum (Mo) *	1.93E-4	2.80E-4	4.15E-4
Nickel (Ni) *	1.47E-3	2.32E-3	7.28E-4
Nitrate (NO_3)	5.01E+0	6.52E+0	8.24E+0
Palladium (Pd) *	2.35E-5	0	3.58E-6
Phosphate (PO_4)	1.37E-2	2.07E-3	3.10E-2
Potassium (K)	1.96E-1	2.25E-1	7.90E-2
Ruthenium (Ru) *	1.25E-4	1.72E-4	3.26E-5
Selenium (Se) *	1.45E-4	0	2.16E-5
Silicon (Si)	3.02E-7	3.08E-4	3.68E-2
Silver (Ag) *	5.29E-6	0	5.64E-6
Sodium (Na)	2.06E+0	2.04E+0	6.03E-1
Strontium (Sr) *	1.19E-4	1.41E-4	2.65E-5
Sulfate (SO_4)	5.40E-2	1.07E-1	2.41E-2
Vanadium (V) *	9.23E-4	2.51E-5	7.78E-6
Zinc (Zn) *	1.05E-3	1.07E-3	2.47E-4
Zirconium (Zr)	6.33E-5	3.57E-4	1.86E-2

*May be left out due to small amount or for make ups without RCRA metals.

3.2 Test Procedure

For each different feed stream, it is necessary to calibrate the liquid feed pump and solids feed hopper due to differing densities. The feed pump and hopper were operated with the respective materials to measure the flow rates in grams per minute verses the variable speed drives in hertz to obtain a linear mass feed rate calibration curve for each feed material. With the calibration curves, the desired grout formulation, in terms of weight percent, can be set into the variable speed drives (see Appendix B).

The initial test runs with the pilot-plant continuous grout mixer were a basic formulation of Portland cement and water. For the grout formulations using either the WM-180 or WM-189 SBW simulant, it was decided to first try the easiest approach of adding the Portland cement and calcium hydroxide together in one step to both neutralize and solidify at the same time. As the mass loadings of SBW simulant varied in the grout, the ratio of cement to calcium hydroxide mass loadings and the solids feed rate was kept constant to avoid having to re-calibrate the solids feeder. The second approach that was tried to make an acceptable grout formulation was to mix the WM-180 or WM-189 SBW simulant with the calcium hydroxide for neutralization in one step. Then mix the cement with this combination as a second step to produce a solid grout waste form. The last approach in this testing involved using the pre-neutralized SBW or NWCF scrub simulants and then combining with the solids (calcium hydroxide, slag, and cement) to make an acceptable grout formulation.

After initial testing with different mixer speeds and feed rates within the anticipated output for a 5-gallon batch, it was decided to make only 1-gallon samples at a time to minimize the amounts of any unacceptable grout formulations. After producing the initial gallon sized sample, the pH and temperature, or the highest temperature measured by a thermocouple on the outside of the mixing chamber, also the wet grout density and viscosity were usually measured if the grout product appeared to be acceptable in thickness and homogeneity. Then a larger sample size up to 5-gallons was produced to help verify the acceptability of the grout formulations produced in the laboratory. After allowing sufficient time for the grout samples to cure, the samples were retained if there was no free liquid or they were disposed of if any free liquid was detected after the appropriate curing time.

4. RESULTS

The calibration curves relate feed flow rates in grams per minute to motor speeds as indicated by the controllers set point in hertz for the alternating voltage frequency. Generating the linear calibration curves for the liquid feed peristaltic pumps using water or the SBW simulants only required running the particular pump once at 3 or 4 representative speed set points for a sufficient time interval within an anticipated output range. The calibration of the solids volumetric feeder required running it several times at the 3 or 4 representative speeds for a timed interval and averaging the results within the anticipated output range. The weight of the feed over a set time interval were measured and the results plotted along an X-axis for speed and a Y-axis for flow rate in grams-per-minute. A linear curve was then fitted to the graph to allow for calculation of the speed set point for the desired feed rate.

Calibration curves were generated for each of the different feeds needed for this initial testing. The volumetric metering liquid pump came with a smaller sized Master-flex pump head with a flow rate that was not large enough to produce a large 55-gallon batch of grout. A larger Master-flex pump head with a flow rate capable of producing the large 55-gallon batch of grout was installed and used with both SBW simulants. The volumetric solids feeder was operated with different ratios of Portland cement, calcium hydroxide, and with or without furnace slag to obtain calibration curves for a linear feed rate. Each of the calibration curves generated for either the liquid feed pump or solids feeder are shown in Appendix B.

4.1 Out-Of-Drum Grout Mixer Tests using Portland Cement and Water

The test results from mixing the ordinary Portland cement with water were fairly straightforward and the results are only a preliminary operational trial test. Several different loadings and speed setting were tried to evaluate the mixer operation, which produced cement that was well mixed and setup without any free liquids. The first sample (OPC-50-1) contained the wrong ratio of cement to water and came out of the mixer at first with a very low viscosity. After adjusting the liquid feed several times, the viscosity of the cement mixture became very thick and eventually plugged up the mixing chamber and stopped the test run. The next formulations of cement contained at least a 2 to 1 ratio of cement to water instead of the 1 to 1 ratio used in the first test run. The viscosity of the second cement sample was still quite thin so the remaining test runs used an even higher loading of cement to produce a consistently thick cement mixture. Varying the starting times of either the liquid or solids feeders produced the remaining test sample results. The data from these samples are being reported for all of this initial grout mixer testing. The testing results of the targeted versus actual mass percent loadings of the Portland cement and water are given in Table 2.

Table 2. Out-Of-Drum Grout Mixer Tests using Ordinary Portland Cement and Water

Samples	Target Water Loading (wt%)	Target Cement Loading (wt%)	Actual Water Loading (wt%)	Actual Cement Loading (wt%)	Sample Batch Size (gal)	Cement Viscosity (cP)	@ Speed (RPM)	@ Torque (%)	Comments
OPC-50-1	50%	50%	NM	NM	5	NM	NM	NM	Mixer plugged
OPC-50-2	33%	67%	56%	44%	1	NM	NM	NM	Too thin
OPC-45-1	31%	69%	36%	64%	1	8000	10	5	Too thin
OPC-45-2	31%	69%	40%	60%	1	7000	10	5	Too thin
OPC-45-3	31%	69%	34%	66%	1	36000	10	15	Not well mixed
OPC-45-4	31%	69%	35%	65%	1	NM	NM	NM	Too thin

4.2 Out-Of-Drum Grout Mixer Tests using WM-180 SBW Simulant

The first series of test runs with the WM-180 simulant still experienced some plugging problems and after consulting with Autocon it was determined that we had been operating the mixer in the wrong direction. The test results of samples MC6-72-1 to MC6-72-4 experienced the plugging problems of the mixer due to incorrect mixer rotation and although not meaningful are included for completeness. The rest of the test samples were produced with the corrected mixer rotation and different mass loadings. At different mixer operating speeds (ranging from 15 to 36 hertz), there was not any difference in the grout performance, but the highest temperatures recorded by the thermocouple attached to the outside of the mixing chamber were at the lower speeds. There was also vapor visible at the outlet of the mixer from the acid base chemical reaction.

The actual mass loadings often varied greatly from the targeted value. This mass loading variation was because of the difficulty in balancing the masses that were processed through the mixer. Material was initially held up in the mixing chamber and then lost when it was rinsed out at the end of each test run. Also, the weights of the liquid feed and the grout product were weighed and recorded on a worksheet while the weight of the solids feed is the difference between the grout product minus the liquid feed. With the initial testing on the small one-gallon batches, the results were easily varied with any changes in the amounts of materials fed into the mixer. For example, grout samples MC6-72-6 and MC6-72-7 were produced using a specific amount of SBW simulant and the actual mass loadings were close to the targeted amounts, except the 4-gallon (MC6-72-7) grout sample had more solids fed into it than was expected.

Sample (MC6-72-8) was run to try neutralizing the WM-180 acidic liquid feed first with the calcium hydroxide and see how well it mixes. It was then fed back through and mixed with the cement to see how well the grout turns out. After mixing the WM-180 simulant with the calcium hydroxide, there were still lumps at the bottom of the bucket and it took about ten minutes of hand stirring to finish dissolving the calcium hydroxide. The measured pH of the neutralized liquid was 3.5 and there was quite a bit of calcium hydroxide still stuck to the walls of the mixing chamber when it was cleaned out. This would explain why the actual mass loadings did not coincide with the targeted loadings for this sample. After mixing the Portland cement with the neutralized simulant, the grout product was too thin for viscosity measurements, but it did set up eventually without any free liquid.

The rest of the samples were tested using the SBW simulants pre-neutralized to about 0.5 molar. These samples included furnace slag in an attempt to prevent the growth of sodium nitrate crystals that appear as the grout cures. Different mass loadings were also tried to produce an acceptable grout formulation. The last samples (MMC49-72 and MMC49-76) are from the 50-gallon batch run that had to be split in two because of plugging problems. The grout viscosity through the mixer seemed all right, but thickens quickly enough that the mixer speed was not fast enough to prevent the grout from plugging up inside the mixing chamber. So after unplugging the mixer, it was run at a higher speed and with a higher SBW loading to produce a thinner grout that would still cure without any free liquids. Keeping the lid on the drum of grout prevented the sodium nitrate crystals from growing like they have done in the lab-sized samples that are kept in a ventilated hood. The data sheets detailing the results of the entire grout mixer testing are given in Appendix C. The test results from mixing the WM-180 SBW simulant with the combined solids are given in Table 3.

Table 3. Out-Of-Drum Grout Mixer Tests using WM-180 SBW Non-Hazardous Simulant

Samples	Actual Liquids Loading (wt%)	Actual Solids Loading (wt%)	Target Liquids Loading (wt%)	Target Solids Loading (wt%)	Sample Batch Size (gal)	Grout Mix (pH)	@ Temp. (C)	Mix Viscosity @10 rpm (cP)	Comments
MC6-72-1	74%	26%	72%	28%	1	9.49	39	8560	Invalid sample, not well mixed
MC6-72-2	71%	29%	72%	28%	1	9.21	39	2400	Invalid sample, too thin
MC6-72-3	77%	23%	72%	28%	1	NM	NM	NM	Invalid sample, solids feed plugged
MC6-72-4	72%	28%	67%	33%	1	10	33	NM	Invalid sample, not well mixed
MC6-68-1	71%	29%	68%	32%	1	9.8	59	25600	Ok
MC6-68-2	72%	28%	68%	32%	1	9.9	45	52000	Ok
MC6-68-3	70%	30%	68%	32%	1	10	47	442000	Ok
MC6-68-4	72%	28%	68%	32%	1	10	59	30480	Ok
MC6-68-5	76%	24%	68%	32%	1	10	49	32000	Ok
MC6-72-6	72%	28%	72%	28%	1	9.48	40	7200	Ok
MC6-72-7	67%	33%	72%	28%	4	NM	32	NM	Good sample
MC6-72-8	108%	-8%	94%	6%	0.76	3	29	NM	Mixed WM-180 with Ca(OH)2
MC6-72-8	77%	23%	77%	23%	0.92	10	29	NM	Mixed WM-180&Ca(OH)2 with OPC
MC49-74-1	78%	22%	77%	23%	1	NM	20	NM	Very thick and well mixed
MC49-72-1	76%	24%	75%	25%	1	NM	24	NM	Very thick and well mixed
MC49-70-1	70%	30%	73%	27%	1	NM	25	NM	Very thick and well mixed
MC49-77-1	77%	23%	80%	20%	1	NM	25	NM	Very thick and well mixed
MMC49-72	80%	20%	75%	25%	15.5	NM	NM	NM	Very thick and well mixed
MMC49-76	76%	24%	79%	21%	26.2	11.37	35	NM	Very thick and well mixed

4.3 Out-Of-Drum Grout Mixer Tests using WM-189 SBW Simulant

At 68 wt% WM-189 SBW loading for the first two samples, the grout product was too thin and not mixed very well. It was decided to increase the solids feed rate to try making the grout thicker. Grout sample MA6-68-3 was mixed well, but was so thick that it could barely be stirred by hand. Sample MA6-68-4 was like the first ones that were too thin and not mixed well enough to set up. There was also some discrepancy in the mass loading results of the first four samples because a large 910 kg capacity drum scale with 0.2 kg precision was used to weigh the actual liquid feed and grout product. The next four samples (MA6-68-5 to MA6-68-8) were done during a test run with a 100 kg capacity scale with 0.01 kg accuracy. The only one of these samples to mix well and be thick enough to set up was MA6-

68-6. It appears that the 68 wt% loading of WM-189 SBW is too high for testing with the Autocon mixer.

The three samples (MA6-62-1, MA6-62-2, & MA6-65-1) at the lower SBW mass loadings were mixed with a specific amount of liquid simulant and produced acceptable grout products without any free liquid. Only the 5-gallon sample (MA6-62-2) was very thick and well mixed, while the other two were of a thinner consistency and not very well mixed. The last sample (MA6-62-3) was prepared by neutralizing the WM-189 acidic liquid feed first with the calcium hydroxide, then fed back through and mixed with the cement. Samples MA6-62-3A & MA6-62-3B resulted from mixing the WM-189 simulant with the calcium hydroxide in two half-sized batches, there were a few small lumps at the bottom of the bucket and it took several minutes of hand stirring to get most of the calcium hydroxide to dissolve. The measured pH of the neutralized liquid was about 2 and there was quite a bit of calcium hydroxide still stuck to the walls of the mixing chamber when it was cleaned out. After mixing the Portland cement with the neutralized simulant, the grout product was too thin for viscosity measurements and it contained some free liquid.

Sample MA42-68-1 contained furnace slag in the solids (8wt% $Ca(OH)_2$, 8wt% slag, and 16wt% cement) to see if it would help to prevent the sodium nitrate crystals from growing as the grout cures. During the test run there was a distinct sulfur smell being given off, as the acidic simulant was being neutralized and grouted at the same time. A concern was given if hydrogen sulfide gas is being generated then this composition could be dangerous if processed in large quantities.

The rest of the samples were tested using the pre-neutralized SBW simulant (about 0.5 molar). The MA49 series of samples contained in the combined solids 10 wt% $Ca(OH)_2$ and either 7 or 8 wt% of the furnace slag or cement components These samples included the furnace slag to try preventing the growth of sodium nitrate crystals and different mass loadings were tried to produce an acceptable grout formulation. Sample MMA55-66 is from the 50-gallon batch run that had 7 wt% $Ca(OH)_2$, 6 wt% slag, and 12 wt% cement to help make the grout cure faster. This grout would come out of the mixer quite thin and quickly thicken without creating very much build up inside the drum. Keeping the lid on the drum of grout prevented the sodium nitrate crystals from growing like they have done in the lab-sized samples that are kept in a ventilated hood. Samples MA55-62-1 and MA55-70-1 were run at the lower and higher waste loadings to get an idea of the range in which an acceptable grout could be produced. Sample MAC55-75-1 is a mixture of equal parts of both WM-180 and WM-189 simulants to use up the remaining test liquids and see if an acceptable grout could be produced from both of these pre-neutralized simulants. The data sheets detailing the results of the entire grout mixer testing are given in Appendix C. The test results from mixing the WM-189 SBW simulant with the combined solids are given in Table 4.

Table 4. Out-Of-Drum Grout Mixer Tests using WM-189 SBW Non-Hazardous Simulant

Samples	Actual Liquids Loading (wt%)	Actual Solids Loading (wt%)	Target Liquids Loading (wt%)	Target Solids Loading (wt%)	Sample Batch Size (gal)	Grout Mix (pH)	@ Temp. (C)	Mix Viscosity @10 rpm (cP)	Comments
MA6-68-1	81%	19%	67%	33%	1	8 or 9	31	NM	Too thin and not well mixed
MA6-68-2	68%	32%	67%	33%	1	8.45	53	NM	Too thin and not well mixed
MA6-68-3	70%	30%	61%	39%	1	NM	32	NM	Too thick, but mixed well
MA6-68-4	68%	32%	64%	36%	1	8.41	55	NM	Too thin and not well mixed
MA6-68-5	75%	25%	68%	32%	1	NM	32	NM	Too thin and not well mixed
MA6-68-6	62%	38%	65%	35%	1	8.58	58	28000	Well mixed and thickens as it cools
MA6-68-7	69%	31%	68%	32%	1	NM	39	NM	Too thin and not well mixed
MA6-68-8	74%	26%	68%	32%	1	8.55	50	3000	Too thin and not well mixed
MA6-62-1	68%	32%	62%	38%	1	8.90	48	8000	Good sample
MA6-62-2	63%	37%	62%	38%	5	NM	57	NM	Good sample
MA6-65-1	71%	29%	65%	35%	1	8.68	52	2560	Ok
MA6-62-3A	96%	4%	95%	5%	5	-0.07	37	NM	Mixed fairly well and lite brn color
MA6-62-3B	96%	4%	95%	5%	5	1.96	50	NM	Mixed fairly well and dk brn color
MA6-62-3	67%	33%	67%	33%	5	8.76	42	NM	Too thin and not well mixed
MA42-68-1	70%	30%	68%	32%	5	7.74	60	NM	Well mixed and very thick, sulfur smell
MA49-68-1	81%	19%	77%	23%	5	NM	21	NM	Mixed well and thick
MA49-70-1	80%	20%	79%	21%	5	9.18	40	NM	Mixed well and thick
MA49-66-1	76%	24%	75%	25%	1.6	NM	20	NM	Mixed well and very thick
MA49-70-2	71%	29%	79%	21%	1	NM	49	~50000	Well mixed and very thick
MA49-68-2	64%	36%	77%	23%	1	NM	48	~50000	Well mixed and very thick
MA49-66-2	61%	39%	75%	25%	1	NM	51	~50000	Well mixed and very thick
MA49-68-3	64%	36%	77%	23%	1	NM	52	~50000	Well mixed and very thick

4.4 Out-Of-Drum Grout Mixer Tests using NWCF Scrubber Simulant

There were also tests run with the grout mixer on the NWCF scrubber non-hazardous simulant that was pre-neutralized to pH of 12 with sodium hydroxide. These samples contained a combined solids of 2 wt% $Ca(OH)_2$, 88 wt% slag, and 10 wt% cement. Sample MB1-35-1 with a 35 wt% scrub simulant loading produced a very thin grout that contained some bleed water. So sample MB1-26-2 with a 26 wt% scrub loading was tried next to produce a thicker grout. This grout appeared to have a good consistency and was well mixed. The next sample MB1-30-3 with a 30 wt% scrub loading was also tried to produce a thinner grout that would still set up without any bleed water. With the remaining amount of simulant, it was decided to produce the larger batch of grout with the 26 wt% scrub loading. The data sheets detailing the results of the entire grout mixer testing are given in Appendix C. The test results from mixing the NWCF scrubber simulant with the combined solids are given in Table 5.

Table 5. Out-Of-Drum Grout Mixer Tests using NWCF Scrubber Non-Hazardous Simulant

Samples	Actual Liquids Loading (wt%)	Actual Solids Loading (wt%)	Target Liquids Loading (wt%)	Target Solids Loading (wt%)	Sample Batch Size (gal)	Grout Mix (pH)	@ Temp. (C)	Mix Viscosity @10 rpm (cP)	Comments
MB1-35-1	54%	46%	53%	47%	1	NM	NM	NM	Very thin, but well mixed
MB1-26-2	42%	58%	40%	60%	1	NM	NM	NM	Fairly thick and well mixed
MB1-30-3	47%	53%	45%	55%	1	NM	NM	NM	Thin, but well mixed
MB1-26-4	45%	55%	40%	60%	2.5	12.35	NM	20000	Fairly thick and well mixed

5. DISCUSSION AND ANALYSIS

The grout mixer testing with the simplified approach of adding the Portland cement and calcium hydroxide together to both neutralize and solidify at the same time resulted in producing the most acceptable grout product. The grout was mixed well and if the targeted mass loadings were met then it was more likely to set up and cure without any free liquid. The grout mixer testing of first neutralizing the SBW simulant with the calcium hydroxide and then mixing with the cement was a more difficult process and did not result in the grout being very well mixed. However they were mixed, after curing most of the grout samples had a white fuzzy crystalline growth of sodium nitrate develop of the top surface. Especially the smaller grout samples that were collected for viscosity, pH, and density characterization.

Grout tests without the blast furnace slag showed that WM-180 and WM-189 SBW simulants could be solidified; however, as noted above sodium nitrate leached out of the grout. This is an indication that other components, possibly radioactive components as well, could leach out. During the latter part of this testing, blast furnace slag was reintroduced into the formulation to determine if the sodium nitrate leaching could be prevented. The smaller lab-scale samples that were stored in a ventilated hood still leached the sodium nitrate crystals. The 5-gallon grout samples that were covered with a lid were less likely to leach, although some of the samples still appear as if they could have the sodium nitrate crystals develop given more time. The 50-gallon grout samples have not yet had any sodium nitrate crystals appear, but it maybe just a matter of time before they may start to develop. A time versus temperature plot is shown in Figure 3 that recorded the cure temperatures for each 55-gallon drum of SBW grouts. This plot includes the temperatures recorded in the center of the drums, the temperature of the outside of the drums, along with the ambient temperature during the time interval.

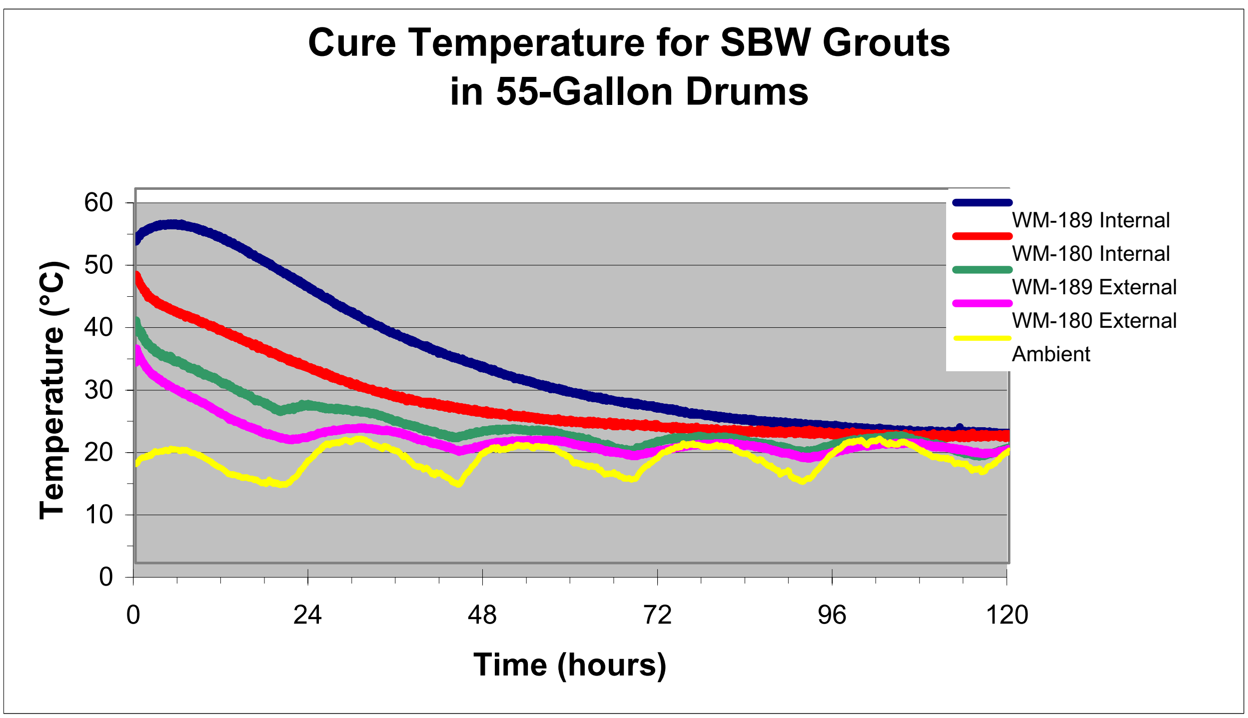


Figure 5. Cure Temperatures for SBW Grouts in 55-gallon drums.

All the initial grout mixer testing was done with WM-180 and WM-189 SBW simulants as is, but the latter tests used simulants that were partially neutralized prior to the grout mixing. Several combinations of the solid components were tried to develop a grout that would cure as soon as possible without any free liquids. Thus there is a need for continued testing to define, for each waste tank composition, the range of acceptable waste loadings to make an acceptable grout waste form.

The problem of the actual mass loadings varying from the target is because of the difficulty to balance the masses that are processed through the mixer. Variations between target and actual mass loadings can be attributed to several factors: (a) some material is retained in the mixer, and this is not included in initial runs, (b) the fact that the feeders are volumetric may not be as accurate as a gravimetric or loss-in-weight feeder. Only the weights of the liquid feed and the grout product can be recorded at the end of each test run, the weight of the solids feed is just the difference between the grout and liquid values. Also, this initial testing with the small one-gallon batches can easily vary the results with any changes in the amounts of materials fed into the mixer.

The problem of varying from the targeted mass loading was discussed with the Autocon mixer vendor. He stated that it was probably related to the very low speed at which we were trying to run the variable speed drives. The rule of thumb is that variable speed drives generally should not be run below three turns (20hz). In our case with these initial small batches, the variable speed drives are running at less than 10hz. This is the same for the Masterflex pump head and tubing that will be used to process the larger 55-gallon batches. On the solids feeder the conditioning arm to prevent bridging or ratholing was disabled because it stalled trying to move through the cement mixture at the lower speeds. Minor voids at the low feed rate could ruin the repeatability / consistency of the feeders flow rate.

6. CONCLUSIONS

The preliminary out-of-drum grout mixer testing demonstrated that the Autocon continuous mixer is capable of producing an acceptable grout waste form from any of the projected simulants. It is questionable though, if the Autocon continuous mixer as configured is capable of processing the grout formulations at the targeted mass loadings.

The best grout formulations were made by the method of combining the solids (calcium hydroxide and Portland cement), then mixing the combination with the liquid SBW to both neutralize and solidify the acidic simulant. The optimum waste loading so far for the WM-180 simulant was 73% and 64% for WM-189 simulant. These optimum waste loadings were easier to achieve using the pre-neutralized simulants. The optimum waste loading using the continuous mixer with the pre-neutralized NWCF scrub simulant was 30%.

With the pre-neutralized simulants there is still a reasonable chance that one formulation could be developed to produce an acceptable grout from any of the projected waste streams.

7. RECOMMENDATIONS

To improve feed rate consistency, the vendor Autocon recommends using DC motors and variable speed drives with a closed-loop-speed control (SCR board and controller) for low speed applications along with a fixed shaft auger for the solids feeder. Other applications that have used these DC motors with variable speed drives report that the repeatability has been excellent.[9] Also the conditioning arm in the solids feeder needs to operated at full speed with it’s own controller or switch.

All the grout mixer testing done with the projected waste simulants vary greatly, thus there is a need for continued testing to define for each waste tank composition, the range of acceptable waste loadings to make an acceptable grout waste form. There would also be the advantage of having one grout formulation make an acceptable grout waste form from the various waste simulants.

8. REFERENCES

1. A. K. Herbst, et al, “Idaho Nuclear Technology and Engineering Center Low-Activity Waste Process Technology Program FY-99 Status Report,” INEEL/EXT-99-00973, Sept. 1999.
2. A. K. Herbst, et al, “Idaho Nuclear Technology and Engineering Center Sodium-Bearing Waste Treatment Research and Development FY-2002 Status Report,” INEEL/EXT-02-00985, Sept. 2002.
3. J. B. Bosley, electronic communications (e-mail), “No Free Liquid/U-134,” forwarding electronic communication from S. Johansen of WIPP, January 29, 2003.
4. J. D. Christian, “Composition and Simulation of Tank WM-180 Sodium Bearing Waste at the Idaho Nuclear Technology and Engineering Center,” INEEL/EXT-2001-00600, May 2001.
5. T. A. Batcheller and D. D. Taylor, “Characterization of Tank WM-189 Sodium Bearing Waste at the Idaho Nuclear Technology and Engineering Center,” Report INEEL/EXT-02-01171, Sept. 2002.
6. C. M. Barnes, et al., “Process Design of SBW Treatment-Alternatives,” Engineering Design File 2373, September 2002.
7. C. M. Barnes, et al., “Calcination with MACT Upgrade Process Design,” Engineering Design File 3387, April 2003.
8. A. K. Herbst, et al, “Idaho Nuclear Technology and Engineering Center Low-Activity Waste Process Technology Program FY-2000 Status Report,” INEEL/EXT-2000-01167, Oct. 2000.
9. American Society for Testing and Materials, “Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle,” ASTM C191-01a.
10. U. S. Nuclear Regulatory Commission Technical Branch of the Low-Level Waste Management and Decommissioning Division, “Technical Position on Waste Form,” Revision 1, January 1991.
11. Tom C. Haas, unpublished e-mail correspondence Re: Grout Mixer Testing; dated 31 May 2003.

Appendix A

Simulant Batch Makeup Sheets

WM-180 Sodium-Bearing Waste -- Non-Hazardous Simulant Calculations
(based on "Composition and Simulation of Tank WM-180 SBW" by J. D. Christian)
Updated: 11/19/02
Make-up Date: 3/11/2003
Made-up By: S. H. Hinckley

Species	Desired Molarity	Stock Chemical	Molecular Weight	1 L Batch Amount		40.00 L Batch Amount	
Al	6.63E-01	2.2 *M* $Al(NO_3)_3$		301.50	ml	12060.00	ml
B	1.23E-02	H_3BO_3	61.832	0.76	g	30.33	g
Ca	4.72E-02	$Ca(NO_3)_2 \cdot 4H_2O$	236.15	11.14	g	445.76	g
Cl	3.00E-02	12.0 *M* HCl		2.50	ml	99.97	ml
Cu	6.97E-04	$Cu(NO_3)_2 \cdot 2.5H_2O$	232.59	0.16	g	6.48	g
F	4.74E-02	27.6 *M* HF		1.70	ml	67.98	ml
Fe	2.17E-02	$Fe(NO_3)_3 \cdot 9H_2O$	404.00	8.78	g	351.16	g
H	1.01E+00	All Acids	-----	-----		-----	
K	1.96E-01	KNO_3	101.10	19.84	g	793.43	g
Na	2.06E+00	$NaNO_3$	85.00	174.92	g	6996.79	g
NO_3	5.01E+00	15.9 *M* HNO_3		46.83	ml	1873.02	ml
PO_4	1.37E-02	14.8 *M* H_3PO_4		0.93	ml	37.03	ml
SO_4	5.40E-02	18.0 *M* H_2SO_4		3.00	ml	119.96	ml
Zn	1.05E-03	$Zn(NO_3)_2 \cdot 6H_2O$	297.47	0.31	g	12.48	g
Zr	6.33E-05	1.53*M* Zr in 5.4 ZrDP	91.22	0.04	ml	1.65	ml

5.4 ZrDP	Molarity
Zr	1.53E+00
F	1.17E+01
B	8.70E-01
H	2.10E+00

Actual NO_3= 5.15 *M*
Actual Acid = 1.01 *M*

***SB-72-4 Grout Mixture** 1 **Batch**
(C6-modified) 5 gallons
19 liters

Component	Weight %	27 kgs	kgs	lbs	liters	gal
SBW Simulant	72%		19.624	43.27	15.57	4.11
$Ca(OH)_2$	5%		1.363	3.00	0.60	0.16
Slag	0%		0	0	0	0
Cement	23%		6.269	13.82	2.75	0.73
Totals	100%		27.255	60.10	18.927	5.00

Densities (kg/L)		combined solids			
SBW Simulant	1.26	7.631	16.83	3.353	0.89
Cured Grout	1.44				
$Ca(OH)_2$	1.90				
Slag	3.00				
Cement	3.00				
Combined Solids	2.28				

*The C6 grout formulation was modified to combine the cement and calcium hydroxide together before mixing with the SBW simulant to eliminate a mixing step with the continuous grout mixer.

WM-189 Sodium-Bearing Waste -- Non-Hazardous Simulant Calculations
(based on "Characterization of Tank WM-189 SBW" by T. A. Batcheller, Table 4-5)
Updated: 2/25/03 to use 13*M* Nitric Acid
Make-up Date: 4/17/2003
Made-up By: S. H. Hinckley

Species	Desired Molarity	Stock Chemical	Molecular Weight	1 L Batch Amount		40.00 L Batch Amount	
Al	7.11E-01	2.2 *M* $Al(NO_3)_3$		323.18	ml	12927.27	ml
B	2.12E-02	H_3BO_3	61.832	1.31	g	52.43	g
Ca	7.30E-02	$Ca(NO_3)_2 \cdot 4H_2O$	236.15	17.24	g	689.56	g
Cl	2.06E-02	12.0 *M* HCl		1.72	ml	68.67	ml
Cu	9.54E-04	$Cu(NO_3)_2 \cdot 2.5H_2O$	232.59	0.22	g	8.88	g
F	1.38E-02	27.6 *M* HF		0.40	ml	16.04	ml
Fe	2.68E-02	$Fe(NO_3)_3 \cdot 9H_2O$	404.00	10.83	g	433.09	g
H	2.86E+00	All Acids	-----	-----		-----	
K	2.25E-01	KNO_3	101.10	22.75	g	909.90	g
Mg	2.21E-02	$Mg(NO_3)_2 \cdot 6H_2O$	256.41	5.67	g	226.67	g
Mn	1.95E-02	$Mn(NO_3)_2$ [50% soln.]	178.95	6.98	g soln.	279.16	g soln.
Na	2.04E+00	$NaNO_3$	85.00	173.39	g	6935.59	g
NO_3	6.52E+00	13 *M* HNO_3		195.52	ml	7820.89	ml
PO_4	2.07E-03	14.8 *M* H_3PO_4		0.14	ml	5.59	ml
SO_4	1.07E-01	18.0 *M* H_2SO_4		5.94	ml	237.78	ml
Zn	1.07E-03	$Zn(NO_3)_2 \cdot 6H_2O$	297.47	0.32	g	12.73	g
Zr	3.57E-04	1.53*M* Zr in 5.4 ZrDP	91.22	0.23	ml	9.33	ml

5.4 ZrDP	Molarity
Zr	1.53E+00
F	1.17E+01
B	8.70E-01
H	2.10E+00

Actual NO_3= 7.25 *M*
Actual Acid = 2.86 *M*

***S89-68-9 Grout Mixture** 1 **Batch**
(A6-modified) 5 gallons
19 liters

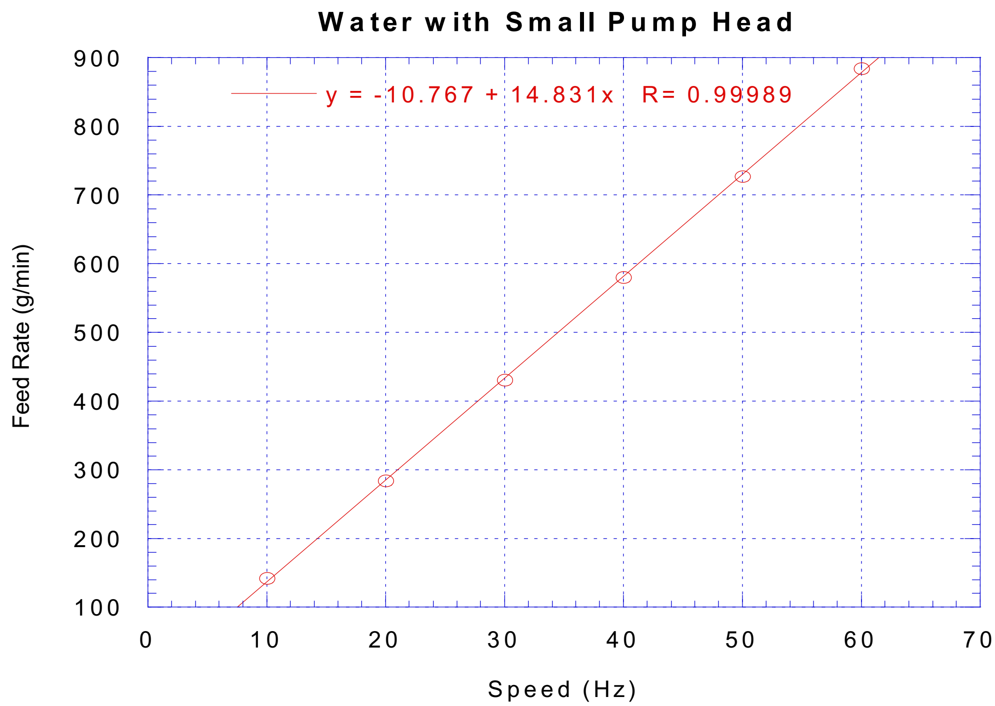
Component	Weight %	30 kgs	kgs	lbs	liters	gal
SBW Simulant	68%		20.593	45.41	15.526	4.10
$Ca(OH)_2$	5%		1.514	3.34	0.531	0.14
Slag	0%		0	0	0	0
Cement	27%		8.176	18.03	2.870	0.76
Totals	100%		30.283	66.77	18.928	5.00

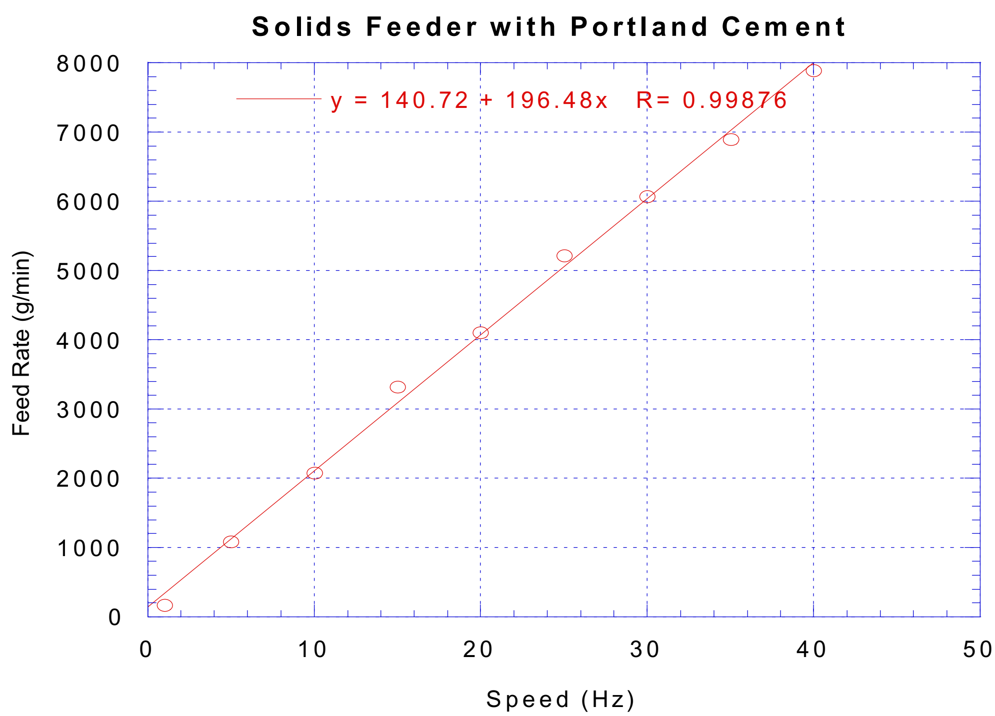
Densities (kg/L)		combined solids			
SBW Simulant	1.33	9.691	21.37	3.401	0.90
Cured Grout	1.60				
$Ca(OH)_2$	1.90				
Slag	3.00				
Cement	3.00				
Combined Solids	2.85				

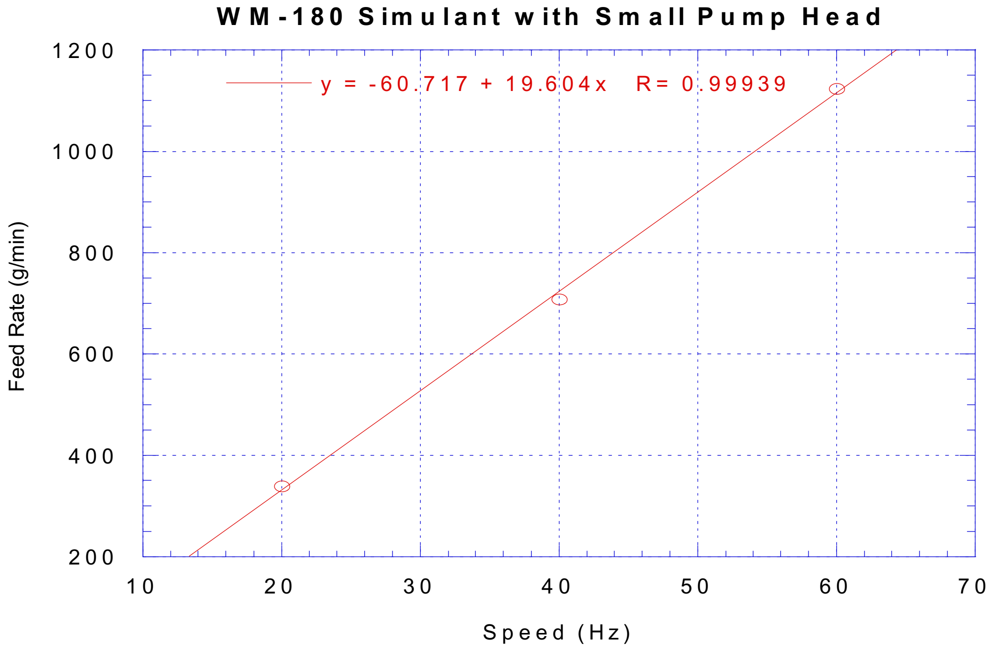
*The A6 grout formulation was modified to combine the cement and calcium hydroxide together before mixing with the SBW simulant to eliminate a mixing step with the continuous grout mixer.

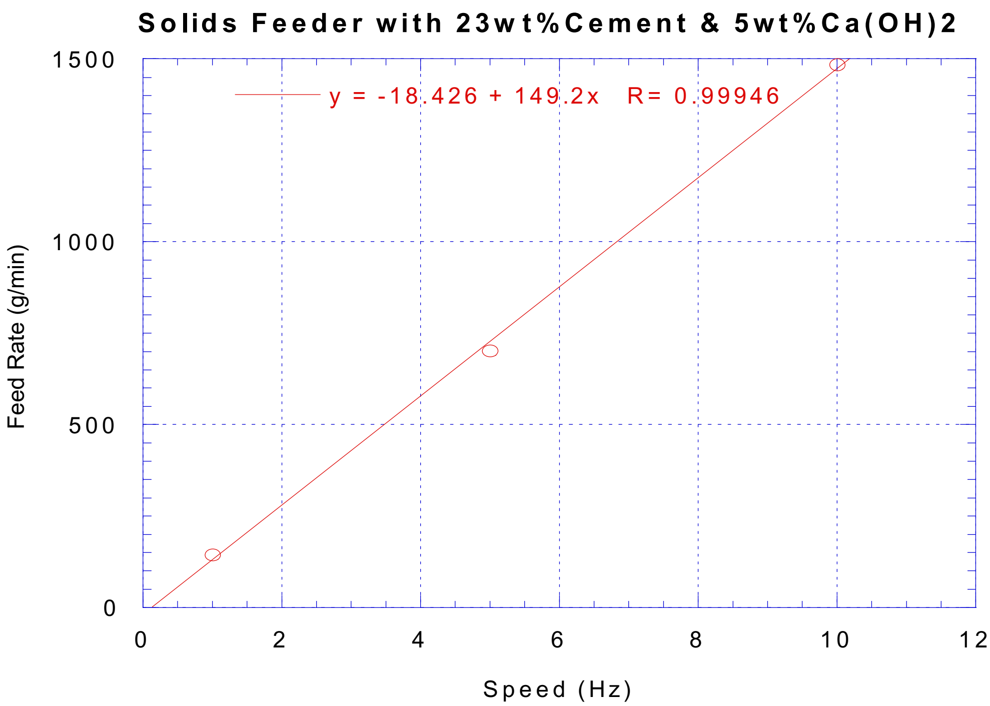
Appendix B

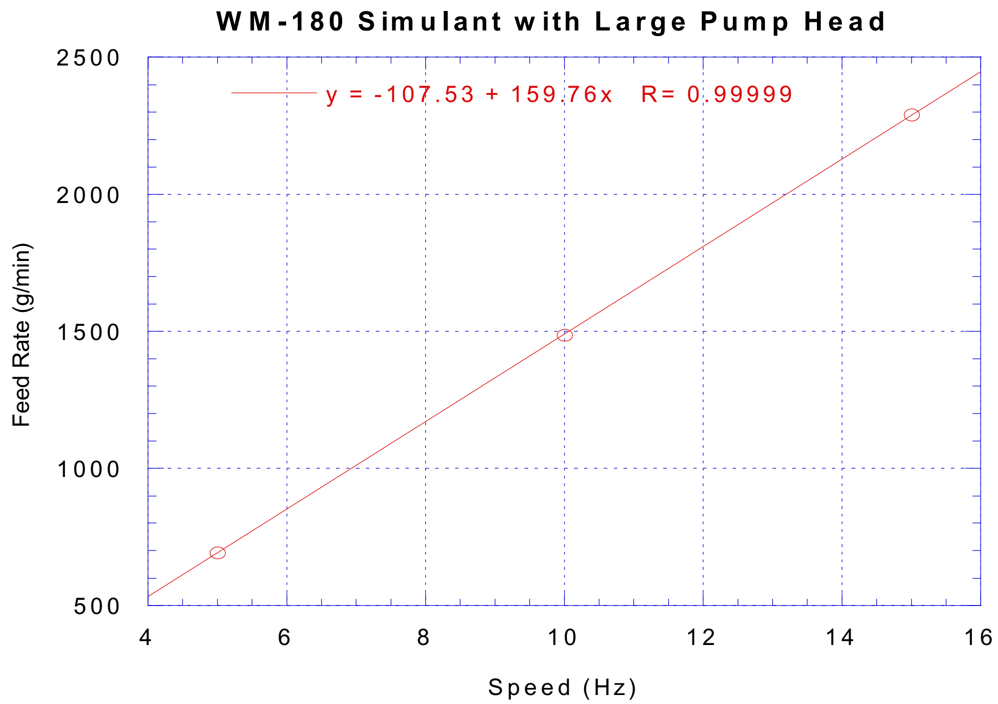
Calibration Curves for Liquid and Solid Feed Rates

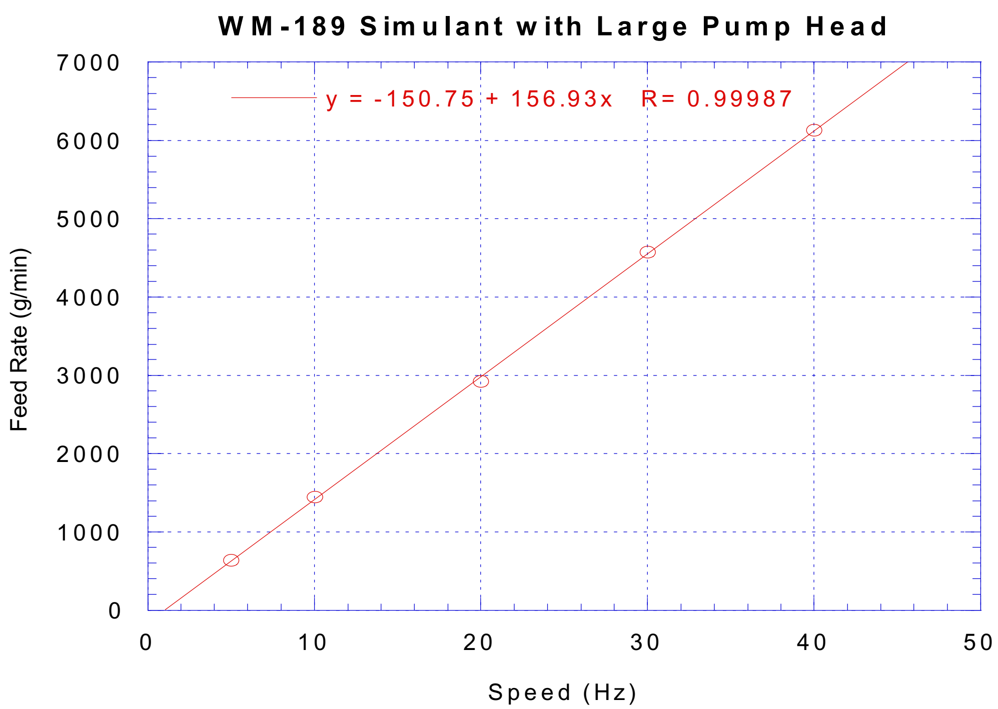

Water with Small Pump Head
y = -10.767 + 14.831x R= 0.99989
Feed Rate (g/min)
900
800
700
600
500
400
300
200
100
0
10
20
30
40
50
60
70
Speed (Hz)

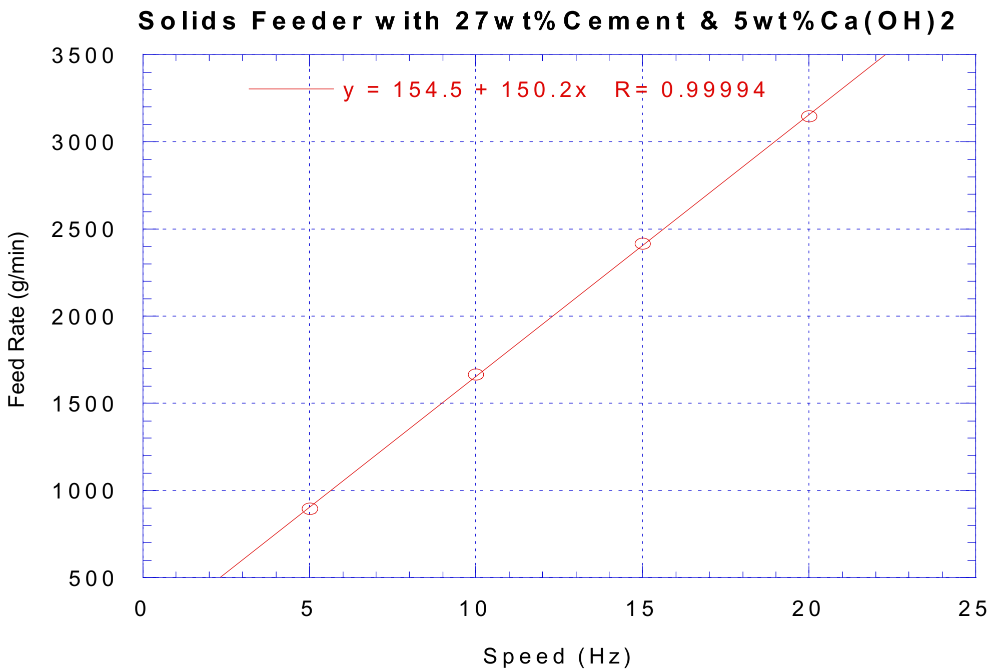

Solids Feeder with Portland Cement
y = 140.72 + 196.48x R= 0.99876
Feed Rate (g/min)
8000
7000
6000
5000
4000
3000
2000
1000
0
10
20
30
40
50
Speed (Hz)


WM-180 Simulant with Small Pump Head
y = -60.717 + 19.604x R= 0.99939
Feed Rate (g/min)
Speed (Hz)
1200
1000
800
600
400
200
10
20
30
40
50
60
70

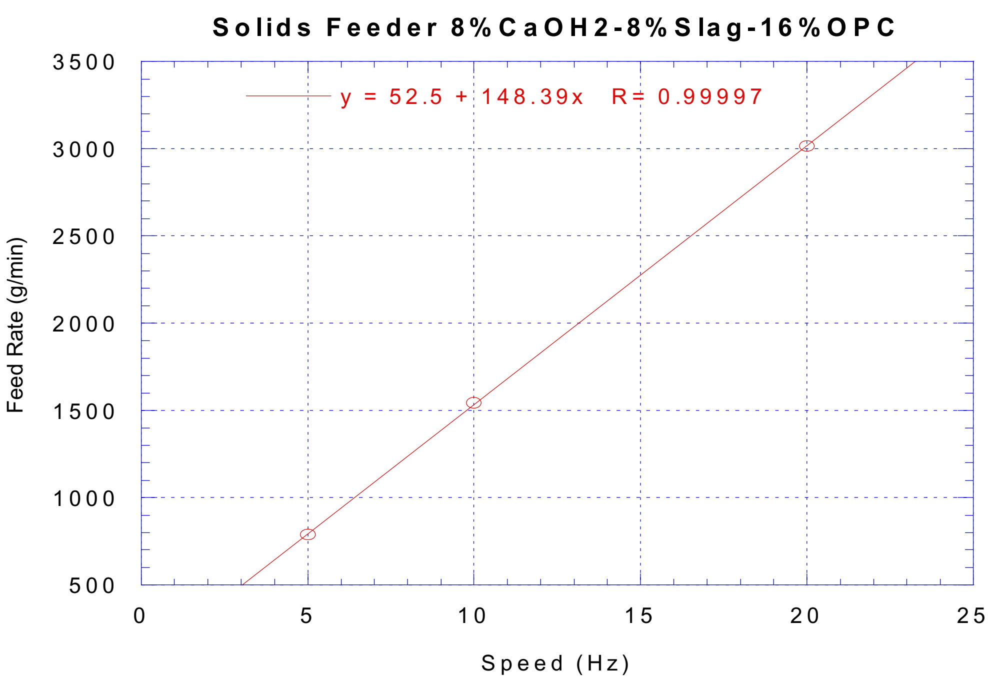

Solids Feeder with 23wt%Cement & 5wt%Ca(OH)2
y = -18.426 + 149.2x R= 0.99946
Feed Rate (g/min)
Speed (Hz)
1500
1000
500
0
0
2
4
6
8
10
12


WM-180 Simulant with Large Pump Head
y = -107.53 + 159.76x R= 0.99999
Feed Rate (g/min)
2500
2000
1500
1000
500
4
6
8
10
12
14
16
Speed (Hz)

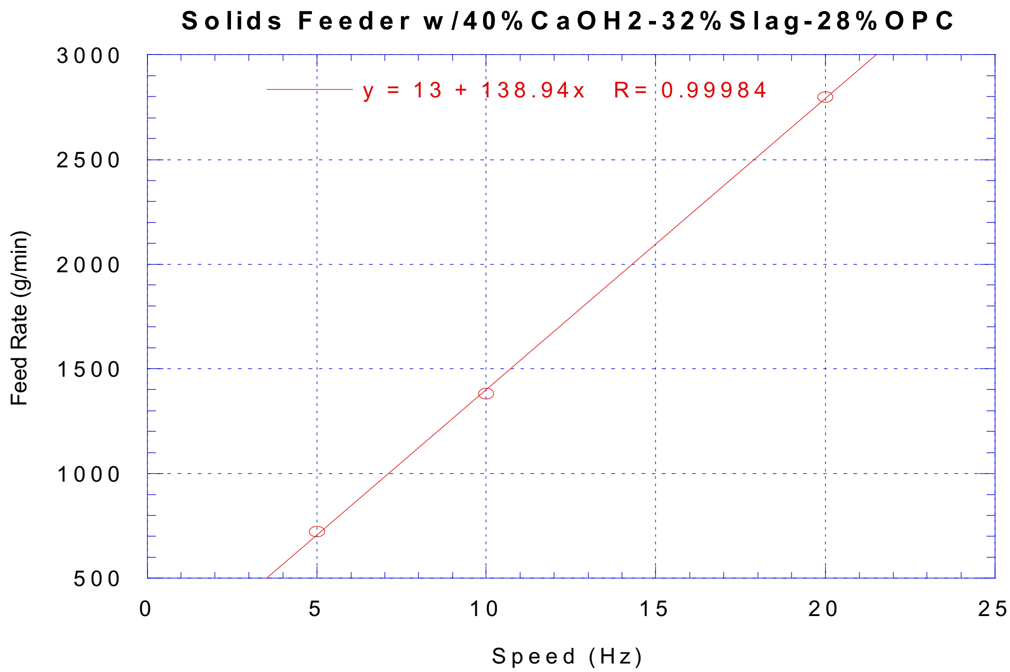

WM-189 Simulant with Large Pump Head
y = -150.75 + 156.93x R= 0.99987
Feed Rate (g/min)
7000
6000
5000
4000
3000
2000
1000
0
0
10
20
30
40
50
Speed (Hz)

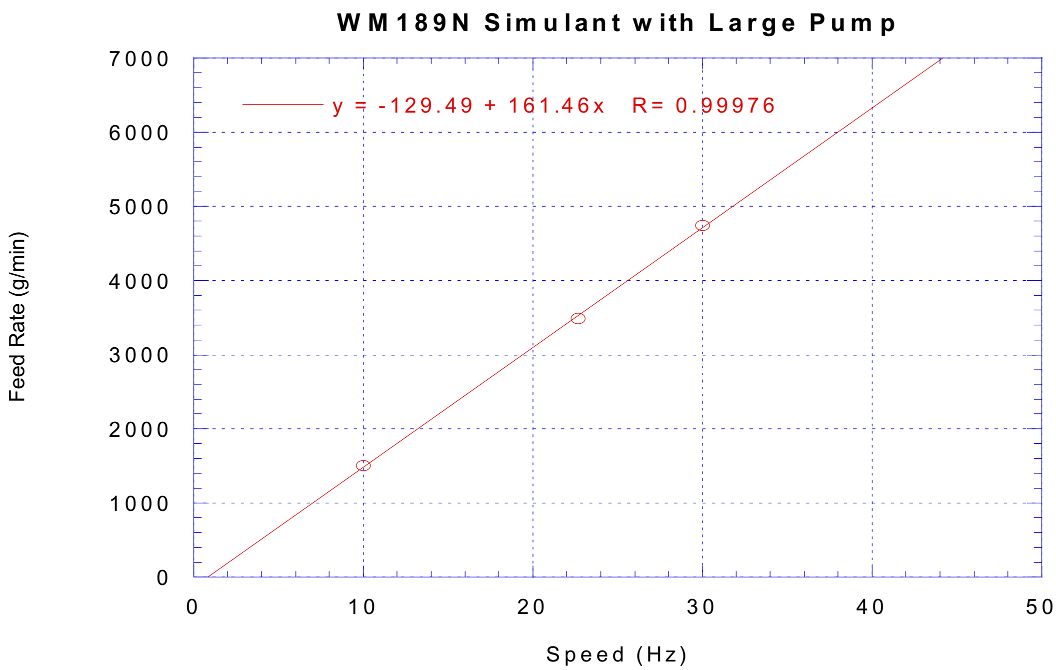


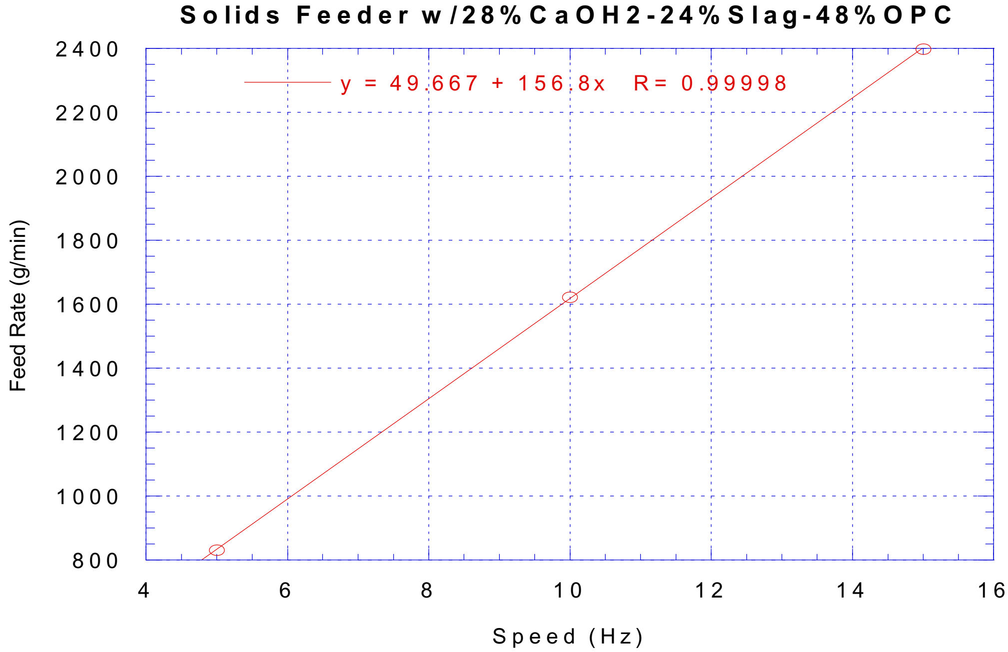
Solids Feeder with 27wt%Cement & 5wt%Ca(OH)2
y = 154.5 + 150.2x R= 0.99994
Feed Rate (g/min)
Speed (Hz)
3500
3000
2500
2000
1500
1000
500
0
5
10
15
20
25

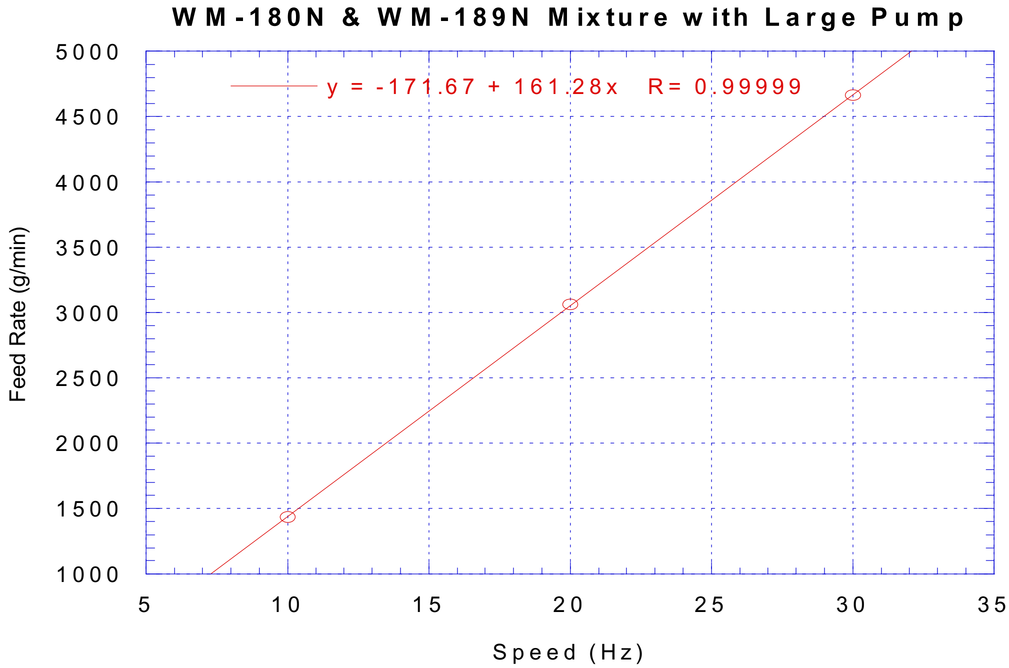


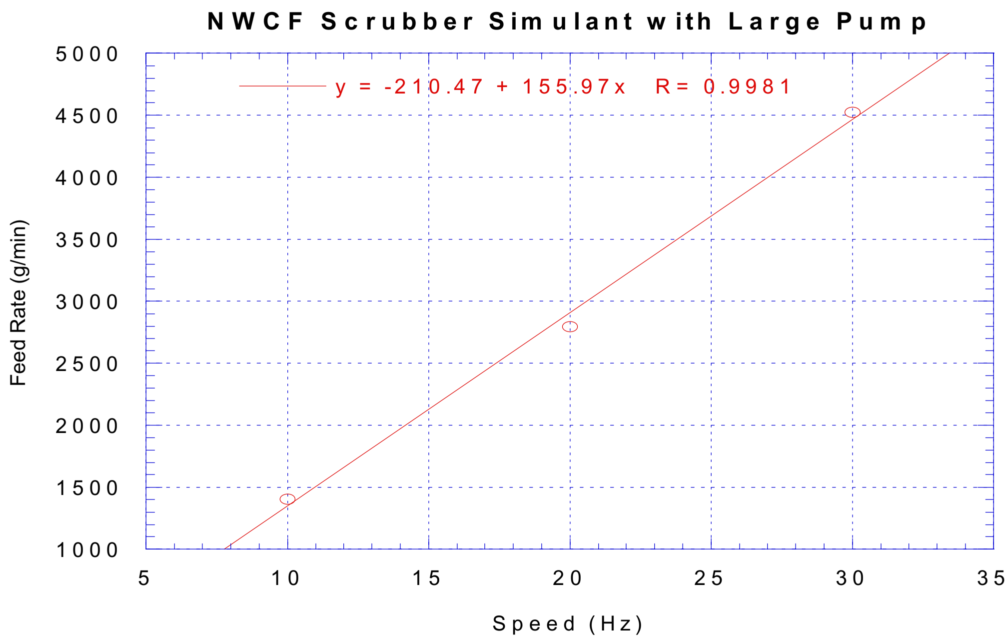
Solids Feeder 8%CaOH2-8%Slag-16%OPC
y = 52.5 + 148.39x R= 0.99997
Feed Rate (g/min)
Speed (Hz)
3500
3000
2500
2000
1500
1000
500
0
5
10
15
20
25

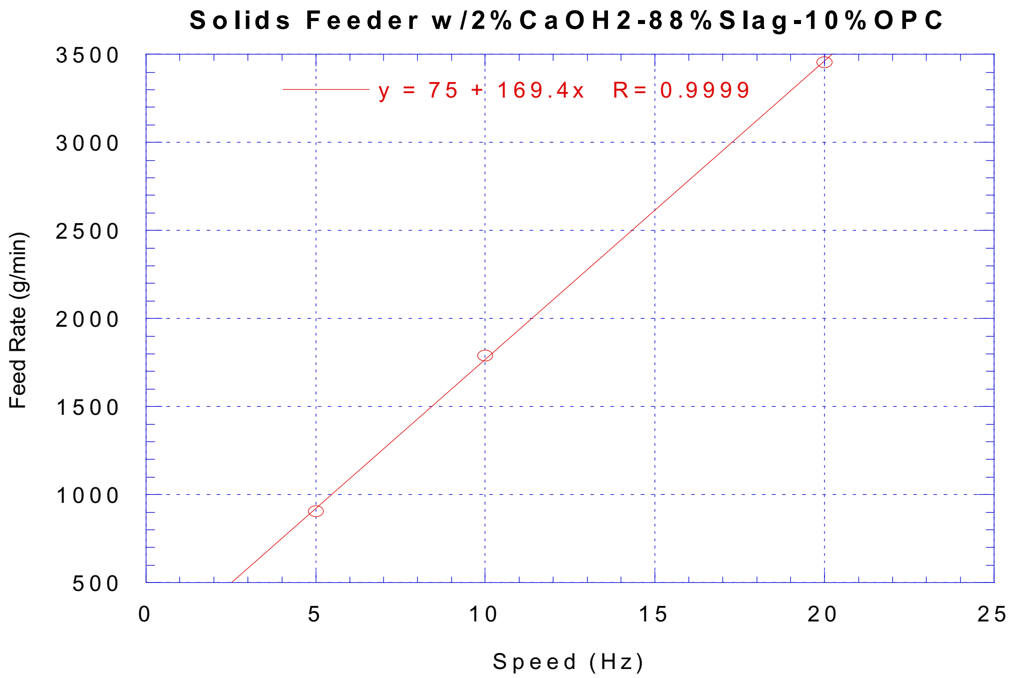

Solids Feeder w/40%CaOH2-32%Slag-28%OPC
y = 13 + 138.94x R= 0.99984
Feed Rate (g/min)
3000
2500
2000
1500
1000
500
0
5
10
15
20
25
Speed (Hz)


WM189N Simulant with Large Pump
y = -129.49 + 161.46x R= 0.99976
Feed Rate (g/min)
7000
6000
5000
4000
3000
2000
1000
0
0
10
20
30
40
50
Speed (Hz)


Solids Feeder w/28%CaOH2-24%Slag-48%OPC
y = 49.667 + 156.8x R= 0.99998
Feed Rate (g/min)
2400
2200
2000
1800
1600
1400
1200
1000
800
4
6
8
10
12
14
16
Speed (Hz)


WM-180N & WM-189N Mixture with Large Pump
y = -171.67 + 161.28x R= 0.99999
Feed Rate (g/min)
5000
4500
4000
3500
3000
2500
2000
1500
1000
5
10
15
20
25
30
35
Speed (Hz)


NWCF Scrubber Simulant with Large Pump
y = -210.47 + 155.97x R= 0.9981
Feed Rate (g/min)
5000
4500
4000
3500
3000
2500
2000
1500
1000
5
10
15
20
25
30
35
Speed (Hz)


Solids Feeder w/2%CaOH2-88%Slag-10%OPC
y = 75 + 169.4x R= 0.9999
Feed Rate (g/min)
3500
3000
2500
2000
1500
1000
500
0
5
10
15
20
25
Speed (Hz)

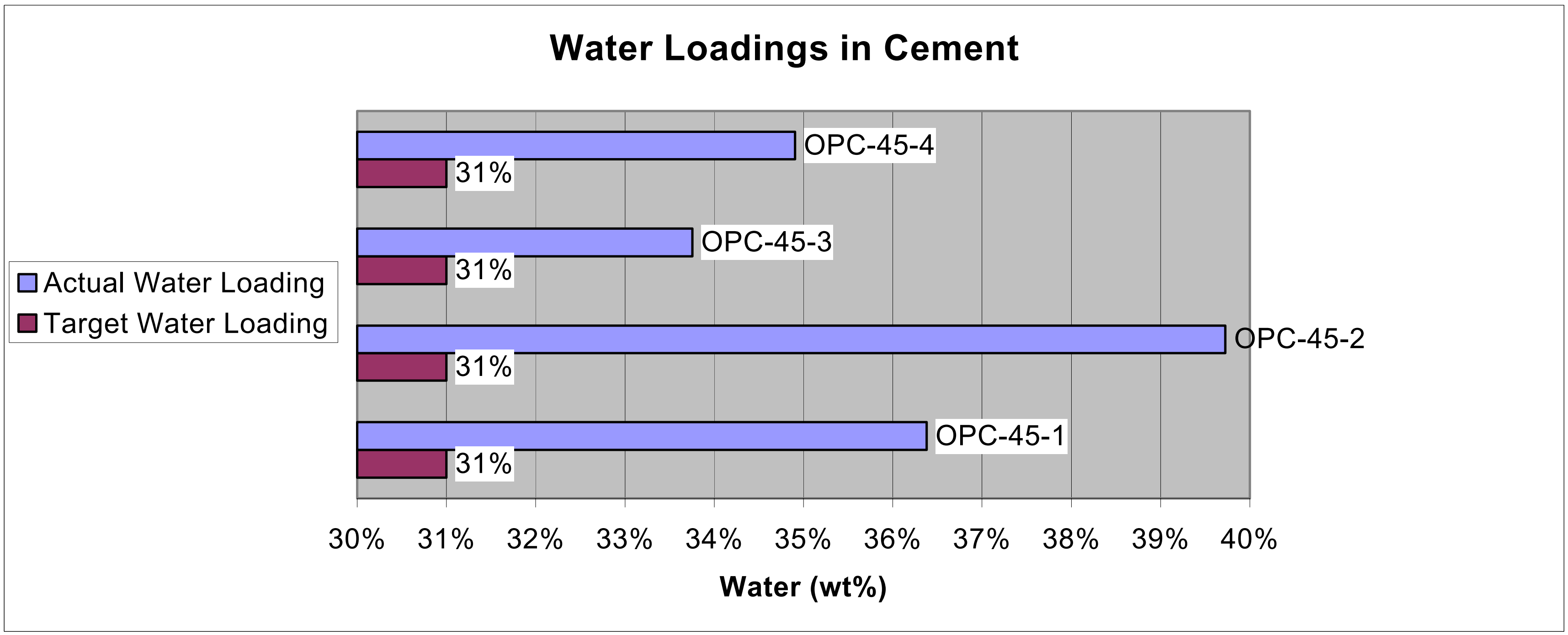
Appendix C

Out-Of-Drum Grout Mixer Test Data

Full-Scale Out-Of-Drum Mixer Test Data using Ordinary Portland Cement and Water

Samples	Test Date	Grout Mixer Speed (Hz)	Liquid Pump Speed (Hz)	Solids Feeder Speed (Hz)	Run Time (min)	Actual Liquid Feed (g)	Actual Solid Feed (g)	Actual Cement Product (g)	Actual Water Loading (wt%)	Actual Cement Loading (wt%)	Target Water Loading (wt%)	Target Cement Loading (wt%)	Sample Batch Size (gal)	Viscosity @ 10 rpm (cP)	@ Torque (%)	Cured Mass (g)	Cured Density (g/mL)	Comp-ressive Strength (psi)	Comments
OPC-50-1	3/19/03	20	54.94	3.38	26	NM	NM	NM	NM	NM	50%	50%	5	NM	NM	NM	NM	NM	Mixer plugged
OPC-50-2	3/20/03	20	48.87	6.57	3	2240	1793	4033	56%	44%	33%	67%	1	NM	NM	NM	NM	NM	Too thin
OPC-45-1	3/20/03	20	48.19	7.26	3	2150	3760	5910	36%	64%	31%	69%	1	8000	5	180.6	1.57	2760	Too thin
OPC-45-2	3/20/03	20	48.19	7.26	3	2030	3080	5110	40%	60%	31%	69%	1	NM	NM	197.9	1.65	3120	Too thin
OPC-45-3	3/20/03	20	48.19	7.26	3	2140	4200	6340	34%	66%	31%	69%	1	36000	15	NM	NM	NM	Not well mixed
OPC-45-4	3/20/03	20	48.19	7.26	3	2000	3730	5730	35%	65%	31%	69%	1	7000	5	191.5	1.63	2760	Too thin

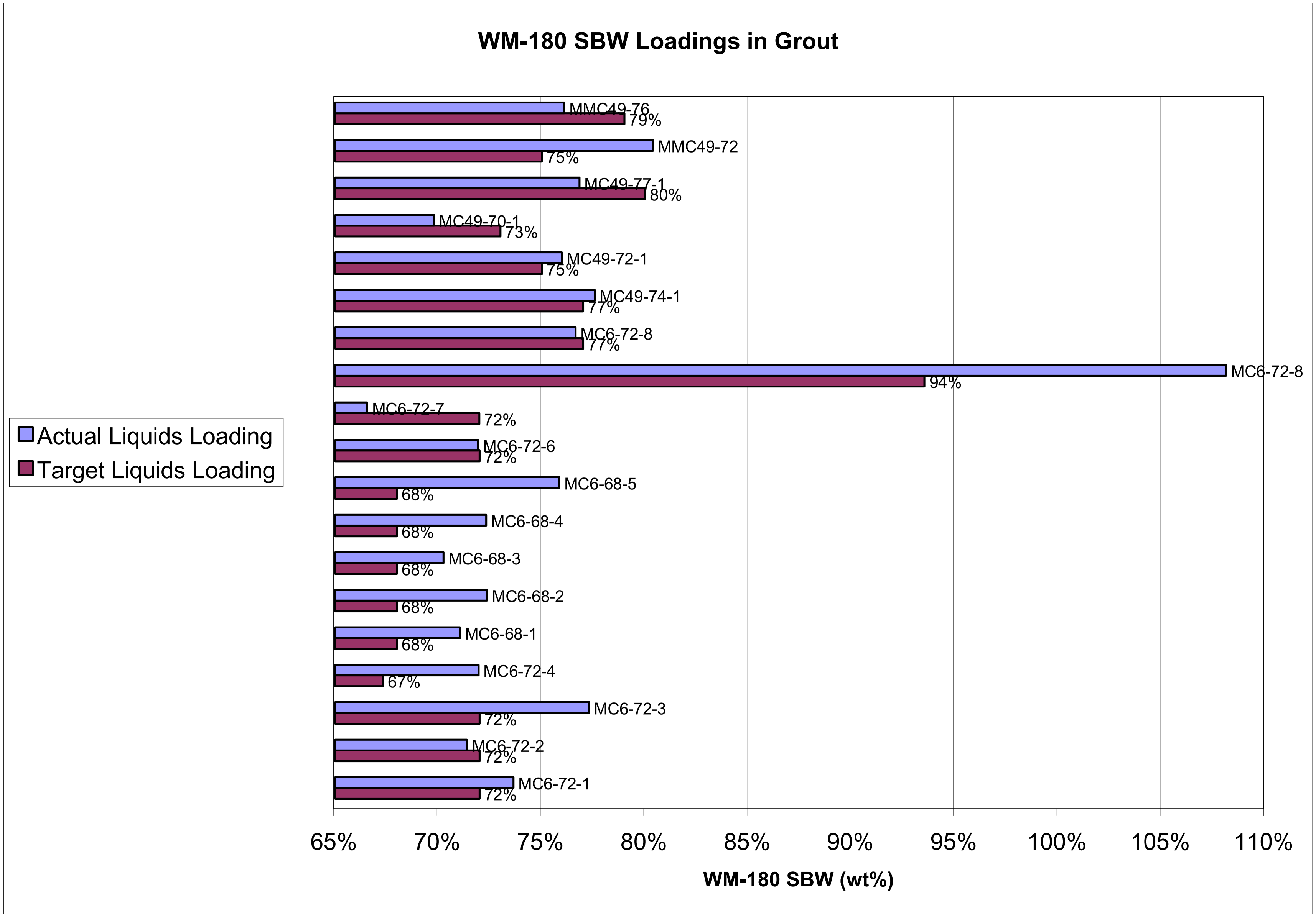
Actual –vs– Target Water Loadings in Cement



out xer eed z)	Liquid Pump Speed (Hz)	Solids Feeder Speed (Hz)	Run Time (min)	Actual Liquid Feed (g)	Actual Solid Feed (g)	Actual Grout Product (g)	Actual Liquids Loading (wt%)	Actual Solids Loading (wt%)	Target Liquids Loading (wt%)	Target Solids Loading (wt%)	Target SBW Loading (wt%)	Target NaOH Loading (wt%)	Target $Ca(OH)_2$ Loading (wt%)	Target Slag Loading (wt%)	Target Cement Loading (wt%)	Target Total Loading (wt%)	
0	53.14	2.68	4	4000	1433	5433	74%	26%	72%	28%	72.0%	0.0%	5.0%	0.0%	23.0%	100.0%	Invalid
5	53.14	2.68	4	3040	1220	4260	71%	29%	72%	28%	72.0%	0.0%	5.0%	0.0%	23.0%	100.0%	Invalid
0	53.14	2.68	4	3540	1041	4581	77%	23%	72%	28%	72.0%	0.0%	5.0%	0.0%	23.0%	100.0%	Invalid
5	50.38	3.14	4	3730	1455	5185	72%	28%	67%	33%	67.0%	0.0%	6.0%	0.0%	27.0%	100.0%	Invalid
0	44.47	2.68	4	3240	1321	4561	71%	29%	68%	32%	68.0%	0.0%	6.0%	0.0%	26.0%	100.0%	Ok, fai
5	44.47	2.68	4	3360	1285	4645	72%	28%	68%	32%	68.0%	0.0%	6.0%	0.0%	26.0%	100.0%	Ok, ve
0	44.47	2.68	4	3200	1356	4556	70%	30%	68%	32%	68.0%	0.0%	6.0%	0.0%	26.0%	100.0%	Ok, ve
0	44.47	2.68	4	3180	1218	4398	72%	28%	68%	32%	68.0%	0.0%	6.0%	0.0%	26.0%	100.0%	Ok, fai
5	44.47	2.68	4	3290	1048	4338	76%	24%	68%	32%	68.0%	0.0%	6.0%	0.0%	26.0%	100.0%	Ok, fai
0	53.14	2.68	4	3930	1535	5465	72%	28%	72%	28%	72.0%	0.0%	5.0%	0.0%	23.0%	100.0%	Ok, fai
0	53.14	2.68	14.5	11810	5936	17746	67%	33%	72%	28%	72.0%	0.0%	5.0%	0.0%	23.0%	100.0%	Ok, ve
0	11.98	.56-1.25	2	3613	-270	3342	108%	-8%	94%	6%	72.0%	0.0%	5.0%	0.0%	0.0%	77.0%	Mixed
0	11.98	2.22	2	3342	1019	4361	77%	23%	77%	23%	72.0%	0.0%	5.0%	0.0%	23.0%	100.0%	Mixed
0	21.40	7.03	1.2	3420	990	4410	78%	22%	77%	23%	74.0%	3.0%	9.2%	6.4%	7.4%	100.0%	Very th
0	21.41	7.85	1.2	3310	1047	4357	76%	24%	75%	25%	72.0%	3.0%	10.0%	7.0%	8.0%	100.0%	Very th
0	21.40	8.72	1.2	3390	1468	4858	70%	30%	73%	27%	70.0%	3.0%	10.8%	7.6%	8.6%	100.0%	Very th
0	21.40	5.87	1.2	3290	993	4283	77%	23%	80%	20%	77.0%	3.0%	8.0%	5.6%	6.4%	100.0%	Very th
0	25.5	9.42	20	64550	15760	80310	80%	20%	75%	25%	72.0%	3.0%	10.0%	7.0%	8.0%	100.0%	Very th
36	26.8	7.90	31.25	103470	32530	136000	76%	24%	79%	21%	76.0%	3.0%	8.4%	5.9%	6.7%	100.0%	Very th
0	22.00	6.93	1.2	3410	877	4287	80%	20%	75%	25%	72.0%	3.0%	7.0%	6.0%	12.0%	100.0%	Fairly t
0	22.00	6.93	6	17740	6270	24010	74%	26%	75%	25%	72.0%	3.0%	7.0%	6.0%	12.0%	100.0%	Fairly t

ple ch e)	Grout Mix (pH)	@ Temp. (C)	Mix Viscosity @10 rpm (cP)	@ Torque (%)	Mix Viscosity @ 5 rpm (cP)	@ Torque (%)	Mix Viscosity @ 2.5 rpm (cP)	@ Torque (%)	Wet Grout Mass (g)	Wet Grout Density (g/mL)	7-day Vicat Depth (mm)	>28-day Vicat Depth (mm)	Cured Grout Mass (g)	Cured Grout Density (g/mL)	Comp-ressive Strength (psi)	Cured Com
	9.49	39	8560	8.3	14700	9	NM	NM	NM	NM	NM	NM	NM	NM	NM	Discarded because of not we
	9.21	39	2400	30	4600	29	NM	NM	NM	NM	NM	NM	NM	NM	NM	Discarded because of not we
	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	Discarded because of not we
	10	33	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	Discarded because of not we
	9.8	59	25600	27	26100	16	32300	10.2	4561	1.55	>50	0	2853	1.60	1740	Fuzzy with white spots, som
	9.9	45	52000	16	60000	9.2	130000	10.1	4645	1.57	>50	0	2940	1.56	3650	Fuzzy without white spots, s
	10	47	442000	14	60260	9	97000	6	4556	1.54	>50	0	2898	1.51	3310	Fuzzy without white spots, s
	10	59	30480	38	38200	24	47700	14	4398	1.49	>50	0	2768	1.55	3580	Fuzzy without white spots, s
	10	49	32000	42	35000	25	40000	12.5	4338	1.47	>50	0	2629	1.58	2790	Fuzzy with white spots, som
	9.48	40	7200	10	12000	7	192000	6.1	4141	1.40	>50	0	2937	1.57	980	Fuzzy with white spots, som
	NM	32	NM	NM	NM	NM	NM	NM	NM	NM	>50	0	15342	NM	NM	Fuzzy with white spots, som
6	3	29	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	Neutralization step prior to g
2	10	29	NM	NM	NM	NM	NM	NM	4299	1.98	>50	0	NM	1.59	NM	white spots, indications of ba
	NM	20	NM	NM	NM	NM	NM	NM	183.2	1.40	>50	0	226.1	1.47	470	no NaNO3 crystals
	NM	24	NM	NM	NM	NM	NM	NM	183.0	1.40	>50	0	201.4	1.41	740	no NaNO3 crystals
	NM	25	NM	NM	NM	NM	NM	NM	185.0	1.41	>50	0	241.0	1.48	740	no NaNO3 crystals
	NM	25	NM	NM	NM	NM	NM	NM	180.0	1.37	>50	0	95.4	0.79	NM	Lots of NaNO3 crystals and
5	NM	NM	NM	NM	NM	NM	NM	NM	186.0	1.42	>50	1	124.6	1.28	NM	cracked, fuzzy, w/lots of shri
2	11.37	35	NM	NM	NM	NM	NM	NM	179.0	1.37	>50	1	105.4	0.86	NM	cracked, fuzzy, w/lots of shri
	9.35	32	NM	NM	NM	NM	NM	NM	187.4	1.43	>50	0	210.8	1.80	100	cracked, fuzzy, w/crusty laye
	NM	NM	NM	NM	NM	NM	NM	NM	191.0	1.46	>51	2	107.3	0.88	NM	cracked, fuzzy, w/crusty laye

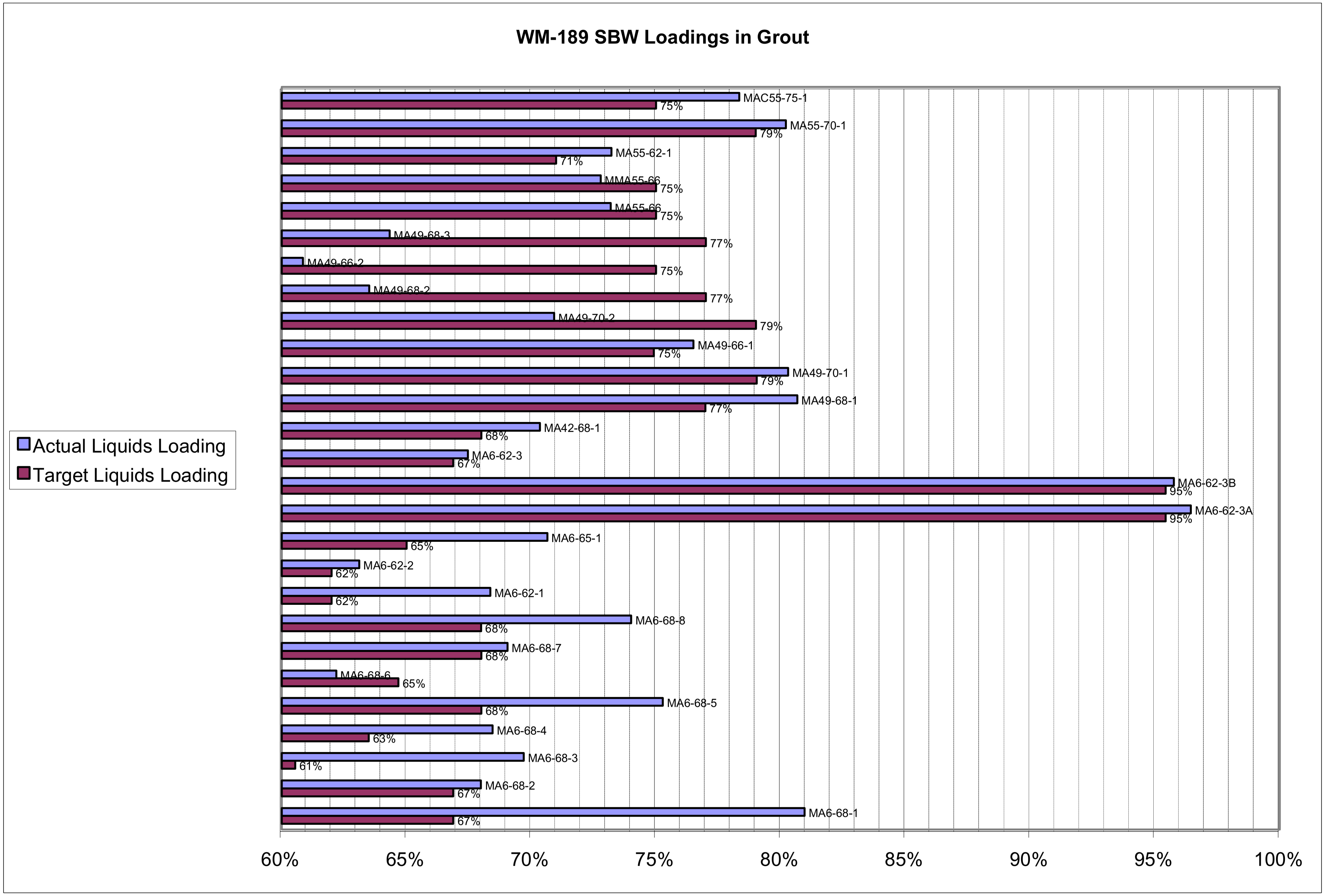
Actual –vs– Target WM-180 SBW Simulant Loadings in Grout



rout xer eed (Hz)	Liquid Pump Speed (Hz)	Solids Feeder Speed (Hz)	Run Time (min)	Actual Liquid Feed (g)	Actual Solid Feed (g)	Actual Grout Product (g)	Actual Liquids Loading (wt%)	Actual Solids Loading (wt%)	Target Liquids Loading (wt%)	Target Solids Loading (wt%)	Target SBW Loading (wt%)	Target NaOH Loading (wt%)	Target $Ca(OH)_2$ Loading (wt%)	Target Slag Loading (wt%)	Target Cement Loading (wt%)	Target Total Loading (wt%)	
20	9.27	3.27	3	4200	988	5188	81%	19%	67%	33%	68.0%	0.0%	5.0%	0.0%	27.0%	100.0%	Too thin and no
20	9.27	3.27	3	3900	1837	5737	68%	32%	67%	33%	68.0%	0.0%	5.0%	0.0%	27.0%	100.0%	Too thin and no
20	9.27	4.63	3	4200	1826	6026	70%	30%	61%	39%	61.0%	0.0%	6.0%	0.0%	33.0%	100.0%	Too thick, but n
20	9.27	3.96	3	4200	1936	6136	68%	32%	63%	37%	63.0%	0.0%	6.0%	0.0%	31.0%	100.0%	Too thin and no
20	9.71	3.27	3	4020	1321	5341	75%	25%	68%	32%	68.0%	0.0%	5.0%	0.0%	27.0%	100.0%	Too thin and no
20	9.71	3.96	3	3830	2329	6159	62%	38%	65%	35%	64.7%	0.0%	5.5%	0.0%	29.8%	100.0%	Well mixed and
20	9.71	3.27	3	3880	1739	5619	69%	31%	68%	32%	68.0%	0.0%	5.0%	0.0%	27.0%	100.0%	Too thin and no
20	7.52	2.20	4	3940	1384	5324	74%	26%	68%	32%	68.0%	0.0%	5.0%	0.0%	27.0%	100.0%	Too thin and no
20	7.68	3.27	3	2900	1342	4242	68%	32%	62%	38%	62.0%	0.0%	5.9%	0.0%	32.1%	100.0%	Good sample is
20	7.68	3.27	15	15810	9244	25054	63%	37%	62%	38%	62.0%	0.0%	5.9%	0.0%	32.1%	100.0%	Good sample is
20	8.61	3.27	3	3150	1309	4459	71%	29%	65%	35%	65.0%	0.0%	5.5%	0.0%	29.5%	100.0%	Good sample is
20	7.68	0.75	15	15400	570	15970	96%	4%	95%	5%	95.4%	0.0%	4.6%	0.0%	0.0%	100.0%	Mixed fairly we
20	7.68	0.75	15	15800	700	16500	96%	4%	95%	5%	95.4%	0.0%	4.6%	0.0%	0.0%	100.0%	Mixed fairly we
20	7.97	2.06	15	16360	7890	24250	67%	33%	67%	33%	66.9%	0.0%	0.0%	0.0%	33.1%	100.0%	Too thin and no
20	21.47	9.85	6	19670	8290	27960	70%	30%	68%	32%	68.0%	0.0%	8.0%	8.0%	16.0%	100.0%	Well mixed and
20	22.57	7.20	5	17400	4170	21570	81%	19%	77%	23%	68.0%	9.0%	9.2%	7.4%	6.4%	100.0%	Mixed well and
20	22.57	6.38	5	16180	3970	20150	80%	20%	79%	21%	70.0%	9.0%	8.4%	6.7%	5.9%	100.0%	Mixed well and
20	22.57	8.08	2.25	6900	2120	9020	76%	24%	75%	25%	66.0%	9.0%	10.0%	8.0%	7.0%	100.0%	Mixed well and
20	22.67	6.42	2	3470	1423	4893	71%	29%	79%	21%	70.0%	9.0%	8.4%	5.9%	6.7%	100.0%	Well mixed and
20	22.67	7.23	2	3470	1994	5464	64%	36%	77%	23%	68.0%	9.0%	9.2%	6.4%	7.4%	100.0%	Well mixed and
20	22.67	8.08	2	3450	2220	5670	61%	39%	75%	25%	66.0%	9.0%	10.0%	7.0%	8.0%	100.0%	Well mixed and
20	22.67	7.23	2	3400	1886	5286	64%	36%	77%	23%	68.0%	9.0%	9.2%	6.4%	7.4%	100.0%	Well mixed and
20	22.05	6.98	6	16160	5920	22080	73%	27%	75%	25%	66.0%	9.0%	7.0%	6.0%	12.0%	100.0%	Well mixed and
-25	18.48	5.75	71	167410	62610	230020	73%	27%	75%	25%	66.0%	9.0%	7.0%	6.0%	12.0%	100.0%	Well mixed and
20	11.21	4.06	6	8200	3000	11200	73%	27%	71%	29%	62.0%	9.0%	8.1%	7.0%	13.9%	100.0%	Very thick, and
20	11.99	2.75	6	7820	1930	9750	80%	20%	79%	21%	70.0%	9.0%	5.9%	5.0%	10.1%	100.0%	Very thin, and f
20	22.48	7.03	5.66	19450	5380	24830	78%	22%	75%	25%	69.0%	6.0%	7.0%	6.0%	12.0%	100.0%	Well mixed, fai

ample Batch Size (gal)	Grout Mix (pH)	@ Temp. (C)	Mix Viscosity @10 rpm (cP)	@ Torque (%)	Mix Viscosity @ 5 rpm (cP)	@ Torque (%)	Mix Viscosity @ 2.5 rpm (cP)	@ Torque (%)	Wet Grout Mass (g)	Wet Grout Density (g/mL)	7-day Vicat Depth (mm)	>28-day Vicat Depth (mm)	Cured Grout Mass (g)	Cured Grout Density (g/mL)	Comp-ressive Strength (psi)	C C
1	8 or 9	31	NM	NM	NM	NM	NM	NM	NM	NM	>50	NM	NM	NM	NM	Discarded because
1	8.45	53	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	Discarded because
1	NM	32	NM	NM	NM	NM	NM	NM	NM	NM	>50	0	3553	NM	NM	Some white spots, n
1	8.41	55	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	Discarded because
1	NM	32	NM	NM	NM	NM	NM	NM	NM	NM	>50	NM	NM	NM	NM	Discarded because
1	8.58	58	28000	NM	NM	NM	NM	NM	NM	NM	NM	0	216.7	1.64	1490	Solid with white oute
1	NM	39	NM	NM	NM	NM	NM	NM	NM	NM	>50	NM	NM	NM	NM	Discarded because
1	8.55	50	3000	NM	NM	NM	NM	NM	NM	NM	>50	NM	NM	NM	NM	Discarded because
1	8.90	48	8000	10	11700	7	NM	NM	199.04	1.59	>50	0	160.0	1.61	1480	Solid with white oute
5	NM	57	NM	NM	NM	NM	NM	NM	NM	NM	>50	0	24114	NM	NM	Solid with white oute
1	8.68	52	2560	3.2	4300	2.7	NM	NM	192.94	1.54	>50	0	212.3	1.70	1660	Solid with white spot
5	-0.07	37	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	Neutralization step p
5	1.96	50	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	Neutralization step p
5	8.76	42	NM	NM	NM	NM	NM	NM	NM	NM	>50	0	23590	NM	NM	Solid, no fuzz
5	7.74	60	NM	NM	NM	NM	NM	NM	201.7	1.54	>50	10	166.9	1.61	110	Still soft, no fuzz, so
5	NM	21	NM	NM	NM	NM	NM	NM	180.8	1.38	35	0	115.3	NM	NM	Too badly cracked a
5	9.18	40	NM	NM	NM	NM	NM	NM	178.7	1.36	40	22	122.5	NM	NM	Too badly cracked a
1.6	NM	20	NM	NM	NM	NM	NM	NM	190.5	1.45	35	0	137.9	NM	NM	Too badly shrunken
1	NM	49	~50000	NM	NM	NM	NM	NM	193.2	1.47	>50	0	147.8	1.58	1040	Solid with fuzz and g
1	NM	48	~50000	NM	NM	NM	NM	NM	200.1	1.53	>50	0	159.6	1.61	900	Solid with fuzz and g
1	NM	51	~50000	NM	NM	NM	NM	NM	203.5	1.55	>50	0	169.5	1.60	1000	Solid with fuzz and g
1	NM	52	~50000	NM	NM	NM	NM	NM	4250	1.44	>50	0	209.5	1.60	380	Solid with fuzz and g
5	NM	56	NM	NM	NM	NM	NM	NM	NM	NM	>50	0	150.8	1.36	1140	Solid with no fuzz or
50	NM	54	NM	NM	NM	NM	NM	NM	NM	NM	>50	0	142.6	1.40	527	Solid with no fuzz or
2.5	NM	NM	NM	NM	NM	NM	NM	NM	195.2	1.49	NM	1	149.5	1.19	NM	very fuzzy
2.5	NM	NM	NM	NM	NM	NM	NM	NM	183.7	1.40	>50	>50	151.1	1.28	NM	still wet
2.5	8.99	40	NM	NM	NM	NM	NM	NM	185.6	1.42	>50	40	145.2	1.21	NM	still wet, cracked and

Actual –vs– Target WM-189 SBW Simulant Loadings in Grout



Full-Scale Out-Of-Drum Mixer Test Data using Neutralized NWCF Scrubber Non-Hazardous Simulant

Pre-neutralized to ~pH of 12 w/NaOH — pH measured at 12.95 @ 16.3C with density of 1.28g/ml

Samples	Test Date	Grout Mixer Speed (Hz)	Liquid Pump Speed (Hz)	Solids Feeder Speed (Hz)	Run Time (min)	Actual Liquid Feed (g)	Actual Solid Feed (g)	Actual Grout Product (g)	Actual Liquid Loading (wt%)	Actual Solids Loading (wt%)	Target Liquid Loading (wt%)	Target Solids Loading (wt%)	Target Total Loading (wt%)	Wet Grout Comments
MB1-35-1	8/5/03	25	19.79	14.61	1.2	3440	2960	6400	54%	46%	53%	47%	100.0%	Very thin, but well mixed
MB1-26-2	8/5/03	20	15.43	19.00	1.2	2570	3490	6060	42%	58%	40%	60%	100.0%	Fairly thick and well mixed
MB1-30-3	8/5/03	20	17.34	17.23	1.2	3030	3400	6430	47%	53%	45%	55%	100.1%	Thin, but well mixed
MB1-26-4	8/5/03	20	15.43	19.00	2.75	5980	7220	13200	45%	55%	40%	60%	100.0%	Fairly thick and well mixed

Samples	Test Date	Sample Batch Size (gal)	Wet Grout (pH)	Grout Viscosity (cP)	@ Speed (RPM)	@ Torque (%)	Wet Grout Mass (g)	Wet Grout Density (g/mL)	7-day Vicat Depth (mm)	>28-day Vicat Depth (mm)	Cured Grout Mass (g)	Cured Grout Density (g/mL)	Comp-ressive Strength (psi)	Cured Grout Comments
MB1-35-1	8/5/03	1	NM	NM	NM	NM	217.0	1.66	5	0	212.6	1.75	1765	Solid, w/lighter colored top layer
MB1-26-2	8/5/03	1	NM	NM	NM	NM	241.9	1.85	1	0	239.1	1.96	4790	Solid, w/lighter colored top layer
MB1-30-3	8/5/03	1	NM	NM	NM	NM	236.5	1.81	1	0	233.5	1.89	4000	Solid, w/lighter colored top layer
MB1-26-4	8/5/03	2.5	12.35	20000	12	26	240.4	1.84	0	0	238.0	1.90	3410	Solid, w/lighter colored top layer

Actual –vs– Target NWCF Scrub Simulant Loadings in Grout